



HAL
open science

Hop stems, a new fibrous bioresource

Roxane Limousin, Ferial Guendoul, Canelle Vibert, Karina Antoun, César Ségovia, Isabelle Ziegler-Devin, Nicolas Brosse

► **To cite this version:**

Roxane Limousin, Ferial Guendoul, Canelle Vibert, Karina Antoun, César Ségovia, et al.. Hop stems, a new fibrous bioresource. *Industrial Crops and Products*, 2023, 201, pp.116966. 10.1016/j.indcrop.2023.116966 . hal-04158773

HAL Id: hal-04158773

<https://hal.univ-lorraine.fr/hal-04158773v1>

Submitted on 11 Jul 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial - NoDerivatives 4.0 International License

Hop stems, a new fibrous bioresource

Roxane Limousin,¹ Ferial Guendoul,¹ Canelle Vibert,² Karina Antoun,¹ César Ségovia,²
Isabelle Ziegler-Devin,¹ Nicolas Brosse^{1*}

¹Université de Lorraine, INRAE, LERMAB, F-54000 Nancy, France

²Université de Lorraine, CETELOR, F-88000 Epinal, France

*nicolas.brosse@univ-lorraine.fr

Abstract

The valorization of agricultural wastes in accordance with the concept of biorefinery is currently a subject of great interest. The present deals with hop stem, a co-product of hop cultivation that is currently not properly used. Hop stem was first decorticated to produce ≈ 20 % w/w of technical fibers and shives. Four different degumming processes were investigated: water retting, enzymatic hydrolysis, alkali treatment, and steam explosion at 200°C during 4 min. The fibers produced were comprehensively characterized in terms of chemical composition (Klason lignin and simple sugars by ionic chromatography), mechanical strength, elongation, and morphology by scanning electron microscope (SEM). In contrast to the other processes tested, steam explosion (SE) resulted in efficient defibration due to a significant decrease in pectin ($\approx -70\%$) and lignin content while maintaining good tensile properties (≈ 500 MPa). It was also shown that the hop single cell fiber has characteristics close to those of hemp. The hop shives were used for the production of an adhesive-free panel with good mechanical properties.

Key words: hop; fibers; shives; steam explosion; degumming

30

31 **1. Introduction**

32 The current evolution of our society towards a more sustainable and eco-responsible economy
33 promotes the development of locally available renewable resources. This approach is
34 particularly relevant for natural fibers. Indeed, fibrous plants have always been used and
35 cultivated by mankind especially for the production of fabrics and clothing. In the 20th
36 century, with the development of petrochemicals and intensive cotton cultivation, these
37 traditional productions have almost disappeared. Nowadays, due to their environmental
38 friendliness, attractive properties and low production costs, there is a clear revival of interest
39 in natural fibers for material applications such as reinforcement in composites (Rangappa et
40 al. 2022, Kalia et al. 2009), textile and non-woven. In this context, traditional bast fibers
41 including hemp and flax in Europe, ramie in China, coir and jute in India are increasingly
42 studied and used (Meng et al. 2018, Segovia et al., 2021, Sauvageon et al., 2017). In addition
43 to traditional fiber plants, raw materials that have so far been exploited to a limited extent for
44 their fibers, such as nettle, are considered with interest for future developments (Jeannin et al.
45 2020).

46 Hop (*Humulus lupulus L.*) is a bast fiber plant belonging to *Cannabis* family cultivated for its
47 cone-shaped flowers for beer brewing since at least the sixth century. Nowadays in Europe,
48 hop culture covers almost 30 000 ha primarily in Germany, Czechia, Poland and Slovenia.
49 Hop is a climbing herbaceous plant with lianas that grow to about 10 m in length. Currently
50 only the hop flowers are used and the stem is considered as a by-product, non-properly
51 valorized. Reddy and Yang (2009) showed that the hop stalk contained high cellulose content
52 fibers with properties similar to those of hemp fibers. In human societies, hops never seem to
53 have been used on a large scale for fiber production. However, historical researches described
54 the utilization of hop fibers for textile application in Scandinavia between the 15th and the 19th
55 centuries. A woman's garment partly made of hemp and hop fiber is exposed in the Nordic
56 Museum in Stockholm (Skoglund et al., 2020). The utilization of hop stem to produce pillow
57 in Danish farms during that period is also documented (Lukešová et al. 2019). Through these
58 examples, it appears that already at that time the research for a valorization of the abundant
59 co-products resulting from the beer production was relevant.

