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Toward the cottonisation of hemp fibers by steam explosion – Part 1: Defibration and morphological characterization

Abstract

Fine hemp fibers (cottonized hemp) were processed using steam explosion. The quantification of the defibration rate was performed by image processing. Based on this method, the hemp defibration was optimized using a response-surface methodology based on three-variable central composite design for the production of elementary fibers with low variability. Optimal parameters for the steam processes were as follow: time = 4.1 min, temperature = 191°C. Biomass was impregnated with a solution of NaOH (8 %) before treatment, leading to a defibration rate of 91.2 %, which is producing \approx 50% fibers with length < 3 mm in good agreement with the experimental data. Damaged fibers originating from the conjugated effect of steam explosion and alkali hydrolysis were also observed.

Key words : Hemp fibers; steam explosion; elementary fibers

1 **Introduction**

2

3 Natural fibers have accompanied human societies since prehistoric times. During the last
4 century, the success of the petrochemical industry dramatically slowed the growth of bio-
5 based materials. Cheap petroleum-based materials have replaced natural fibers like hemp and
6 flax in almost all their traditional utilizations (1). Recently, environmental awareness and the
7 growing global waste problem have spurred much interest in renewable resources. The current
8 development of natural fibers and bio-based materials are motivated by legislation as well as
9 customers' demands for more sustainable and environment-friendly products (2). As a result,
10 the environmental impact of a material is now to be considered at all stages of its life cycle,
11 including during production, recycling and final disposal. The major advantages of natural
12 fibers over synthetic ones are their low specific weight, their local availability and their
13 biodegradability and/or possible thermal recycling (3). This has motivated researchers to
14 utilize natural plant fibers for material applications such as textile, non woven and as
15 reinforcement in composites. New developments in packaging, furniture, building and
16 automotive industries have been described. (4,2,5-7)

17 Hemp is one of the bast fibers grown in temperate climates which have provided many
18 important products such as fibers for textile, seeds (oil), as well as pulp and paper. Hemp
19 elementary fibers are on average 13 to 15 mm long with a diameter between 15 to 20 μm
20 (5,8). They potentially display very interesting properties for industrial applications such as
21 high tenacity, good tensile strength and excellent moisture resistance (9-11). On the other
22 hand, one of the major characteristic of hemp fibers and of natural fibers in general, limiting
23 large-scale high-quality industrial developments, is their non-uniformity, their wide variation
24 in dimension and in quality such as strength and fineness. In addition to the natural variability
25 of the feedstock, the procedures used for the extraction of the technical fibers from the stem of

1 bast plant (retting process) greatly impact the morphology and the properties of the fibers
2 (12–16). Hemp technical fibers are coarser and stiffer than other bast fibers such as flax and
3 they have relatively high lignin content (4 to 8 %) (14). They are composed of elementary
4 fibers, cemented by intercellular components (mainly lignin and pectin). Refined processing,
5 chemical and enzymatic approaches have already been proposed in the literature to produce
6 fine / elementary fibers from technical hemp. Chemical processing (also called degumming)
7 traditionally performed using sodium hydroxide solution is effective for removing non
8 cellulosic components attached to hemp fibers and to release unitary fibers. Nevertheless, the
9 limitations of this chemical approach include the production of relatively short fibers with low
10 tensile strength and environmental issues regarding the liquid effluent disposal. Thus the
11 availability of (elementary) fibers with the required quality for textile and composite
12 industries (cottonised hemp) is still a challenging issue to fulfill the industrial demand (4,17–
13 20).

