

Revealing the Potential of Waste Fibers from Timber Production and Clearings for the Development of Local Bio-based Insulation Fiberboards in French Guiana

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Revealing the potential of Guianese waste fibers from timber production and clearings for the development of local and bio-based insulation fiberboards.

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Research Article

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- 6

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20 Abstract

21 In French Guiana, the development of bio-circular value chains to convert residual biomass into insulation 22 fiberboard represents a promising opportunity to build energy-efficient houses. The objectives of this work are 23 to select and characterize resources of interest and discuss the relationships between fiber properties, 24 manufacturing parameters and fiberboards performances. Five local wood and bark residuals fibers are 25 fractionated and fractions are analyzed by laser granulometry, thermal gravimetric analysis (TGA) and pyrolysis 26 combustion flow calorimeter (PCFC). Fiberboards are produced using a thermomechanical process and their 27 microstructure, thermal and sorption properties are characterized by X-ray microtomography, hot plate 28 technique, and dynamic vapor sorption (DVS). Morphological analysis shows that large and elongated wood 29 fractions allow the formation of thick and cohesive fibrous networks, while the bark small-sized fractions are 30 more difficult to process, requiring more compaction and synthetic fibers as additives. However, despite 31 processing difficulties, bark fiberboards show the best insulation performances, comparable to commercial 32 reference products. In addition, TGA and PCFC tests reveal that bark fibers show better thermal stability and fire 33 behavior compared to wood. Finally, they also show a strong affinity for water, highlighting the need for further 34 investigations on their ageing under tropical conditions of use.

35

36 Keywords

37 Tropical biomass valorization, residual fibers, bio-circular economy, insulation fiberboards, microstructure,

- 38 thermal properties.
- 39

40 Graphical abstract



41

42

43 Statement of Novelty

44 This work constitutes the first attempt to explore the potential of Guyanese residuals biomass to produce 45 insulation fiberboards, in alignment with the objective of developing sustainable constructive solutions adapted 46 to tropical regions. The interdisciplinary experimental strategy developed here is innovant and enables discussing 47 the relations between fibers morphology, fiberboards' microstructure and their final use performances. The 48 outcomes of this work will help driving the selection of new resources to be considered for fiberboard production 49 in French Guiana, and help identifying the key processing parameters to be controlled. Our results highlight two 50 promising residual fibers for fiberboard production, evidencing similar to better insulation properties than the 51 ones reported for commercial reference products. This work paves the way towards future research projects.

52 Text

53

54 INTRODUCTION

55 French Guiana is located in the Amazon region, a hot and humid area. To provide comfortable living conditions, 56 the methods and materials used for buildings construction must guaranty good solar protection and regulation 57 of humidity. In the past, local traditional houses were designed using passive architecture methods and durable 58 massive wood elements. Today, because of the extreme demographic growth observed in the region (around 59 120 000 houses would need to be built by 2040, according to the regional environment, planning and housing 60 agencies, DEAL, 2018), the building of traditional houses has been progressively replaced by large-scale 61 construction of less expensive and poorly ventilated western-styled houses. The massive use of air-conditioning 62 used to refresh and dry interior parts represents a large share of domestic energy consumption. To support 63 ecological transition, efficient and affordable biomaterials must be developed to improve houses' energy 64 efficiency and encourage the next generation to change the path towards sustainable housing. Today, most of 65 the insulating products used in French Guiana are imported with high transportation costs, based on fossil 66 sources and non-adapted to high-moisture conditions. A long-term objective of the region is to shift toward local 67 production of bio-based materials for sustainable construction. To do so, the use of wood fibers can be 68 considered as a promising perspective to produce high-performance insulating materials [1]. Indeed, natural 69 fibers exhibit several benefits compared to conventional mineral and synthetic fibers: low density, good specific 70 mechanical properties, non-abrasive properties, low acoustic and thermal conductivity and biodegradability, and 71 ability to maintain hygrothermally stable room conditions thanks to their high hydric inertia [2, 3].

72 Regarding the manufacturing of insulation fiberboards, the non-woven process appears to be a promising 73 technological solution: this technique, adapted from the textile industry, uses a thermo-mechanical process 74 where the cohesion of the fiber mat is mostly ensured by the interlocking of the fibers [4]. It requires light 75 industrial equipment, as well as a small proportion of synthetic fibers, and could be easily developed in French 76 Guiana. With regard to the fiber resources available among the local biodiversity, the preferred option is to 77 consider using residual biomass, in order to avoid further exploration of the Amazonian rainforest. Waste wood 78 fibers resulting from timber production operations are available, abundant and qualitative (species selected and 79 explored for their good technical performances). Two Guyanese commercial species were selected: Dicorynia 80 guianensis and Sextonia rubra, representing 62.4 % of the local timber production (data from the National Forest 81 Office, 2020). In parallel, small-sized trees of fast-growing species are frequently cut during clearings but remain 82 non-valorized. Again, two species have been selected: Cecropia obtusa, the most abundant in French Guiana, 83 also harvested for the extraction of the high-value chemical compounds found in its bark; and 84 Virola surinamensis, which has also been proved to produce a large amount of bark showing well defined fiber 85 bundles [5], expected to be easily isolated during defibering operations.

The general objective of this study was to assess the potential of these Guyanese residual fibers for the production of insulation fiberboards adapted to tropical environment, since the development of such materials

88 still faces important knowledge gaps:

89 - The quality of the tropical species studied here has never been evaluated at fiber scale.

First, it is difficult to anticipate the achievable quality of the milled fractions. The selected wood and bark specimens are characterized by different anatomical microstructure (cells type, shape and organization), densities and chemical composition, which are expected to influence the final particles size distribution and morphology of the fractions after milling. Moreover, we have no information about the thermal properties and reaction to fire of these materials, which is of prior interest when it comes to insulating materials intended to be used for building and construction.