60 As for hemp and for bast fiber plants in general, the fibers in the hop stem are embedded in
61 peripheral bast and cemented by non-cellulosic intercellular components, forming bundles

62 composed of several dozen single cell fibers (Bismarck et al. 2005). For industrial crops like
63 hemp or flax, a field retting is traditionally done to facilitate the separation of the fibers from
64 the stem after mowing. The goal of this step is to eliminate the gum composed of pectins,
65 lignin and hemicelluloses by the action of the soil microorganisms and to release fine fibers
66 (Dodd and Kin, 2005). However, the difficult-to-control character of this process makes it not
67 very reproducible and sometimes inefficient. Moreover, this type of field retting which is
68 carried out for plants like hemp or flax would not be possible for hops considering the
69 cultivation techniques and the size of the plant. The conversion of technical fibers into cotton-
70 like fibers, called degumming or “cottonization”, can also performed using chemical (alkali)
71 or enzymatic processes. To the best of our knowledge, the degumming of hop stem has been
72 reported in only one publication (Reddy and Yang, 2009) using alkali.

73 Degumming is generally a limiting step in bast fiber plants conversion, as the development of
74 an efficient and environmentally friendly process is challenging. In addition, this step greatly
75 impacts the chemical, morphological and mechanical properties of the fine fibers produced.
76 Innovative processes have been reported including ultra-sons, enzymatic treatments (Zhang
77 and Yang, 2013)), deep eutectic solvents (Huang et al., 2021; Song et al. , 2019), ionic liquid
78 (Yang et al., 2016), organosolv process (Qu and Zhang, 2020). However, the development of
79 a credible industrial-scale degumming method requires a cost-effective process with low
80 operating costs and low water consumption.

81 Steam explosion (SE) has been described as a green and efficient alternative to traditional
82 degumming processes. SE process can be divided into 2 stages: in the first stage, technical
83 bast fibers are treated with hot steam at $T = 180^{\circ}\text{C} - 210^{\circ}\text{C}$ during few minutes (Ziegler-
84 Devin et al., 2021). During this cooking step, a preferential hydrolysis of the pectin-rich
85 middle lamella occurred. The second stage consists of an explosive decompression generating
86 high mechanical stress and promoting defibration. In our group, this technic has been
87 successfully employed for the degumming of hemp (Sauvageon et al 2017) and ramie
88 (Segovia et al., 2021). In addition to its effectiveness for defibration, the interest of this
89 process is twofold: scaling up is feasible and it can be carried out only with water without any
90 chemical product.

91 The production of binderless panels by thermo-pressing of steam exploded fibers is a long-
92 known process that was industrialized before the Second World War. Gluing is based on the
93 thermo-cross-linking of wood components, primarily lignin, relocated on the surface of the
94 fibers during the steam explosion (Zhang et al., 2015). This type of process is currently
95 gaining interest because of the absence of adhesive in the wood panel and formaldehyde

96 emission. The management of the end of life and the recycling of the adhesive-free material
97 are also greatly facilitated. The work described in the literature concerns primarily the use of
98 exploded wood fibers but, due to shortage of wood supply, the production of panels based on
99 alternative feedstock such as agricultural co-products was also reported (Quintana et al 2009,
100 Gerardi et al. 1998, Velasquez et al. 2002).

101 In the present paper, the decortication of hop stem and the degumming of resulting raw fibers
102 for the production of fine hop fibers was studied. The hop fibers were extracted using
103 traditional water retting, enzymes, alkali treatment and SE. A binderless panel was also
104 produced and characterized from the woody cores tissues (shives) of the hop stem.

105

106 **2. Material and methods**

107

108 **2.1 Feedstock and decortication.**

109 The hop stems were collected in September 2020 in Obernai (France, Lycée agricole) and air
110 dried. Technical fibers were first extracted from the dried hop stems manually by peeling.

111

112 **2.2 Degumming**

113 Water retting was done by immersing the fibers in distilled water (S/L = 1/20) at room
114 temperature for 1 week. The enzymatic degumming was performed at 50°C, pH5 during 2 h
115 using Viscozyme ® (Novozyme) 50 µL/1 g of fibers. *The alkali degumming* was performed
116 according to a method described by Jiang et al. (ref) using NaOH 1%, H₂O₂ 3%, NaSiO₃ 3%,
117 carbamide 3% and sodium polyphosphate 3%, in boiling water during 1 h. *SE degumming:*
118 Technical fibers were impregnated in water during 15 hours at room temperature (L/S =
119 15/1). The fibers (50 g) were transferred into a 2 L steam gun and cooked at 200°C for 4
120 minutes by addition of steam. The pressure was then released into the blast chamber by
121 sudden depressurization. The fibers are recovered after filtration.