14 Steam explosion (SE) is currently one of the most valuable pretreatment technologies for
15 cellulosic bioethanol production (21–24). After impregnation, biomass is treated with hot
16 steam (around 200°C) under pressure (around 1.5-3 MPa) during a few minutes followed by
17 an explosive decompression. The effect of SE on biomass combines a chemical hydrolysis
18 during the steam treatment and defibration during the explosive decompression (25). Steam
19 explosion process has been previously experimented for the extraction of fibers and primarily
20 micro, nano/fibers from biomass. Reported on Kessler et al. (25), the production of linen from
21 flax by SE is made after a soda impregnation. Cellulose nano-fibrils were successfully
22 produced from pineapple leaf, banana stem, jute and hemp by successive steam explosion
23 techniques combined with chemical treatments (26–28). Vignon et al. (29) studied the
24 production of fibers from chenevotte by steam explosion after acid impregnation. It was
25 shown that temperatures around 220°C – 230C were required. At $T < 210^{\circ}\text{C}$ delignification

1 rates were low while at $T > 240^{\circ}\text{C}$, degradation and fiber damage were observed. The
2 production of hemp fibers and microfibers using steam treatments has already been studied
3 (30-32). In these previous studies, the chemical composition of the produced fibers was
4 generally examined (lignin, sugar content, cellulose DP). However, SE has never been
5 successfully employed and optimized for the production of elementary fibers starting from
6 technical hemp and in the perspective of minimizing variability as well as fibers damage.
7 Although this technique has been described in several works for the production of fibers,
8 interaction effect of process parameters and complexity of fiber characterization have caused
9 difficulties for the evaluation and optimization of the SE process. Three major parameters
10 were found to have significant effects in a SE process: the temperature and the duration of the
11 steam treatment and the conditions of the impregnation of the fibers prior to the SE. Statistical
12 methods were often employed to investigate the optimum conditions for a biomass treatment.
13 Response Surface Methodology (RSM) was used to design a systematic experimental method
14 and has been extensively described for the optimization of biomass pretreatment in cellulosic
15 biomass-to-fermentable sugars conversion (30, 33). To the best of our knowledge, RSM has
16 never been described for the optimization of a defibration process.

17

18 In this paper, the optimization of elementary NaOH-impregnated hemp fibers extraction using
19 SE is described. An original quantification method was developed in order to follow the
20 defibration rate by image processing. Defibration was evaluated and optimized using RSM to
21 design systematical experimental method for the production of superior quality fibers with a
22 low variability.

23

24 **Materials and methods**

25

1 **1. Raw material**

2 In this study, fibers of industrial hemp (*Cannabis sativa* L.) were investigated. The hemp was
3 cultivated close to Shefford, Quebec, Canada (N45.21, O72.34). It was sown in April 2013
4 and harvested in September 2013. Plants were cultivated according to common farming
5 practices in Quebec. Stems were cut a few centimeters above ground and were subjected to
6 winter retting. Fibers were isolated, dried and kept in a dry and well ventilated place. Prior to
7 impregnation and steam explosion treatment, samples were cut into 2-3 cm pieces.

8

9 **2. Impregnation**

10 Biomass was impregnated with a solution of sodium hydroxide (0; 4; 8 wt %). The fibers
11 were macerated during 15 hours at room temperature using a weight ratio water/biomass of
12 15/1 after what the biomass was filter-pressed (6.8 atm = 100 psi) to remove the excess water.

13

14 **3. Steam explosion treatment**

15 After pressing, the fibers were transferred into a 4.8 L steam gun where about 100 g (dry
16 basis) of biomass was cooked at temperatures ranging from 190°C to 210°C for 1, 3 or 5
17 minutes by addition of steam in a pressure-resistant reactor. After the cooking period, a
18 pneumatic valve is open and the vapor phase exits the reactor through a nozzle entraining as
19 well the fibers. The investigated biomass was treated at the Industrial Research Chair on
20 Cellulosic Ethanol and Biocommodities (CRIEC-B) of the University of Sherbrooke, Canada.

21

22 **4. Washing, drying and carding**

23 A washing step was necessary after the steam treatment to remove the residual NaOH in the
24 fibers. Therefore biomass was washed with 1.5 L of water at 12°C, without stirring during 15
25 minutes and then filter-pressed (6.8 atm = 100 psi). This washing step was repeated three

1 times after what the fibers were dried in an oven at 104°C during 24h. After drying the fibers
2 were conditioned at 20°C ± 2°C and 65% H.R. ± 4% H.R.

3 A typical carding machine with a main cylinder and three pairs of worker and stripper rollers
4 was used, whereby a cylindrical surface of the cylinder is provided with a specifically
5 clothing for natural fibers.