- 96
- The manufacturing process of fiberboards has never been adapted to tropical resources.

97 Previous works have been conducted with wood fibers from pine, poplar and eucalyptus of 2 mm length [6], but 98 tropical wood and bark fibers characterized by different structures and morphology have never been tested. The 99 process parameters need to be optimized, since the specificities of tropical resources might influence their 100 conversion into fiberboards.

101 - The relations between fiber characteristics and fiberboard properties are understudied.

Since no glue was used during the production of fiberboards, thermal conductivity is expected to depend on the intrinsic properties of the fibers, as well as the resulting microstructure and porosity distribution within the fiber mat [7]. This raises the question of which is the most important parameter to be taken into account when selecting quality fibers for the production of insulation fiberboards: What is the effect of fiber morphological features on microstructure and porosity of fiberboards? Does the microstructure dominate over the intrinsic insulating properties of the fibers?

108 The specific goals of the present study were therefore to (i) characterize the morphological and thermal 109 properties of the selected tropical waste fibers and particles obtained after milling; (ii) optimize the production 110 process for each resource; (iii) discuss the microstructure-properties relationships; (iv) report on the final 111 achievable insulation performance and water affinity of the produced fiberboards, and (v) propose solutions for 112 process improvement.

- 113 This work constitutes, to our knowledge, the first attempt to study the thermal properties of Guyanese waste
- $114 \qquad {\rm fibers \ and \ their \ suitability \ for \ the \ production \ of \ insulation \ fiberboards}.$
- 115

116 MATERIAL AND METHODS

117 Selection of resources

118 For commercial wood species Dicorynia guianensis Amsh. (D. guianensis) and Sextonia rubra (Mez) van der Werff

119 (S. rubra), a residual heartwood board resulting from sawing operations in the laboratory was selected. For fast-

120 growing species, wood and barks fibers from *Cecropia obtusa* Trécul (*C. obtusa*; Bark *C. obtusa*) and bark fibers

121 from Virola surinamensis (Rol. ex Rottb.) Warb. (Bark V. surinamensis) were collected on young specimens (5 m

height, approximately 2 years-old) harvested in a secondary forest near the Paracou experimental station (5°16'27N; 52°55'26W).

124 Our final sampling thus consisted in 3 wood samples and 2 bark samples that appeared to be promising resources

125 to be considered for the development of a local sustainable value chain to produce insulation fiberboards

126 (Figure 1). The main characteristics of each wood species, based on past surveys on Guyanese wood properties

- 127 (Gérard et al. 2016; Gérard et al. 2019) are given in Table 1. No information could be found in the literature for
- 128 the studied barks.
- 129



131Figure 1. Sampling strategy. Wood fibers were collected from *D. guianensis*, *S. rubra* and *C. obtusa*. Bark fibers were collected from *C. obtusa*132and *V. surinamensis*. Fibers were oven-dried at 60°C for 3 days and milled into fine powder using an impact mil SM 300 (1.5 kg/h and 2000 rpm133with 4 mm then 2 mm grid). Photos credits: *D. guianensis*: Romain Lehnebach; *S. rubra*: Emeline Houël; *C. obtusa*: Yves Caraglio;134*V. surinamensis*: Inventaire National du Patrimoine Naturel.

135

136Table 1. Wood physical and chemical characteristics based on past surveys on Guyanese wood properties (Gérard et al. 2016; Gérard et al.1372019). N: number of tested specimens; D12: wood density at 12% of relative humidity; FSP: Fiber Saturation Point. Standard deviation is

- 138 given in brackets. Cellulose and lignin contents were measured by the Kürschner and Klason methods respectively. Extractives were extracted
- 139 in water. *Database reports measurement performed on *Cecropia* genus without specifying the studied species.
- 140

	Physical properties				Biochemical composition					
SPECIES	N	D12	FSP (%)	N	Cellulose (%)	Lignin (%)	Extractives (%)	Ashes (%)		
D. guianensis	90	0.79 (0.05)	28.6 (2.3)	7	45.5 <i>(2.0)</i>	35.9 (2.1)	4.4 (1.5)	0.7 (0.3)		
S. rubra	28	0.66 (0.04)	29.4 (2.8)	3	47.8 (2.7)	31.7 (3.3)	9.1 (2.7)	0.4 (0.2)		
Cecropia sp*	6	0.32 (0.06)	40.7 (3.7)	2	52.5	23.3 (1.2)	4.8 (1.5)	1.2 (0.3)		

141

142 Fibers' production and characterization

- 143 Fibers' production
- 144 In previous studies on softwood, fiberboards were successfully manufactured from fibers with an average length
- 145 of 1.6 mm [8]. To achieve such dimensions, an impact mill has been used. This technology enables to reach a
- satisfying comminution of ligno-cellulosic materials while limiting energy consumption [9]. More than 2 kg of
- 147 material were collected for each resource. Samples were manually cut into chips and air-dried in the laboratory
- 148 during one month. Chips were first oven-dried at 60°C for 3 days to avoid caking during grinding. Thereafter,

chips were fractionated into fine powder (Figure 1) using an impact milling system (SM 300, Retsch) in continuous
 mode, at 1.5 kg/h and 2000 rpm; two fractionation steps were carried out, using 4 mm and 2 mm grids
 successively. A final dry mass of 1.3 kg per resource was produced.

152

153 Morphological analysis

The morphological analysis has been performed on the PLANET platform (UMR IATE, Montpellier). The particle size distributions of the studied fractions were analyzed with a laser granulometer (LS 13 320 XR, Beckman Coulter) in dry mode. Five replicates of 5 g per material were tested, showing good reproducibility.

Mean diameter (d50) and specific surface area (SSA) were determined for each sample. In addition, the milled fractions were automatically air-sprayed onto a glass slide and analyzed using a particles shape analyzer (Morphologi 4, Malvern). High-definition images of each isolated particle were recorded, providing reliable and accurate data for the determination of particles' shape parameters (circularity and elongation). A mean number of 47 727 particles were analyzed for each sample.