122

123 **2.3 Binderless panel**

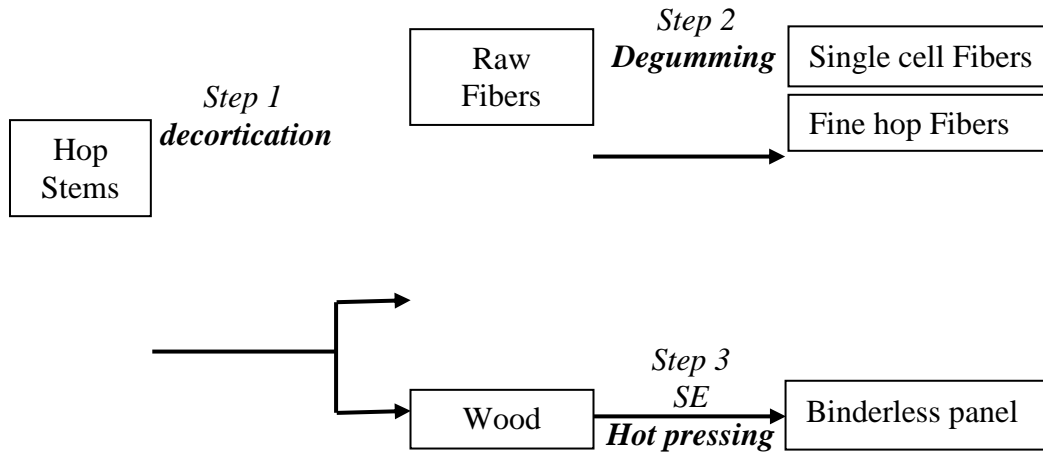
124 The wood (shives) was crushed, impregnated in dilute acid (sulfuric acid, 1%) during 15
125 hours at room temperature and then pretreated by SE (200°C, 5 min). The exploded particles
126 were separated from the liquid fraction by vacuum filtration and dried. Binderless
127 particleboard of 200 x 200 x 5 mm, were prepared by pressing at 230°C 200 g of exploded
128 wood. The pressing is carried out by applying a charge of 4.2 MPa for 5 min followed by a

129 one-minute discharge at 1 MPa and a second charge of 4.2 MPa for 5 min. After pressing, the
130 resulting panels are allowed to cool to room temperature before characterization.

131

132 **Figure 1. Process flow**

diagram



133

134

135

136

137

138 **2.4 Chemical characterization**

139 Hydrolysis and the sugars analysis was performed according to a protocol described
140 previously. Briefly, approximately 0.175 g of ground extractable-free fiber was hydrolyzed in
141 two steps with 72 % sulfuric acid at 30°C for 1 h and then at 4 % at 121°C for 1 h. The
142 sample is filtered (glass microfilter, 1.2 µm), the solid residue is washed with water, dried
143 and weighed. The soluble fraction is diluted with water and analyzed by high-performance
144 anion exchange chromatography – pulse amperometric detection (HPAE-PAD).

145

146 **2.5 Mechanical and microscopical characterization**

147 Scanning electron microscopy (SEM) analysis was carried out with Hitachi 3000 microscope.
148 All images were taken at an accelerating voltage of 15 kV. For measuring single fiber
149 properties, Favimat (Textechno Herbert Stein GmbH and Co. KG, Mönchengladbach,
150 Germany) was used. The fiber ends are clamped between two sets of jaws. The displacement
151 is insured by a constant speed motor with interchangeable equipment to vary the rate of
152 elongation. The data of the load and elongation are transferred to a computer to be plotted and
153 analyzed.

154

155 **2.6 Panels mechanical tests**

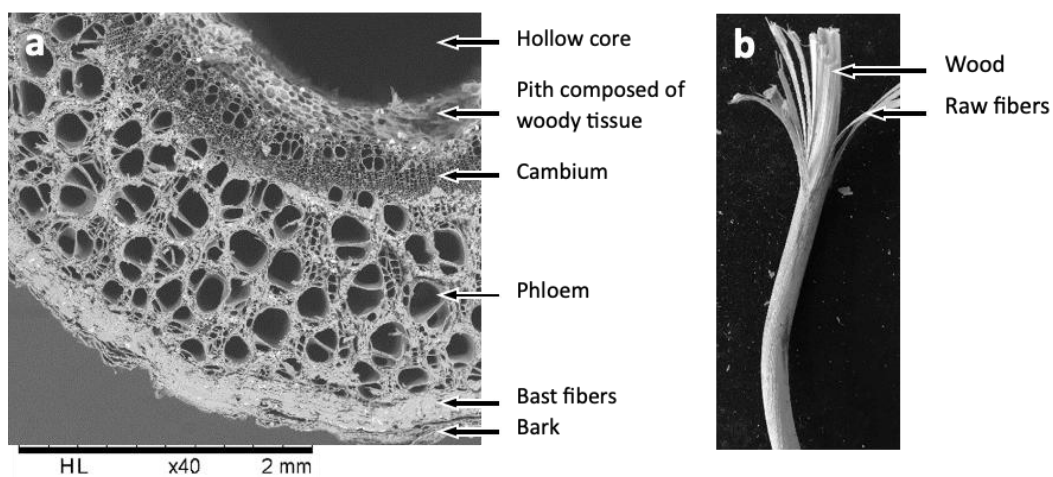
156 The density and the swelling were determined by the EN 323 and EN 317 standard
157 respectively. The strength and flexural modulus of the panels were evaluated using an
158 INSTRON (model 4206) in a climate-controlled room at 20°C and 65 % RH with a charge of
159 1 kN according to EN 310.