6

7 **5. Statistical approach**

8 A systematic experimental method was designed to optimize the steam explosion treatment of
9 hemp fibers using Central Composite Design (CCD) and Response Surface Methodology
10 (RSM). Sodium hydroxide (NaOH) concentration in the impregnation liquid, reaction
11 temperature and residence time were studied as three independent variables. The experiment
12 matrix is representated in Table 1 and JMP software (version 11.0.0) was used for regression
13 analysis.

14

15 **6. SEM analysis**

16 Scanning electron microscopy (SEM) analysis was carried out with Hitachi 3000 microscope.
17 All images were taken at an accelerating voltage of 15 kV. The fibers were put parallel to
18 obtain a lock and cut. The cross section was observed with the SEM and pictures were taken
19 Image Pro Premier 9.2 was used for the pictures analysis.

20

21 **7. Defibration rate**

22 A wick of fibers was made for each sample and cut to obtain a cross sections of a lot of fibers.
23 Four pictures of each wick were taken with a X250 magnification SEM. The perimeter of
24 each fiber was manually drawn with Image-Pro Plus software version 6.0.0.260. From these
25 data, the area of each section of unit fiber was measured. Then, the area of each section of

1 fibers bundle was measured. A formula was determined to calculate a rate of defibration (D
2 %):

$$D (\%) = \frac{\Sigma(\text{areas of unit fibers})}{\Sigma(\text{areas of all the fibers})} * 100$$

3

4 **8. Diameter of hemp fibers**

5 A wick of fibers was made for each sample and cut to obtain a cross sections of a lot of fibers.
6 Four pictures of each wick were taken with a X250 magnification SEM. The perimeter of
7 each fiber was drawn with Image-Pro Plus software version 6.0.0.260. From these data, the
8 mean diameter was measured. Between 600 and 1500 fibers were measured for each sample
9 and the mean diameter was measured for each fiber.

10

11 **9. Length of hemp fibers**

12 Fibers were spread on a glass plate using oil allowing their optimal separation. Six pictures of
13 each sample were taken with an optical microscope and they were analyzed with the software
14 Image Pro Premier 9.2. The lengths of the fibers were measured (between 150 and 300 fibers)
15 for each sample.

16 **Results and discussion**

17

18 Technical hemp fibers were processed using SE after 15 h impregnation at room temperature
19 in a sodium hydroxide solution for which concentrations varied from 0-8%. Prior to
20 impregnation, hemp fibers had to be cut into 2-3 cm pieces in order to facilitate their transfer
21 from the reactor into the blow down chamber during the explosive decompression. Table 1
22 shows the results obtained from 17 SE experiments performed at temperature varying from
23 190°C to 210°C, for 1 min to 5 min. Log R° which describes the SE treatment severity as a

1 function of the treatment time and temperature (30, 33) are also given in the Table 1. It was
 2 previously shown that the thermal degradation of cellulose was limited for a SE with $\text{LogR}^\circ <$
 3 4.0 (34). As a result, the experimental conditions for the SE procedures covered a large range
 4 of severity but below 4.0 ($\text{Log R}^\circ = 2.6 - 3.9$) in order to limit the cellulosic fibers
 5 degradation. The morphological characteristics of the untreated and treated fibers were
 6 examined. The evaluation of the results for each experiment was based on the SEM analysis
 7 of the cross section aiming of the changes in the structure of the fibers bundles after carding.
 8 Examples of SEM pictures cross sections for untreated and treated hemp samples (samples 1
 9 and 3) are given in Fig. 1. The defibration rate (in %) for each sample was established by
 10 image processing based on the sections of undamaged elementary fiber and on the sections of
 11 bundles (see experimental section). The defibration rates for untreated (control) and treated
 12 fibers (sample 1 to 7) are given in Table 1.