162

163 Thermal Gravimetric Analysis (TGA)

164 TG experiments have been carried out to study the thermal behavior of the milled fibers during pyrolysis under 165 oxygen atmosphere, using a Mettler TGA2 apparatus (Schwerzebbach, Switzerland) equipped with a XP5U 166 balance. About 10 mg of each sample was heated from room temperature to 500 °C under air flow (50 mL.min⁻¹ 167 ¹) at a heating rate of 10 °C.min⁻¹. Onset temperature (T_{onset}) was computed as the intersection point of the 168 extrapolated baseline and the inflectional tangent at the beginning of the degradation peak. The peak 169 degradation temperatures (T_{peak}) were taken at the maximum values of the mass-loss derivative curve. Analysis 170 was done in duplicate for each sample with good reproducibility.

171

172 Pyrolysis combustion flow calorimeter

173 Tests were carried out using a Fire Testing Technology microcalorimeter. 2 (±1) mg of sample was heated under 174 nitrogen flow up to 750 °C at a heating rate of 1 °C/s. The released gases were mixed with oxygen ($N_2/O_2 80/20$) 175 and burned into a combustion chamber at 900 °C. Under these conditions, the combustion is guaranteed to be 176 complete. Heat release rate (HRR) was calculated from the oxygen consumption according to Huggett's relation 177 [21] (1 kg of consumed oxygen corresponds to 13.1 MJ of heat release). Each test was performed twice. The 178 quantity of char, the peak of heat release rate (pHRR) and corresponding temperature (T(pHRR)), the total heat 179 release (THR), and the heat of complete combustion (Δ H, calculated as the ratio between THR and mass loss) 180 were determined.

181

182 Fiberboard's processing and characterization

183 Non-woven fiberboards' processing

184 This first step was carried out using an air-laid machine from Laroche (Figure 2). Firstly, wood/bark fractions were

185 carded with a rotating spiked roller (1100 to 2300 rpm), mixed with 6 mm-length synthetic bicomponent fibers

(polyester/polypropylene, adjustable quantity around 10% of addition per mass of natural fibers) and blown (at 0.5 to 2 m.min⁻¹) onto a vacuum perforated drum. Fibers in air-laid webs tended to be isotropically oriented in horizontal plane. Mat consistency was modulated by the stretching ratio (speed of entry delivery roller / speed of exit pressure rollers; adjustable from 0.5 to 2). Machine settings were adjusted in order to obtain a homogeneous fiber mat in mass and thickness. The formed fiber mats were then superimposed to form a fiberboard. At this stage, the cohesion of the fiberboard was only ensured by the entanglement of fibers.



- 193
- 194 195

196

Figure 2. Processing equipment used for 3D fiberboard formation: napper opener machine from Laroche.

- 197 Non-woven fiberboard consolidation
- 198 This second processing step was carried out using a hot-air oven from Strahm (Figure 3). This is a dry bonding
- 199 process that takes advantage of the thermoplastic properties of synthetic fibers. This thermal post-treatment
- 200 results in very low density non-woven fibrous structures with good cohesion. The operating temperature for
- 201 melting the thermoplastic fiber sheath has been set at 160°C.



- 202 203
- Figure 3. Processing equipment used for insulation fiberboard consolidation: a) hot air oven from Strahm b) fiberboard at the entrance of the oven c) fiberboard in the cooling part of the oven.
- 204 205
- 206 Fiberboards' characterization
- 207 3D microstructure

The internal microstructure of the fiberboards was characterized through a non-destructive imaging technique, using a SkyScan X-ray microtomograph. First, a small cylindrical sample of 9-mm diameter and 10-mm height is carefully extracted from the initial fiberboards. The X-ray beam passes through the sample and the signal transmitted is recorded by CCD sensors. After 3D reconstruction of X-ray projections, a subvolume is extracted from the grey level 3D images to remove borders and to avoid any boundary effect, induced both by the cutting of the sample and the X-Ray conic source. The size of the pores of a set of wood/bark fibers was measured by performing successive openings of growing size on the virtual volume. The 3D images resolution was 6.9 microns/voxel and the analyzed volume was 5.5 x 5.5 x 5.5 mm³.

216

217 Insulation properties

Insulation properties of the fiberboards were measured on 100 x 100 x *e* mm³ samples (with *e* the thickness of the fiberboard, samples stabilized at 20 °C and 65 % of relative humidity.) by the hot plate technique, with a Desprotherm device (Epsilon Alcen). The thermal imbalance of the sample was induced by providing a uniform and unidirectional heat flow on one of its isolated faces. The apparent thermal conductivity λ^*_{app} (W.m⁻¹.K⁻¹) of the material could be calculated by assessing its equivalent thermal effusivity *b* (W.K⁻¹.m⁻².s^{1/2}) and its bulk volumetric heat capacity $\rho^*.C_p$ (W·m⁻³·K⁻¹), where ρ^* (*kg* m⁻³) is the bulk density and C_p (W kg⁻¹·K⁻¹) is the specific heat capacity (*Equation 1*). Measurements were done in triplicate.

$$\lambda_{app}^{*} = \frac{b^{2}}{\rho^{*} \cdot C_{p}}$$
 (Equation 1)

225

226

227 Dynamic Vapor Sorption (DVS)

228 Sorption measurements were performed by dynamic vapor sorption (DVS), using a gravimetric sorption 229 instrument (Service Solution Limited, UK, London). Each fiberboard specimen (10 +/-2mg) was air-dried during 230 two weeks prior loading in the instrument at RH = 0%. Mass changes were constantly recorded along five 231 different relative humidity stages (RH = 0%, 25%, 50%, 75% and 90%). Stabilized mass was recorded after reaching 232 a mass variation ratio dm/dt below 0.001 %.min⁻¹, allowing the calculation of the moisture content (MC). These 233 conditions have been shown to be adequate for reaching constant mass uptake at each step of water vapor 234 sorption for wood and biomaterials [10, 11]. The specific mass stabilization time (SMST_{75>90%RH}), was calculated 235 as the time per mass unit necessary to reach equilibrium after a change in RH from 75% to 90% (Equation 2).