160 The internal bond of the panels was determined in accordance with the requirements of the
161 European standard EN 311 using specimens of 50 x 50 mm with ± 1 mm attached to steel
162 plates.

163 3. Results and discussion

164 A micrograph of hop stem cross section is given in Fig. 2A. The hop stem has a structure
165 comparable to that of all bast fiber crops and close to that of the hemp stem. From inside to
166 outside, the stalk is composed of (a) hollow core, (b) pith composed of woody tissue, (c)
167 cambium, (d) phloem (e) bast fibers layer and (f) bark. The bast fibers are thus located
168 between the bark and the woody core and are associated in bundles, by gummy components
169 mainly composed of pectin (Di Virgilio et al., 2015) As seen in Fig 2B, the adhesion between
170 the fibers layer and the wood is relatively weak, the outside layer containing the fibers being
171 easily peeled off from the rest of the dry stem. As a result, a mechanical decortication using
172 breaking rollers with a shaped cross section was efficiently processed from the dry hop stems
173 to isolate the outer layer composed of technical fibers (step 1, Fig. 1). The technical hop fibers
174 yields was ≈ 20 % w/w (based on dry hop stem) which is comparable to hemp fibers yield.

175



176

177 **Figure 2 :** (a) Anatomical description of a cross section of hop stem by SEM and (b) Raw fibers
178 peeled off the stem

179 For the degumming step (step 2, Fig. 1) and based on literature data, four processes were
180 experimented and compared: (1) water retting (2) enzymatic degumming performed using a

181 commercial pectinase- and hemicellulose-rich mixture (Tahri et al. 2016) and (3) a traditional

Retting process	Chemical composition %						Single cell fibers		Technical fibers		
	Lignin	Glc	Xyl	Man	GalUA ¹	Rha +Ara	Other sugars	Diameter (μm)	DI% ²	Tensile strength (MPa)	Elongation %
no	20.35 ± 0.4	45.96 ± 0.33	1.99 ± 0.34	1.52 ± 0.1	4.51 ± 0.21	2.42 ± 0.6	4.56	21.7	1.9	580 ±99	2.5 ±0.92
Water	23.11 ±0.49	33.41 ±0.17	1.33 ± 0.01	0.97 ±0.03	8.02 ±0.01	1.93 ±0.04	4.69	16.9	17.8	261 ±69	3.84 ±1.78
Enzymatic	18.39 ±1.50	34.60 ±47.3 0	1.80 ±2.17	0.94 ±1.27	3.52 ±12.13	2.35 ±2.48	2.56	15	20	479±70	2.60 ±0.61
Chemical	28.45 ±0.69	52.79 ±0.49	1.88 ±0.09	1.34 ±0.12	n.d.	0.44 ±0.08	1.79	16.6	22.7	196 ±68	3.86 ±0.81
Steam explosion	19.02 ±0.46	53.92 ±0.39	3.51 ±0.02	1.77 ±0.07	1.59 ±0.002	0.39 ±0.04	2.31	16.3	29.1	501 ±50	2.97 ±1.33