13

14 Table 1. SE conditions and defibration rates D (%) for untreated and treated fibers.

15

Steam explosion conditions					
sample	T(°C)	t(min)	LogR[°]	NaOH (%)^a	D (%)^b
control	-	-		-	2.1
1	190	1	2.6	0	34.3
2	190	3	3.1	4	75.3
3	210	5	3.9	8	82.7
4	200	5	3.6	4	77.7
5	210	3	3.7	4	63.7
6	210	1	3.2	0	49.7
7	200	3	3.4	4	85.1

8	190	5	3.3	0	33.3
9	210	5	3.9	0	37.1
10	200	3	3.4	8	91.6
11	200	1	2.9	4	46.8
12	200	3	3.4	0	34.1
13	200	3	3.4	4	79.4
14	210	1	3.2	8	75.6
15	190	1	2.6	8	71.1
16	190	5	3.3	8	83.5
17	200	3	3.4	4	76.5

1

2 ^aNaOH concentration in water of the solution used for the fibers impregnation at room temperature3 during 15h. ^b Defibrillation rate

4

5 From Fig. 1A it appears that the untreated hemp sample is composed of bundles having an
6 average diameter of 100 μm to 500 μm and containing 5 to 30 single fibers. The percentage of
7 isolated single fibers in the sample is very low (2.1%). This observation is in accordance with
8 previously described works (34). In contrast, after treatment, it can be seen from Fig. 1B and
9 C that the fibers are well separated. In Fig1B (sample 1), the sample is composed of thinner
10 bundles with significantly more individual fibers ($D = 34.3\%$) while in Fig 1C, the sample is
11 mostly composed of single fibers ($D = 82.7\%$).

12

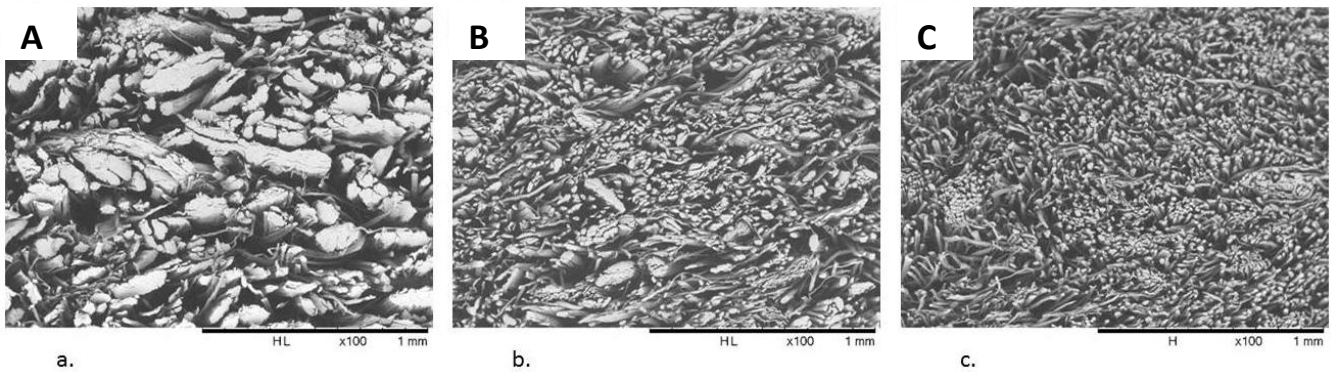


Fig. 1. SEM photos of cross sections of untreated and SE treated hemp samples : A. Hemp fibers without treatment, sample 0. B. Hemp fibers after SE, sample 1; C. Hemp fibers after SE, sample 3.

The response-surface plots concerning the defibration rates as a function of the three variables studied (t , T , NaOH %) are provided in Fig. 2. Figure 2A and 2B depict the interaction between temperature and catalyst, as well as time and catalyst respectively. These figures demonstrate the important impact of sodium hydroxide concentration on the defibration rates even at low cooking time and temperature. This effect was already observed by Sutka et al. (31,32) and is justified by the mild alkali hydrolysis, which degraded the middle lamella thus separating the elementary fibers. The alkali presoaking allowed the production of thinner fibers, with a smooth surface and removed a large part of the non-cellulosic components (32).