$$SMST_{75>90\%RH} = \frac{mass \, stabilization \, time \, from \, RH=75\% \, to \, 90\%}{Stabilized \, mass_{RH=0\%}}$$

(Equation 2)

238 239

240 **RESULTS AND DISCUSSION**

241 Morphological and thermal properties of wood and bark fractions

Morphological analysis

243 Considering all specimens, morphological characterization of the milled fractions by laser granulometry revealed 244 that most of the ground particles were much smaller than 2 mm (average d₅₀ of 0.56 mm for all tested fractions) 245 (Figure 4). Indeed, many authors have already reported particle sizes much smaller than the grid opening after

impact milling operations. Using a 2 mm grid and a similar milling protocol to the one used in this study, Mayer-

Laigle et al. (2018), David (2019) and Rajaonarivony et al. (2019) all observed average particle sizes below 0.7 mm,

248 using wheat straw, vine shoots and pine barks fibers, respectively. It thus appears reasonable in our case,

249 studying short-length tropical hardwood fibers, to obtain such dimensions.

250 At the inter-specific level, it can be noticed that identical milling conditions resulted in fractions of variable size 251 distributions depending on the species (Figure 4 and Table 2). All wood specimens showed similar size 252 distributions ($d_{50} = 0.73$ mm +/-0.051 mm), whereas milling bark specimens resulted in smaller particles 253 (d₅₀ = 0.42 and 0.21 mm for Bark V. surinamensis and Bark C. obtusa, respectively). Since no subsequent sieving 254 operation was conducted after milling, a large amount of fine particles has thus been obtained for bark 255 specimens as illustrated in Figure 4. Maharani et al. (2010) studied the effect of different mill types on five 256 different tropical species. They showed that impact milling leads to the highest content of fine particles under 257 0.3 mm (around 71.8%, in volume). We used a similar classification to sort the different fractions produced in 258 this study (oversized particles > 0.7 mm; 0.7 mm < coarse particles < 0.3 mm; fines particles < 0.3 mm). The 259 results obtained showed that there is a large amount of coarse and fine particles in all fractions (minimum 50 % 260 in volume), and confirmed the existence of maximal amounts of fine particles for the bark specimens (up to 261 66.8 % in volume for Bark C. obtusa and 40.3 % in volume for Bark V. surinamensis) (Figure 5). Most likely, the 262 small dimensions obtained for both bark specimens indicate their lower resistance to impact milling and their 263 tendency to break into shorter length fractions compared to wood fibers.

264



265

266 Figure 4. Particle size distribution of the milled fractions for each tested material measured by laser granulometry (LS 13 320 XR, Beckman

Coulter).

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 $\bar{2}\bar{7}\bar{3}$

Figure 5. Representation of the particle size distribution of wood and bark powder samples over three dimensional categories: oversized (OS) > 700 μ m; coarse particle size (CPS) : [300-700 μ m]; fine particle size (FPS) : [100-300 μ m].

Optical observations of air-sprayed fractions confirmed the size distributions previously obtained, showing larger and lengthen particles for wood samples compared to bark samples (Figure 6). In addition, this method allows a better characterization of the particle aspect ratio, revealing that *S. rubra* and *C. obtusa* wood fractions were composed of a larger share of elongated elements, while *D. guianensis,* and bark fibers even more extensively, displayed smaller and circular elements (Table 2).

279 The fractions produced in this study are therefore quite dissimilar in terms of morphology, but representative of 280 the achievable quality that can be obtained using raw residual biomass from the wood industry sector, which is 281 the focus of this study. However, the milling process could be optimized if willing to attain a specific particle size. 282 For example, the initial drying step could be reduced since it has been shown to negatively impact fibers' 283 mechanical properties and load-bearing capacities during milling by limiting the plasticizing effect of water, 284 resulting in the generation of a larger share of fine particles [9]. Also, the rotation speed and the milling time 285 could be reduced to limit the degradation of the fibers. Thereafter, subsequent sieving operations could be 286 applied to select a specific particle size class while maintaining satisfactory milling yields. It would also be possible 287 to use another fiber separation method such as defibering, but such methods require heavy technical 288 installations which might be difficult to implement in French Guiana for the foreseeable future. 289 The heterogeneous morphological characteristics of the fractions described in this section are expected to

influence both the processing and the performances of the fiberboards. Specifically, while reduced fibers' lengthmay complicate fiberboards shaping, it has been found that fine particles promote desirable microstructuration

292 of the fiberboards and thus lead to interesting thermal properties (Vignon, 2020).



299

Figure 6 – Optical observations of the milled fibers air-sprayed on a glass plate using a particles shape analyzer (Morphologi 4, Malvern)

Table 2. Results of the morphological analysis of wood and bark fractions. N: number of detected elements; d₅₀: mean diameter; Elongation; Circularity; CV%: coefficient of variation.

Material	N	d ₅₀ (mm)	Elongation	Circularity	CV%
D. guianensis	67235	0.70	0.62	0.92	10.8
S. rubra	219852	0.69	0.72	0.86	3.4
C. obtusa	183148	0.79	0.70	0.89	6.7
Bark C. obtusa	320135	0.21	0.53	0.91	4.9
Bark V. surinamensis	118443	0.42	0.53	0.93	3.4

300

301 • Thermal analysis

Fiber samples were analyzed by thermal gravimetric analysis (TGA) to study their thermal decomposition kinetics
 (from room temperature up to 600°C at 10 °C.min⁻¹, under oxygen gas flow), and by Pyrolysis Combustion Flow
 Calorimeter (PCFC) (anaerobic pyrolysis and complete combustion) to characterize their flammability. Results
 are presented in Figure 7 and 8 and the characteristic values are reported in Table 3.