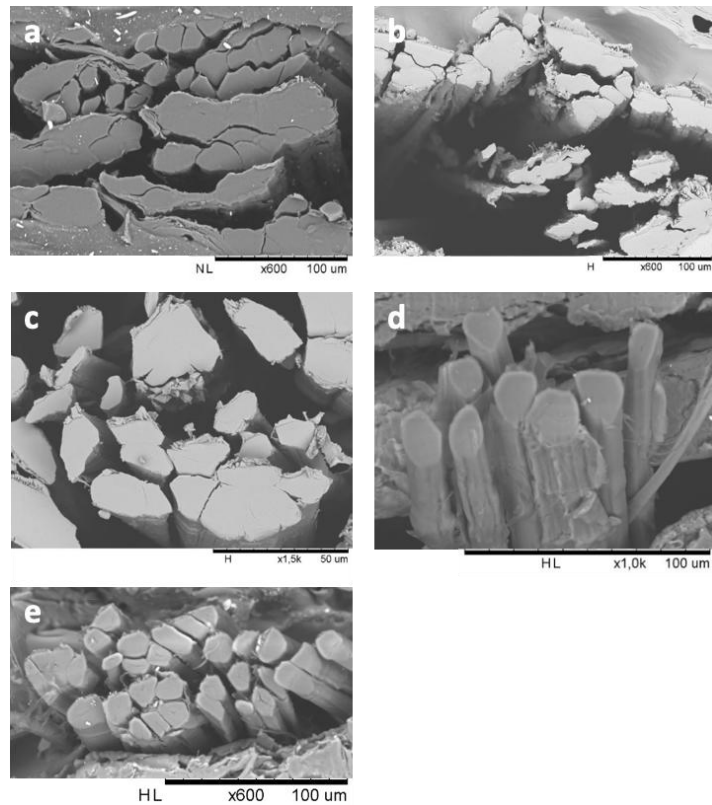
182 alkali-based chemical method (Jianget al., 2018) (4) SE performed in neutral conditions. In
 183 the present study, a water impregnation of the fibers was performed followed by a SE at
 184 200°C during 4 min. The chemical composition of hop technical and treated fibers are
 185 gathered in Table 1. The mechanical and morphological properties of the fibers are also given.
 186 The diameter of single cell fibers and the defibrating rate (DI %) were established by image
 187 processing of SEM pictures of the cross section.

188 **Table 1. Chemical composition and physical properties of technical and retted hop fibers**

189
 190 ¹Galacturonic acid ; ²Defibrating index determined from fibers cross section SEM pictures
 191 DI % = [Σ (area of single cell fibers) / Σ (area of all the fibers)] x 100
 192

193 Fig 3 gives the SEM pictures of the raw and treated fibers. Hop technical fibers displayed a
 194 relatively high lignin content (≈ 20 %), significantly higher compared to hemp and ramie
 195 technical fibers (x % and 10 % respectively). This lignin content, comparable to that of wood,
 196 is explained by the structure of the hop stem, which is very different from that of herbaceous
 197 plants and similar to that of woody plants. The raw fibers also retained relatively high glucan
 198 sugars (≈ 46 %) but also galacturonan, rhamnann and arabinan sugars (≈ 4.5 % and ≈ 2.4 %)
 199 suggesting high cellulose and pectin contents. It can be seen in Fig. 3A that the raw hop fibers
 200 are composed of large bundles with a very low content in single cell fibers (DI ≈ 2 %, see
 201 Table 1). As seen in Table 1, water and enzymatic retting did not lead to an efficient
 202 degumming, the content of non-cellulosic compounds being little impacted. An increase of
 203 the DI was observed (DI ≈ 18-20 %) nevertheless, the SEM photos (Fig. 3 A and C) showed
 204 relatively little separated fibers and the presence of thick bundles. For both processes, a
 205 significant decrease in glucose content was observed, implying a degradation of the cellulosic

206 fibers. This cellulose degradation was confirmed by a decrease in mechanical strength of the
207 fibers especially after water retting (from 580 Mpa for raw fibers to 260 MPa for water retted
208 fibers). This result suggested the presence of cellulolytic enzymes degrading the fibers.
209 For chemical and SE retting, the Glc content relatively increased ($\approx 53\text{-}54\%$) meaning that
210 cellulose of the fibers was globally preserved during both processes. On the other hand, a
211 strong and significant decrease in pectin and hemicellulose markers (GalUA, Rha, and Ara)
212 for both chemical and steam explosion treatments suggested efficient degumming. In
213 accordance with a previous paper (Wang et al., 2003), the alkali treatment allowed an almost
214 total pectin removal characterized by a complete hydrolysis of galacturonans. Interestingly, it
215 is noted that the SE treatment performed in neutral conditions led to a lower lignin content in
216 the resulting fibers than that obtained by the alkali-based chemical treatment (lignin = ≈ 19.0
217 % and $\approx 28.5\%$ respectively). This result was unexpected because the SE process performed
218 under neutral (or acid) conditions is not described as delignifying. Indeed, previous studies
219 have shown that lignin is impacted, depolymerized and relocalized at the fiber surface but is
220 not removed. In our study and in accordance with a previous work performed on ramie fibers
221 (Segovia et al., 2021), a joint and significant decrease in lignin and pectin content by the SE
222 process led to an efficient defibration. Indeed, well separated and thinner fibers were observed
223 by SEM, with significantly more elementary fibers (DI = 29.1 %). In addition, the tensile
224 properties of the SE fibers displayed higher properties than the other retted fibers. In our
225 previous works carried out using hemp and ramie fibers, we have shown that a basic
226 impregnation prior to SE resulted in a very efficient fiber degumming, producing fine fibers
227 with a low variability. However, these basic conditions also produced damaged fibers with
228 cracks and lower tensile properties. The use of SE in neutral conditions seems to be more
229 suitable to produce fibers with low degradation and high tensile properties.