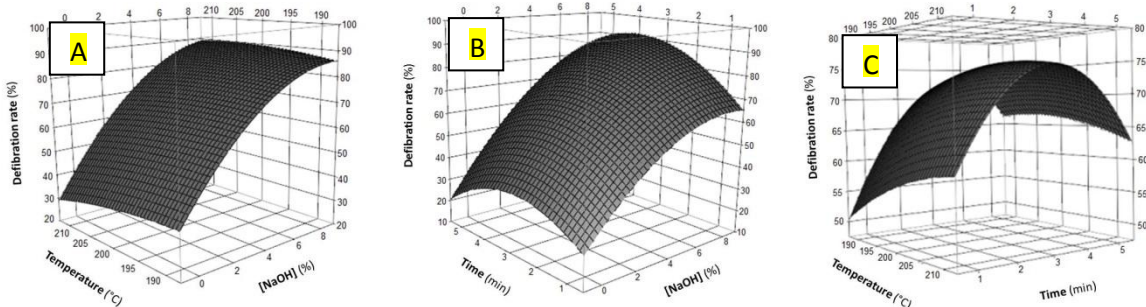
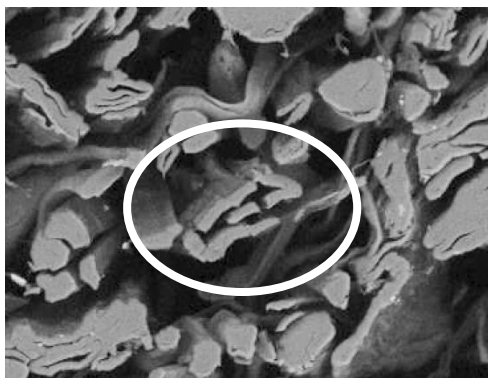


Fig. 2. Response surface plot on defibration rate. A – Effect of temperature and NaOH concentration at residence time of 3 min. B – Effect of residence time and NaOH concentration at reaction temperature of 200°C. C – Effect of temperature and residence time at NaOH concentration of 4%.

1 Fig. 2C shows the interaction between time and temperature. Results show that the defibration
2 rate was low for short residence time and increased with temperature. D% increased with
3 cooking time, although it reached a maximum and decreased again for longer cooking
4 periods. This can be explained by a degradation of individual cellulose fibers due to a
5 combination of the SE severity (33) with the phenomenon of peeling commonly observed in
6 alkaline conditions (34). An example of the damaged fiber observed in sample 3 is given in
7 the Fig.3.

8



9

10

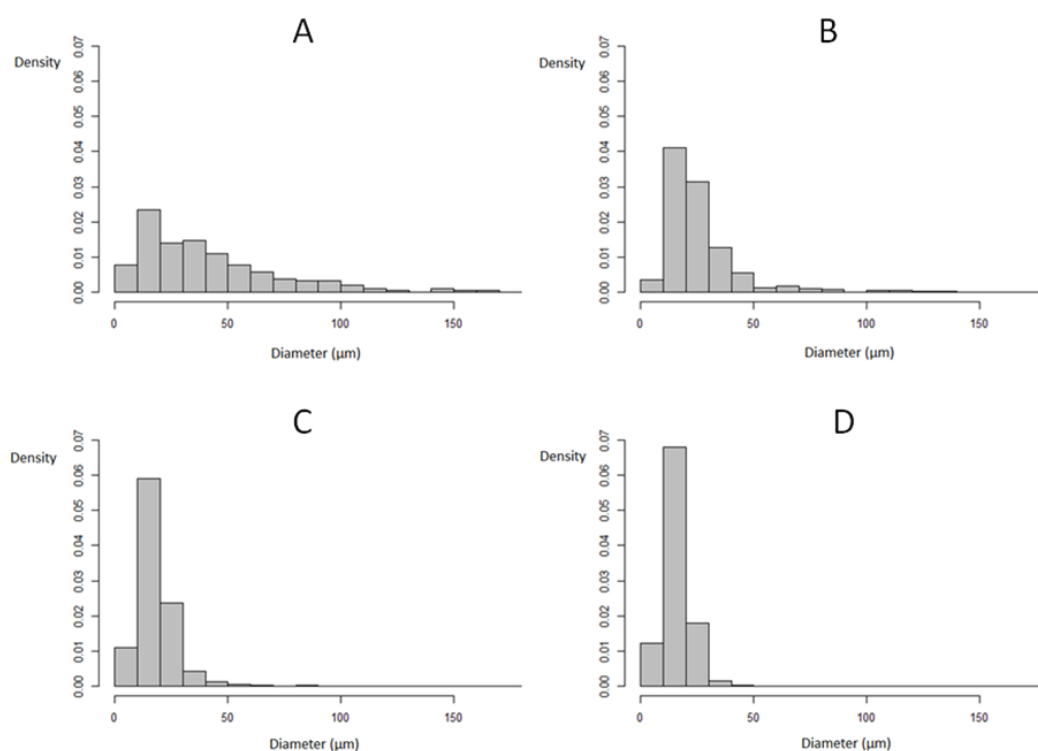
11 Fig. 3. Example of a damaged fiber. (x 500) (T = 210°C. t = 5 min, NaOH % = 8 %)