306 In this work, the thermal degradation was studied under aerobic pyrolysis conditions. Compared to anaerobic 307 pyrolysis, far fewer studies have been published on biomass combustion. Indeed, thermal degradation under 308 oxidative atmosphere is more complex, considering that the presence of an oxidizing agent (air, oxygen, etc.) generates reactions between oxygen and solid reactants [16-18]. All fractions started losing weight at about 309 310 200 °C (i.e., 40 °C above the temperature used for fiberboards processing) and their degradation behavior was a 311 complex process, composed of two apparent degradation stages: the first rapid weight loss corresponding to 312 pyrolysis or devolatilization stage (Stage I. on Figure 7); then secondary weight loss corresponding to char 313 oxidation due to the presence of oxygen (Stage II. on Figure 7) [19]. Under nitrogen flow, hemicelluloses and 314 cellulose pyrolysis occurs around 295 °C and 370 °C, respectively [20]. The decomposition of lignin is slower and 315 more complex since lignin fractions have different stabilities. This component thus covers a wider range of 316 temperature from 200 °C to 450 °C under nitrogen [21]. In presence of oxygen, the devolatilization peaks appear 317 at lower temperature (-15 °C and -35 °C for hemicelluloses and cellulose, respectively) [22]. As a result, specific 318 degradation peaks of hemicelluloses and cellulose are expected around 280 °C and 340 °C in aerobic conditions 319 (peaks (a) and (b) on Figure 7).

320 These values match the double peaks observed during pyrolysis of wood fractions D. guianensis and S. rubra. 321 Both species showed similar behavior with almost identical cellulose degradation peaks (at 334 °C and 332 °C 322 respectively, with identical intensity), likely because of their close chemical composition (Table 1). In contrast, 323 pyrolysis of hemicelluloses and cellulose in C. obtusa wood fraction was more pronounced, and the cellulose 324 degradation peak appeared at lower temperature (around 310 °C). This could possibly originates from its lower 325 lignin content (around 23%, Table 1), which has been proved to decompose slowly during pyrolysis [23]. Char 326 oxidation peak also occurred at lower temperature and was really marked, confirming its lower thermal stability 327 compared to other samples. However, it has also been proved that the combination of a low content of lignin 328 with a high content of cellulose, as it is the case for C. obtusa, could lead to charring and to incomplete 329 combustion of these fibers, limiting their contribution to the heat evolved during burning.

Regarding bark samples, thermal stability is significantly higher compared to wood samples. Indeed, for the same species *C. obtusa*, degradation peaks were much less pronounced and shifted to higher temperatures for bark samples (around 320 °C), indicating that a higher activation energy is needed to decompose woody biomass, which could be the result of the presence of a higher amount and different type of lignin compared to wood [23].

- 334 *Bark V. surinamensis* showed the best thermal stability over the studied temperature range.
- 335

PCFC tests were also conducted to study the fire reaction of the studied fractions. Measured peak values (pHRR) are similar to the ones found in the literature for wood [24]. The heat release rates (HRR) versus temperature curves (Figure 8), confirmed that wood fibers have similar thermal behavior. The only difference between wood fibers concerned the temperature peak: T(pHRR) was observed to be lower for *C. obtusa*.

340 Bark samples released markedly less energy than woods, with pHRR equals to 71 W/g (compared to 147 W/g on 341 average for wood fractions) and THR equal to approximatively 7 kJ/g (compared to 11.6 kJ/g on average for wood 342 fractions). They also generate more char during their decomposition (750 °C): around 25% char (compared to 343 16.2% on average for wood fractions), which is desired since it enhances the fire resistance of materials: the 344 material which forms carbonaceous char has a reduced ability to supply the gaseous fuels required to fuel the 345 fire. ΔH represents the intrinsic energy of the release gases during combustion, and lower values were obtained 346 for bark fractions. The higher quantity of chars formed from bark can explain this result, since chars store high 347 quantities of carbon, which tend to reduce the energy of gases.

348

Bark fractions thus revealed better thermal stability and fire reaction compared to wood, which could originate from higher rates of extractives and/or lignin, which are associated with a high carbon content and result in higher yields of char [22]. Indeed the presence of specific chemical components such as suberin which contribute to shielding against heat-induced degradation [25] could also be responsible of the observed differences.

- 353
- 354

Table 3. Specific degradation temperatures determined from DTG thermograms.

	TGA measurements		Microcalorimeter measurements				
	T _{50%} (°C)	T _{peak} (°C)	Char (%)	pHRR (W/g)	T(pHRR) (°C)	THR (kJ/g)	ΔH (kJ/g)
D. guianensis wood	347	334; 481	18.32	154	372	11.1	13.5

S. rubra	342	332; 465	15.55	146	363.5	12.7	15.0
C. obtusa	314	313; 406	14.79	141	341	11.1	13.0
Bark C. obtusa	352	325; 438	25.24	67.5	354.5	7.1	9.4
Bark V. surinamensis	395	315; 438	24.83	74	354	7	9.3





Figure 7. Results of the thermal analysis (TGA) performed on the studied fractions. Mass variations (top) and its derivative (bottom) during heating under air flux at 10°C.min⁻¹ up to 500°C. Dotted lines (a) and (b) represent specific the specific degradation peaks of hemicelluloses and cellulose in oxidative conditions. Lignin degrades over a wider temperature range, from 350°C to 450°C.
 360



Figure 8. Results of the PCFC tests conducted on the studied fractions. The evolution of the heat release rates (HRR) is shown as a function of the pyrolysis temperature.