230
 231 **Figure 3.** SEM pictures of fibers. Retting process: (a) no, (b) water, (c) chemical, (d) enzymatic, (e)
 232 steam explosion

233
 234 Table 2 gathered the main characteristics of hop single cell obtained in the present study and
 235 compared them with data from previous reports performed on hemp, flax, cotton and hop sing
 236 cell fibers. The single cells were identified and isolated in each batch of fibers. In addition to
 237 the morphological and strength properties of the fibers previously discussed, the microfibrillar
 238 angle (MFA) is also considered, MFA being one of the most critical variables to determine
 239 the overall properties of the fibers. In our study MFA was determined by X-ray diffraction.
 240 The data in the table shows that the results obtained for the hop fiber are relatively close to
 241 those described by Reddy and Yang (2009) but with significantly longer individual fiber
 242 lengths. Moreover, compared to other industrial natural fibers, it appears that the hop fiber
 243 presents morphological and mechanical characteristics close to those of hemp.

244
 245 **Table 2. Comparison of cotton, hemp and flax fibers with hop single cell fibers**

Fibre	Diameter (μm)	Length (mm)	Tensile strength (MPa)	Elongation (%)	MFA	ref
flax	20 ± 3	6 ± 4	400 - 1300	2.5 - 3.3	6 - 9	Mariana et al, 2014
hemp	27 ± 2	1 ± 0.5	203 - 1100	1 - 5	2 - 6.2	

Cotton	18 ± 2	3 ± 1	300 - 780	6 - 7	/	
Hop	16.5				8	Reddy and Yang, 2009
Hop (this study)	11 - 18	6±5	501	2.5 – 3.9	6.7	

247
248 Binderless panels were produced from hop shives using SE pretreatment according to a
249 previously described method (Zhang et al., 2015). The shives were presoaked in dilute
250 sulfuric acid and then steam exposed at 200°C - 5 min and the particleboards were obtained
251 using a three-stage pressing method. The mechanical properties of the panels prepared are
252 given in Table 3 and compared with those of panels produced using the same protocol from
253 spruce particles. The internal bond strength IB of the panels is a direct measure of bond
254 performance. The IB of the hop board is close to that of the spruce board (0.49 Mpa and 0.54
255 Mpa respectively), the densities of the two panels being very similar ($\approx 0.87 \text{ kg/m}^3$). This
256 result shows that the internal cohesion of the hop board is good and met the international
257 standard specifications for interior grade boards (IB > 0.35 MPa). The thickness swelling of
258 the hop board was greater than that of the spruce board (12 % and 5 % respectively). This
259 result is in agreement with previously reported studies that showed better water resistance for
260 woody materials than non-woody materials (Ye et al., 2007). Nevertheless, compared to
261 literature data, the swelling of the hop board is relatively low showing a good resistance to
262 moisture.

263
264 ***Table 3. Characteristics of the binderless panels produced from steam exploded hop shives***
265 ***and spruce wood.***

	Hop	Spruce
Density (kg/m ³)	868 ±13	875 ±90
Swelling (%)	12 ±0.31	5.2 ±2.6
IB (MPa)	04.9 ±0.05	0.54 ±0.01
Bending strength (MPa)	16 ±0.59	11 ±0.46
Elasticity (MPa)	2066 ±97	940 ±14

266
267 **4. Conclusion**
268 In this study a full valorization of the co-products of hop cultivation is proposed. The
269 technical and fine fibers were successfully isolated by a technique without retting. It has been
270 shown that the steam explosion process used in neutral medium allows a particularly efficient

271 degumming by removing an important part of pectin and lignin and producing fibers with
272 promising mechanical and morphological characteristics. The elementary fiber has
273 characteristics close to those of hemp. Finally, the hop wood recovered during the
274 decortication stage was valorized by the production of an adhesive-free panel. We hope that
275 these results will allow the development of valorization ways for this biomass currently not
276 properly used.