12

13 The cooking parameters were optimized using the CCD based on the model obtained and the
14 input criteria. The optimization of the defibration rate was carried out based on the three
15 variables which were in the range of experimental runs. JMP software predicted that
16 optimized conditions for defibration rate could be obtained when the residence time, reaction
17 temperature and NaOH concentration were at 4.1 min, 191°C and 8 %, respectively, giving a
18 defibration rate of 91.2 %. These optimized conditions are very close to those of sample 10
19 (200°C, 3 min, NaOH 8%) which gave an actual defibration rate of 91.6%, in good agreement
20 with the value calculated using the model.

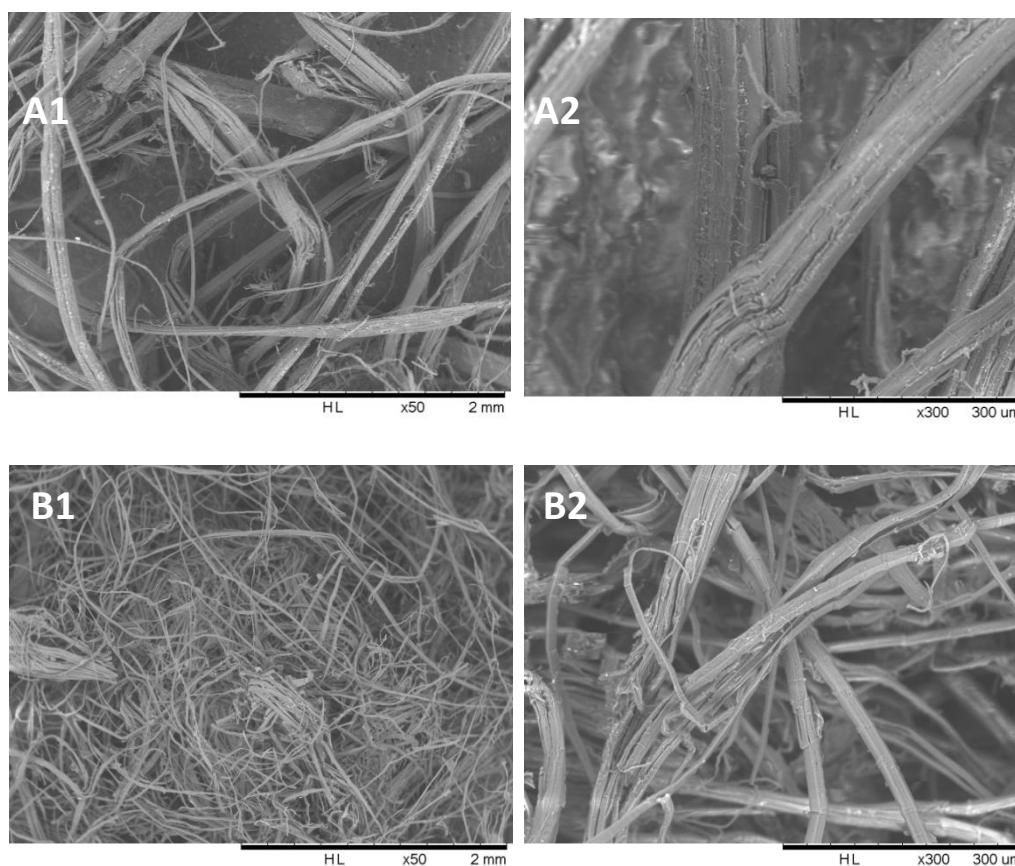
21

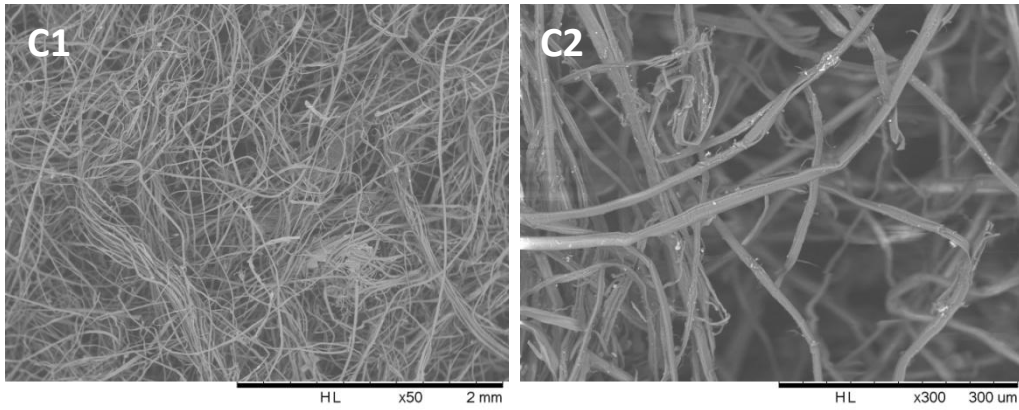
1 The average diameters of all the fiber bundles were determined from the cross section
 2 obtained from their SEM pictures (Fig. 1). The average diameter of the raw fibers (41.1 μm)
 3 is slightly higher than the value reported by Ilczyszyn F et al. (11) (30 μm). Fig 4.A shows
 4 that the raw hemp displayed a broad range of diameters with very thick fiber bundles (> 150
 5 μm). After SE, significantly finer fibers were collected with average diameters ranging from
 6 15.5 μm to 25.5 μm . It appears that thicker fibers ($> 19 \mu\text{m}$) were obtained for the treatments
 7 without sodium hydroxide (samples 1, 6, 8, 9 and 12). This observation confirms the major
 8 role of this reagent in removing the non-cellulosic material attached to fibers (essentially