367 Fiberboard manufacturing and microstructure: effect of wood and bark fractions' morphology

368 Uniformity of air-laid webs depends on the degree of opening of the fibers, the proportion of synthetic fibers 369 and the airflow profile. Processing parameters were first set based on previous works conducted with pine, 370 poplar and eucalyptus fiber fractions of about 2 mm in length [6]. Using the same protocol with the small-sized 371 fractions studied in this work, we first observed a severe mass loss during the web formation step (more than 372 80%) and a bad cohesion of the resulting fiber mats. Changes in the processing parameters have thus been 373 operated. Firstly, to limit mass loss due to the aspiration of fine particles, air suction has been halved from 50 to 374 25%. Secondly, the initial opening step on the carding machine has been eliminated since no fiber tufts could be 375 observed and because controlling fiber orientation was not possible for such small dimensions. On the contrary, 376 opening step for synthetic fibers was enhanced (two successive opening steps were operated), to facilitate their 377 mixing with lignocellulosic fibers and improve mat cohesion. Finally, the stretching ratio was decreased from 2 378 to 0.5 in order to increase the compaction of the mat and improve its cohesion. Thanks to these adjustments, 379 average mass losses were greatly reduced (Table 4). However, the final results were still higher than the values 380 reported by [15] for hardwood species (25% and 28% for poplar and eucalyptus respectively). It is clear that even 381 after optimizing the process, the large share of fine particles present in the studied fractions remains an issue: 382 the suction of the smallest particles leads to high mass losses. 383

As a result, a higher rate of synthetic fibers was needed to reach satisfying mat cohesion (from 8% for C. obtusa 384 to 17% for Bark C. obtusa, compared to 6% for the reference pine fibers of 2 mm length, according to Vignon 385 (2020)). Here, a strong negative correlation (Pearson's r = -0.99; p-value < 0.01) was found between fiber particle 386 size (d_{50}) and the amount of synthetic fibers needed to reach satisfying cohesion (Figure 9a). As a result, the bark 387 fibers-based fiberboards were composed of a higher proportion of synthetic fibers and more compact with 388 thicknesses from 15 to 18 mm (Table 4 and Figure 10), which is expected to lower their insulation performances. 389 C. obtusa and S. rubra fiberboards were the easiest to manufacture and resulted in the thickest mats (up to 390 30 mm) produced in this study (Table 4 and Figure 10). Comparing the morphological features of the fibers and 391 the resulting thickness of the mats, we found a significant positive correlation (Pearson's r = 0.92; p-value < 0.01), 392 with greater fibers elongation leading to greater mat thickness due to better cohesion (Figure 9b). Indeed, many 393 researches have already reported that increasing fiber elongation is an efficient strategy to enhance the 394 mechanical performances of paper and bio-composite materials. Fiber inter-bonding is favored, allowing the 395 formation of a cohesive 3D interlocked fiber network [26, 27].

396 In summary, besides controlling the amount of fine particles, our results also showed that fiber elongation is a 397 key morphological parameter to be considered to enhance the manufacturing and structuration of fiberboards. 398 Besides morphological properties, the chemical composition of the fractions can also influence the 399 microstructuration of the fiberboards during the heating phase and their adherence to synthetic fibers. Indeed, 400 the chemical reactions and physical consolidation of the fibers induced by applied heat and pressure can favor 401 self-bonding reactions in binderless fiberboards [28]. In particular, fibers with lignin-rich surfaces might fuse 402 together as the softened lignin molecules flow from one fiber surface to another one, and possibly form covalent 403 bonds [29]. In the case of the studied wood and bark fractions, a detailed analysis of their chemical composition

404 will be necessary to better understand the mechanism at play.

410 consolidated at 160°C.

Material	N	Synthetic fibers Rate (%)	Mass (g)	Loss rate (%)	Thickness (mm)	Apparent density (kg.m ⁻³)
D. guianensis	4	10	401	53	22.0	203
S. rubra	4	10	500	38	25.5	267
C. obtusa	5	8	382	43	30.0	166
Bark C. obtusa	3	17	200	72	18.0	141
Bark V. surinamensis	3	15	293	66	15.2	202

411





413

Figure 9. Influence of fiber size (a) and elongation (b) on the processing of fiberboards in terms of final thickness and synthetic fiber rate.
 Points stands for wood fiberboards (red: *D. guianensis*; black: *S. rubra*; blue: *C. obtusa*) and triangles for bark fiberboards (blue: Bark
 C. obtusa; green: Bark *V. surinamensis*); r : correlation coefficient of Pearson; **: p-value < 0.01.



- 418 419
- 420

Figure 10. Final aspect of the produced insulation fiberboards from each wood/bark fraction.

The internal microstructure of the fiberboards produced from the two most contrasted fibers fractions in terms of morphological characteristics, i.e., *C. obtusa* (d₅₀: 0.79 mm; Elongation: 0.70) and Bark *C. obtusa* (d₅₀: 0.21 mm; Elongation: 0.53), was studied using X-ray microtomography. 3D volume elements were reconstructed from successive X-ray projections (Figure 11a). After cleaning and thresholding, successive openings by spheres of growing size were performed on the 3D images (Tran et al. 2013; Délisée et al., 2017; Hamdi et al. 2020). This analysis enabled the determination of pore size distribution within the fiberboard thickness (Figure 11b). X-ray
microtomography images confirmed that a larger share of large-sized and elongated particles can be observed
in *C. obtusa* compared to Bark *C. obtusa*. As a result, these two fiberboards were characterized by different
apparent densities (166 and 141 kg.m⁻³ for *C. obtusa* and Bark *C. obtusa* fiberboards respectively, Table 4).
However, probably because of the large share of FPs observed in bark fractions, pore size distributions were
similar for both fiberboards, with slightly larger pores observed for Bark *C. obtusa* (average distance between
two fibers of 323 µm compared to 297 µm for wood; total porosity: 75% for wood and 76% for bark) (Figure 11b).