277
278 **Acknowledgements:** EA 4370 LERMAB is supported by the French National Research
279 Agency through the ARBRE Laboratory of Excellence (ANR-12-LABXARBRE-01). The
280 authors acknowledge the financial support of the Conseil Regional de Lorraine through 3BR
281 project and the European Commission through FEDER funds.
282

283
284 **Statements & Declarations. Competing Interests:** Authors declare they have no financial
285 interests.
286

287 **Author Contributions: Isabelle Ziegler-Devin, Roxane Limousin, Ferial Guendoul,**
288 **Canelle Vibert, Karina Antoun :** Analysis. Investigation **Arnaud Besserer, César Segovia:**
289 Methodology, investigation, analysis. **Nicolas Brosse:** Project administration and supervision.
290 Writing- Original draft preparation.
291

292 **References**

- 293
- 294 Bismarck, A., Mishra, S., Lampke, T. Plant Fibers as Reinforcement for Green Composites. Chap 2
295 In: Mohanty A, Misra M, Drzal L, editors. Natural fibers, Biopolymers, and biocomposites.
296 Taylor & Francis. CRC Press. 37–108 (2005). DOI:[10.1201/9780203508206.ch2](https://doi.org/10.1201/9780203508206.ch2)
- 297 Di Virgilio, N., Papazoglou, E. G., Jankauskiene, Z., Di Lonardo, S., Praczyk, M., Wielgusz, K., 2015.
298 The potential of stinging nettle (*Urtica dioica* L.) as a crop with multiple uses. *Ind. Crops Prod.* 68,
299 42-49. doi.org/10.1016/j.indcrop.2014.08.012
- 300 Dodd, R.B. and Akin, D. E. Recent development in retting and measurement of fiber quality in natural
301 fibers: Pro and cons. Chap 4 In: Mohanty A, Misra M, Drzal L, editors. Natural fibers,
302 Biopolymers, and biocomposites. Taylor & Francis. CRC Press. 37–108 (2005).
303 doi: 10.1201/9780203508206.ch4
- 304 Gerardi, V., Minelli, F., Viggiano, D., 1998. Steam treated rice industry residues as an alternative
305 feedstock for the wood based particleboard industry in Italy. *Biomass Bioener.* 14 (3), 295-299.
306 doi.org/10.1016/S0961-9534(97)10042-3
- 307 Huang, H., Tang, Q., Lin, G., Yu, C., Wang, H., Li, Z., 2021. High-efficiency and recyclable ramie
308 cellulose fiber degumming enabled by deep eutectic solvent. *Ind. Crops Prod.* 171 (1), 113879.
309 doi: 10.1016/j.indcrop.2021.113879
- 310 Jeannin, T., Yung, L., Evon, P., Labonne, L., Ouagne, P., Lecourt, M., Cazaux, D., Chalot, M., Placet,
311 V., 2020. Native stinging nettle (*Urtica dioica* L.) growing spontaneously under short rotation
312 coppice for phytomanagement of trace element contaminated soils: Fibre yield, processability and
313 quality. *Ind. Crops Prod.* 145, 1119997. doi.org/10.1016/j.indcrop.2019.111997
- 314 Jiang, W., Song, Y., Liu, S., Ben, H., Zhang, Y., Zhou, C., Han, G., Ragauskas, A., 2018. A green
315 degumming process of ramie. *Ind. Crops Prod.*, 120, 131-134.
316 doi.org/10.1016/j.indcrop.2018.04.045
- 317 Kalia, S., Kaith, B.S., Kaur, I., 2009. Pretreatments of natural fibers and their application as
318 reinforcing material in polymer composites—A review. *Polym. Eng. Sci.* 49 (7), 1253-1272. doi
319 10.1002/pen.21328
- 320 Lukešová, H., Andersen, H. L., Kolínová, M., Holst, B., 2019. Is It Hop? Identifying Hop Fibres in a
321 European Historical Context, *Archaeometry.* 61 (2), 494-505. doi: 10.1111/arcm.12437).
- 322 Meng, C., Yang, J., Zhang, B., Yu, C., 2018. Rapid and energy-saving preparation of ramie fiber in
323 TEMPO-mediated selective oxidation system. *Ind. Crops Prod.* 126 (15), 143-150
324 doi.org/10.1016/j.indcrop.2018.09.030
- 325 Moriana, R., Viaplana, F., Karlsson, S., Ribes, A., 2014. Correlation of chemical, structural
326 and thermal properties of natural fibres for their sustainable exploitation. *Carbohydr.*
327 *Polym.*, 112, 422-431. doi.org/10.1016/j.carbpol.2014.06.009
- 328 Qu, Y., Yin, W., Zhang, R., Zhao, S., Liu, L., Yu, J., 2020. Isolation and characterization of cellulosic
329 fibers from ramie using organosolv degumming process. *Cellulose.* 27, 1225–1237.
330 doi.org/10.1007/s10570-019-02835-w
- 331 Quintana, G., Velásquez, J., Betancourt, S., Gañán, P., 2009. Binderless fiberboard from steam
332 exploded banana bunch, *Ind. Crops Prod.* 29 (1), 60-66 doi.org/10.1016/j.indcrop.2008.04.007
- 333 Rangappa, S., M., Siengchin, S., Parameswaranpillai, J., Jawaid, M., Ozbakkaloglu, T., 2022.
334 Lignocellulosic fiber reinforced composites: Progress, performance, properties, applications, and
335 future perspectives. *Polym. Compos.*, 43 (2), 645-691. doi.org/10.1002/pc.26413
- 336 Reddy, N. and Yang, Y., 2005. Properties and potential applications of natural cellulose fibers from
337 cornhusks. *Green Chem.* 7, 190-195. doi:10.1016/j.carbpol.2009.03.013
- 338 Reddy, N. and Yang, Y., 2009. Properties of natural cellulose fibers from hop stems. *Carbohydr.*
339 *Polym.* 77 (4), 898-902. doi:10.1016/j.carbpol.2009.03.013
- 340 Sauvageon, T., Lavoie, J.-M., Segovia, C., Brosse, N., 2017. Toward the cottonisation of hemp fibers
341 by steam explosion - Part 1 - Defibrillation and morphological characterization. *Tex. Res. J.* 88 (9),
342 1047-1055. doi.org/10.1177/0040517517697644
- 343 Segovia, C., Hedri, M., Huot, H., Zigler-Devin, I., Liu, C., Tang, Y., Qiu, R., Morel, J.-L., Brosse, N.,
344 2021. Industrial Ramie growing on reclaimed ion-adsorption Rare Earth Elements mine tailings in
345 Southern China: Defibrillation and fibers quality. *Waste Biomass Valorization.* 12, 6255–6260.
346 doi:10.1007/s12649-021-01442-w