 9 lignin and pectic components) thus releasing individual fibers. Fig.4B, C, D shows the
 10 diameter distributions for steam-exploded samples 1, 5, 10 respectively. A narrower range of
 11 diameters was observed with average diameters usually below 50 μm . Sample 10 (Fig 4.D)
 12 corresponds to the highest defibrations yields ($D = 91.6 \%$), the diameter range obtained was in
 13 accordance with the values described for hemp individual fibers (17-23 μm).



14
 15 Fig. 4. Diameters distribution of hemp fibers. A – Untreated fibers ; B – 190°C, 1min, NaOH 0% ; C – 210°C,
 16 3min, NaOH 4% ; D – 200°C, 3min, NaOH 8%.

1
2 Longitudinal SEM views of hemp fibers before and after SE treatment are shown in Fig. 5.
3 For untreated samples, fiber bundles showing some splitting due to the mechanical separation
4 during carding can be seen clearly (Fig. 5, A1 & A2). As previously observed, the effect of
5 SE are clear in Fig 5, B & C. Using less severe conditions (sample 1, Fig. 5, B1 & B2), less
6 thick fibers bundles composed of 2 to 5 elementary fibers were observed. For sample 3, the
7 uniformity of the fibers was clearly improved. Nevertheless, fibers damages due to the harsh
8 conditions used (NaOH impregnation and explosive decompression) and/or the subsequent
9 mechanical carding were also observed.





1

2 Fig. 5. SEM images of untreated and treated hemp samples (longitudinal view). A. Hemp fibers without
 3 treatment, sample 0, A1 x50, A2 x300. B. Hemp fibers after SE, sample 1, B1 x50, B2 X300. C. Hemp fibers
 4 after SE, sample 3, C1 x50, C2 X300.