433



434

435Figure 11. a) 3D volume elements reconstructed from successive X-ray projections, corresponding to the fiberboards made436of C. obtusa (left) and Bark C. obtusa (right); P: porosity. b) Pore size distribution computed from the 3D volume elements.

437

438 Evaluation of fiberboards' performances

439 Insulation performances

440 The thermal conductivity of the fiberboards produced in this study is illustrated in Figure 12, and compared to 441 the commercial reference fiberboards made from pinewood (Pinus pinaster) (orange squares on Figure 12). For 442 the commercial reference, testing samples of varying densities has shown that it exists an optimal density to 443 achieve the lowest thermal conductivity (0.045 W.m⁻¹.K⁻¹ for a fiberboard density of 60 kg.m⁻³ in this case) 444 (Vignon, 2020). Because of the small size and low elongation of the fibers used in this work, compaction of the 445 fiberboards was increased to obtain suitable cohesion of the mats. As a result, the density of the fiberboards 446 produced in this study ranged between 141.9 and 267.7 kg.m⁻³, hence corresponding to the upper range of the 447 pinewood reference curve. Therefore, our results might not cover the optimum thermal conductivity / fiberboard 448 density ratio reachable for each fraction. Nevertheless, without any optimization of the fiber morphological 449 characteristics, the fiberboards produced in this study from tropical wood and bark have a thermal conductivity 450 close to or even lower than those reported for the commercial reference considering the same density range 451 (Figure 12). These results are promising and it is reasonable to assume that highly efficient insulating material 452 could be produced from these resources with further optimization of fiber preparation and fiberboard 453 manufacturing.

It is interesting to note that contrasting results were observed depending on the fibers used. *S. rubra and D. guianensis* wood-based fiberboards offer medium to high thermal conductivities, respectively, ranging from
0.04 W.m⁻¹.K⁻¹ to 0.1 W.m⁻¹.K⁻¹. Quite high scattering of thermal conductivity values was noticed for these

457 samples, which might originate from the existing structural and chemical tree natural variability, already reported 458 by several authors for these two species [32–34]. The natural variability of the fractions will be unavoidable, and 459 must be considered in order to evaluate realistic final properties of the fiberboards produced from residual fibers. 460 On the contrary, fiberboards produced from bark fractions showed the best and less scattered results, with 461 equivalent to higher insulating performances compared to the commercial reference in the same density range. 462 Belley (2009) suggested that mat density, fiber size and pore sizes mostly influence the thermal conductivity of 463 fiber mats. Here, the small-sized bark particles could contribute to the creation of a specific pore network, thus 464 promoting better insulation performance. Interestingly, for the C. obtusa species, a clear difference can be 465 observed between fiberboards produced from wood or bark fractions. Indeed, despite easy processing and 466 promising microstructuration, C. obtusa wood showed the poorest thermal performances, i.e., high thermal 467 conductivity around 0.1 W.m⁻¹.K⁻¹.

468 Since no glue was used during the production of fiberboards (and given that synthetic fibers are not supposed to 469 influence the material's thermal properties), and samples were stabilized to the same water content prior 470 testing, thermal conductivity should only depend on the intrinsic properties of natural fibers used for the 471 manufacturing of the boards, as well as the resulting microstructure of fiberboards, their overall porosity and 472 pore size distribution [7, 36]. X-ray tomography confirmed that pore size distribution and porosity were similar 473 for fiberboards made from wood and bark fibers of C. obtusa, with only slightly larger pores in bark-based 474 fiberboards. This suggests that the good thermal conductivities obtained for fiberboards produced from bark 475 fibers could be related to the good intrinsic thermal properties of the fibers. This assumption seems consistent 476 with the fact that bark fibers naturally display protecting functions for the tree, including thermal insulation to 477 resist to climate variations (T °C and RH). But further investigations are needed to better understand the 478 insulating performance of bark-based fiberboards in relation to their microstructure.





481 Figure 12. Evolution of the thermal conductivity of fiberboards as a function of their density at 12% of moisture content.

In summary, barks fibers from *C. obtusa* and *V. surinamensis* appear to be promising resources to produce insulation fiberboards, and wood fibers form *S. rubra* and *D. guianensis* show encouraging results. After improving the milling production step, to better control particle size and elongation and resulting fiberboards microstructure, even better insulating performance could be achieved. These results also raised an important point regarding the selection of the fractions. We observed that besides correct processing of the mats, the microporous network structure of the mat and the specific properties of the selected fibers are critical for insulating performance.

490

491 • Water affinity

Finally, since the produced fiberboards are intended to be used for the insulation of local habitations, in a humid environment, their sensitivity to water need to be investigated. In French Guiana, relative humidity (RH) is high (ranging from 58% to 73%). Also, for outdoor applications, this moisture range is relevant since degradation by wood-decaying fungi occurs at high humidity levels. Affinity to water at high RH and behavior over several RH cycles is thus a key parameter to rule on the fiberboards' quality as insulating material under tropical conditions.

498 Figure 13 displays the obtained sorption isotherms for each fiberboard sample, where the stabilized values of 499 moisture content (MC) are represented along five different RH stages for wood (solid lines) and bark fibers 500 (dotted lines). The obtained curves superimposed at low RH stages but clearly differentiated at higher RH. 501 Fiberboards produced from wood fibers showed lower maximal water uptake compared to the pinewood 502 reference (at 90% RH: MC = 11.84% for S. rubra, 13.02% for D. guianensis, 14.02% for C. obtusa and 18% for the 503 pinewood reference). Fiberboards produced from Bark C. obtusa and Bark V. surinamensis showed the highest 504 values of water uptake at 90% RH (MC = 18.70% and 15.15%, respectively). Samples made from Bark C. obtusa 505 showed a behavior very close to that of the reference pinewood fiberboard: MC already increases significantly 506 at low RH stages (being in average more than 30% higher compared to other samples at 50% RH). Like the 507 commercial pinewood reference, Bark C. obtusa thus showed a marked affinity to water compared to other 508 studied species, which might be due to a high content of hemicellulose or low content of extractives. 509



511 Figure 13. Sorption isotherms obtained for each fiberboard sample, using DVS. Wood species are presented in solid lines 512 while bark species are represented in dotted lines. The influence of relative humidity on moisture content is represented at 513 each RH stage. Stabilized moisture content values consist in the quantity of water sorbed by the samples after reaching the 514 equilibrium criteria of dm/dt < 0.001%.