347 Skoglund, G., Holst, B., Lukešová, H., 2020. First experimental evidence of hop fibres in historical
348 textiles. *Archaeol. Anthropol. Sci.* 12, 214. doi.org/10.1007/s12520-020-01171-6
349 Song, Y., Kai, N., Jiang, W., Zhang, Y., Ben, H., Han, G., Ragauskas, A., 2019. Utilization of deep
350 eutectic solvent as a degumming protocol for Apocynum venetum bast. *Cellulose*. 26, 8047–8057.
351 doi.org/10.1007/s10570-019-02654-z
352 Tahri, I., Ziegler-Devin, I., Ruelle, J., Segovia, C., Brosse, N., 2016. Extraction and Characterization
353 of Fibers from Palm Tree. *Bioresour.* 11 (3), 7016-7025. 10.15376/biores.11.3.7016-7025
354 Velásquez, J. A., Ferrando, F., Farriol, X., Salvado, J., 2003. Binderless fiberboard from steam
355 exploded Miscanthus sinensis. *Wood Sci. Technol.* 37, 269–278. doi.org/10.1007/s00226-003-
356 0182-8
357 Wang, H. M., Postle, R., Kessler, R. W., Kessler, W., 2003. Removing pectin and Lignin During
358 Chemical Processing of Hemp for Textile Applications. *Tex. Res. J.* 73(8), 664-669.
359 doi:10.1177/004051750307300802
360 Yang, F., Ma, Y., Qian, Y., Lv, L., Zheng, L., Zhao, Y., 2016. A novel ionic liquid degumming
361 process for Apocynum venetum. *J. Text. Inst.* 107 (11), 1450-
362 1455. doi: 10.1080/00405000.2015.1127550
363 Ye, X. P., Julson, J., Kuo, M., Womac, A., Myers, D., 2007. Properties of medium density fiberboards
364 made from renewable biomass. *Bioresour. Technol.* 98, 1077-1084.
365 doi.org/10.1016/j.biortech.2006.04.022
366 Zhang, Q., Yan, S., 2013. Degumming of ramie bast fibers by Ca²⁺-activated composite enzyme. *The*
367 *J. Tex. Inst.* 104 (1), 78-83. doi: 10.1080/00405000.2012.693273
368 Zhang, D., Zhang, A., Xue, L., 2015. A review of preparation of binderless fiberboards and its self-
369 bonding mechanism. *Wood Sci. Technol.* 49, 661–679. doi.org/10.1007/s00226-015-0728-6
370 Ziegler-Devin, I., Chrusciel, L., Brosse, N., 2021. Steam Explosion pretreatment of lignocellulosic
371 biomass: a mini-review of theoretical and experimental approaches. *Frontiers in Chemistry, section*
372 *Green Sus. Chem.* 9, 705358. doi:10.3389/fchem.2021.705358
373
374
375
376
377