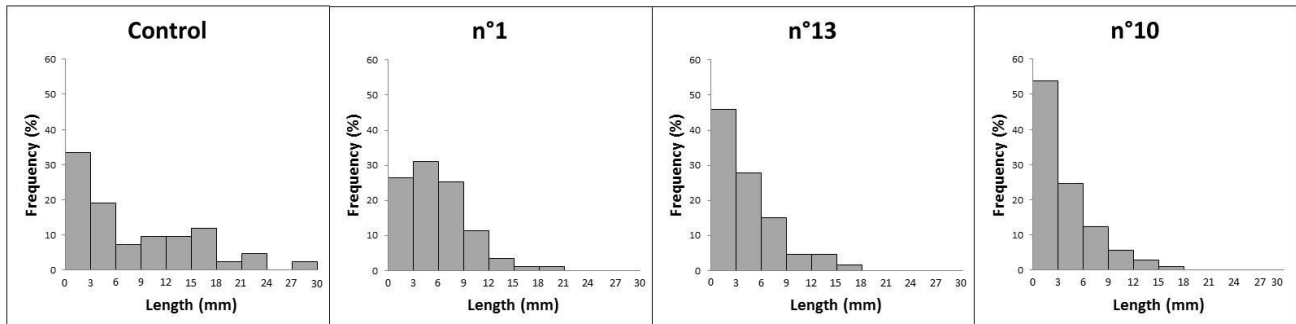
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6

7 The fibers length distributions for samples 1, 10, 13 and for the raw fibers are reported in Fig.
 8 6. The raw hemp fiber gauge length was found in a 1 to 30 mm range which is in accordance
 9 with previous works. After SE, shorter fibers were recovered (< 20 mm). Results show that a
 10 significant decrease of the fibers length with a high content in short fibers (< 3 mm) was
 11 observed for the more severe conditions (Fig. 6). Samples 13 and 10 were produced using the
 12 same conditions but with 2 different concentrations of NaOH during the impregnation step
 13 (4% and 8 % respectively). The larger proportion ($\approx 50\%$) of short fibers (< 3 mm) obtained
 14 with higher soda concentration can be justified by (1) the reduction in the number of
 15 elementary fibers in the bundles as previously proposed and in accordance with the
 16 defibration rate values (79.4 % and 91.6% respectively), (2) elementary fibers damage
 17 because of the conjugated effect of steam explosion and alkali hydrolysis, (3) the technical
 18 options in the present study : as previously explained the hemp fibers had to be cut into small
 19 pieces prior to the treatment because of the geometry of the steam explosion facility. As a

1 result, future developments for textile applications would necessitate a technological
2 adaptation of the reactor for the production of longue fibers.

3



4

5

6 Fig. 6. Frequency distribution of lengths for untreated and treated hemp samples: Control: Hemp without
7 treatment; n°1: Hemp fibers after SE sample 1. n°13: Hemp fibers after SE with $T = 200^{\circ}\text{C}$, $t = 3$ min and
8 $[\text{NaOH}] = 4\%$; n°10: Hemp fibers after SE with $T = 200^{\circ}\text{C}$, $t = 3$ min and $[\text{NaOH}] = 8\%$.

9

10 Conclusions

11

12 In this paper we described the optimization of hemp fine fiber extraction using SE processes.

13 A method by image processing was developed to quantify the defibrating rate and to

14 characterize the fibers. Based on this method, a systematical experimental approach was

15 developed for the first time for the production of superior quality short fibers (< 3 mm length)

16 with low variability, composed of $\approx 92\%$ of elementary fibers. However, damaged fibers due

17 to the conjugated effect of steam explosion and alkali hydrolysis were also observed. A full

18 chemical and morphological characterization of exploded fibers will be presented in a

19 following publication. Utilizations of these fibers in the fields composite should be considered

20 because of the physical characteristics of the fibers.

21

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4 Sauvageon's grant during his stay in Canada and for providing access to experimental (steam
5 explosion) apparatus.

6

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