515

516 In addition to the stabilized values (MC), it is also interesting to study the kinetics of moisture sorption since great 517 humidity fluctuations can be observed in French Guiana at day-scale between mid-day (59%RH at 11:00am) and 518 night (92%RH at 05:00am). The specific mass stabilization time (SMST, corresponding to the time per unit of mass 519 necessary to reach a mass equilibrium after a change in RH from 75% to 90%) is reported for each sample on 520 Figure 14. For fiberboards made from wood fibers, variations in SMST seems to be related to variations in fiber 521 density and extractive content (see Table 1). Indeed, sorption mechanisms could be influence by the 522 hydrophilicity of extractives and fiber cell wall microporosity, making the sorption sites more or less accessible 523 [37, 38].

524

525 Compared to wood fibers, bark fibers (i) absorb a larger amount of water at high RH (Figure 13) but (ii) need a 526 longer time to reach saturation (highest values of SMST). Because of their strong affinity for water, bark 527 fiberboards might fluctuate in the critical range of moisture content favorable for fungal degradation. It thus 528 points out the need of investigating their durability in service-use conditions. However, to protect the tree 529 against pests and diseases, bark generally contains more extractives than wood and is expected to be naturally 530 durable. For example, Morris, Grace, and Troughton (1999) reported good natural durability for a board material 531 made from pressed spruce bark with no additives. As evidenced for oak wood, the inhibiting effect of tannins 532 and suberin on fungal growth could bring a higher resistance to the bark than to the wood [40]. Bark extracts are 533 even considered for developing new bio-based preservative treatments to improve the biological resistance of 534 wood [41]. Finally, fiberboards produced from bark fibers might also be of interest for humidity regulation: they 535 might act as water buffers since water sorption kinetics is slower compared to wood. 536 These hypotheses must be verified through long-term and RH cycling experiments. A perspective of this work

537 would be to conduct tests on the produced fiberboards in real service-use conditions to assess (i) their durability

538 and (ii) their insulating performance under fluctuating humidity conditions.

539





544 **CONCLUSION**

545 This study was a first basis of prospection to evaluate the potential of Guyanese waste fibers for the production

546 of insulation fiberboards. Wood residues from two of the most common local commercial species (*D. guianensis*

547 and S. rubra) and from an abundant fast-growing species (C. obtusa), as well as bark fibers of interest (Bark C.

548 *obtusa* and Bark *V. surinamensis*), were selected and harvested to manufacture bio-based insulation fiberboards.

- 549 Fiberboards were produced from milled fiber fractions according to an environmentally friendly process using
- the self-bonding properties of the fibers and a small fraction of synthetic fibers.
- 551 The morphological analysis operated on the milled fractions highlighted the need for an optimization of the 552 milling process to achieve suitable fiber/particle size and shape. Regarding fiberboards production, adjustments 553 had to be made according to the specific morphology of the obtained fractions. Processing parameters were 554 optimized to limit mass losses and favor mat cohesion. Our results showed that fibers shape is a key 555 morphological parameter to be considered for fiberboards manufacturing process. It appeared to positively 556 influence self-bonding of the fibers, improving fiberboards cohesion. However, in addition to the proper 557 manufacturing of the fiber mats, the specific properties of the selected fibers are likely to influence the 558 performances of the fiberboards at first order.
- 559 Regarding final thermal insulation performance, despite no optimization of the fractions' dimensions and 560 fiberboard densities, the fiberboards made from tropical wood and bark residues displayed quite interesting low 561 thermal conductivities. Some of the selected residual fiber resources even enabled the production of fiberboards 562 with performances comparable to those reported for the commercial reference. Bark fractions of C. obtusa and 563 V. surinamensis appeared to be really promising materials and wood fractions of S. rubra and D. guianensis gave 564 encouraging results. Moreover, bark fibers displayed the best resistance to thermal degradation and fire 565 reaction, two parameters of interest for building applications. On the other hand, bark fibers were also 566 characterized by higher affinity to water compared to wood fibers, showing the highest water uptake at 90% RH. 567 However, we also reported longer stabilization time to reach equilibrium for bark fiberboards, which might be of 568 interest for humidity regulation.
- To go further, a future work will consist in developing in-situ, long-term RH cycling methods to better characterize the insulation performance of fiberboards. Indeed, temperature, moisture content and their variation/cycling are important parameters affecting thermal properties of wood and wood-based materials. New quantitative data are required to assess thermal conductivity and durability of insulation fiberboards in real conditions of use. In conclusion, it was possible to produce competitive insulation fiberboards from tropical residues, paving the way for promising future research.
- 575

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586	Refei	rence	S

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706 **COMPETING INTERESTS**

707 The authors have no relevant financial or non-financial interests to disclose.

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709 AUTHOR CONTRIBUTIONS

- 710 J. Bossu contributed to the study conception and design. Material preparation, data collection and analysis were
- performed by J. Bossu, J. Moreau, C. Delisee and R. Sonnier. The first draft of the manuscript was written by
- 712 J. Bossu and all authors commented on previous versions of the manuscript. All authors read and approved the
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- 714

715 DATA AVAILABILITY

- 716 The datasets generated during and/or analyzed during the current study are available in the "Guyavalofibres"
- 717 repository, https://doi.org/10.18167/DVN1/KVNAQ6.