



OATAO is an open access repository that collects the work of Toulouse researchers and makes it freely available over the web where possible

This is an author's version published in: <http://oatao.univ-toulouse.fr/26572>

Official URL:

<https://doi.org/10.1016/j.matpr.2020.01.365>

To cite this version:

Jeannin, Thomas and Yung, Loïc and Evon, Philippe[✉] and Labonne, Laurent[✉] and Ouagne, Pierre[✉] and Lecourt, Michael and Cazaux, David and Chalot, Michel and Placet, Vincent *Are nettle fibers produced on metal-contaminated lands suitable for composite applications?* (In Press: 2020) Materials Today: Proceedings. ISSN 2214-7853

Any correspondence concerning this service should be sent to the repository administrator: tech-oatao@listes-diff.inp-toulouse.fr

Are nettle fibers produced on metal-contaminated lands suitable for composite applications?

Thomas Jeannin^a, Loïc Yung^b, Philippe Evon^c, Laurent Labonne^c, Pierre Ouagne^d, Michael Lecourt^e, David Cazaux^f, Michel Chalot^b, Vincent Placet^{a,*}

^aFEMTO-ST Institute, Université Bourgogne Franche-Comté, Besançon, France

^bLaboratoire Chrono-Environnement (LCE), Université Bourgogne Franche-Comté, Montbéliard, France

^cLaboratoire de Chimie Agro-Industrielle (LCA), INP-ENSIACET, Toulouse, France

^dLaboratoire Génie de Production (LGP), Université de Toulouse-ENIT, Tarbes, France

^eInTechFibres Division, FCBA, Grenoble, France

^fInovyn, Tavaux, France

A B S T R A C T

This work assesses the potential of nettle (*Urtica dioica* L.) fibers produced on contaminated lands for composite applications. The nettles studied in this work grew spontaneously and in a prevalent manner in poplar short rotation coppice planted for the phytomanagement of a land contaminated by traces of metals. Results show that the contaminant contents in nettle bast fibers are low: only traces were measured. It makes it possible to consider this biomass for material use. The measured matter yield is lower than those obtained with traditional fiber crops cultivated in Europe on agricultural lands but the tensile properties of the bast fibers are equal to or better than those of hemp and flax, making spontaneous nettle an interesting supplement to traditional European fiber crops for composite applications.

Keywords:

Nettle fibers
Metal-contaminated lands
Phytomanagement
Composite
Single fiber test

1. Introduction

Environmental contamination is a threat to global sustainable development. The UN Sustainable Development Goals (SDGs) have a strong focus on reducing and managing environmental pollution. Pollution can be in air, water, workplace and soil. This work is focused on soil pollution and more precisely on the phytomanagement of metal-contaminated lands. Among the whole phytoremediation options, phytostabilisation is a sustainable and profitable use of marginal lands. It creates value from contaminated land while minimizing environmental risk and even rendering environmental services such as the restoration of important ecosystem, carbon storage and erosion control. This study is part of a wider research project, called PHYTOFIBER (<http://phytofiber.fr/>), which main objective is the multi-purpose valorization of wood and plant fibers produced on mixed-metal-contaminated soils. Interestingly, it has been observed simultaneous growth of poplar short rotation coppice (SRC) cropping systems and spontaneous dioecious nettle

in phytomanaged land sites. Thus, poplar coppice and nettles constitute a multilevel canopy (Fig. 1, left). Biomass from nettle could be used to extend the productivity of the marginal land from coppice alone, whilst still maintaining a functioning phytomanagement system of land rehabilitation. This nettle biomass could constitute an interesting fiber source and thus contribute to answer the increasing demand in plant fibers for material application, by mitigating at the same time the land-use conflict between the needs of food and non-food production. Indeed, recent literature underlines that the use of marginal land is a sustainable method to expand purpose-grown biomass and essential for meeting the emerging massive requirement for biomass in the future [1]. However, for the metal-contaminated lands the question of the translocation of trace elements (TE) in nettle biomass can be raised. Previous studies revealed that nettle is a poor TE accumulating species when grown on contaminated soils, as compared to woody accumulating or hyperaccumulating species. Indeed, in previous works, Zn and Cu contents in nettle leaves collected at a sediment landfill were within physiological ranges, and Cd content was inferior to 10 mg/kg DWt [2–4]. But, there is no clear conclusion regarding the content in stems and bast fibers. For other fiber

* Corresponding author.

E-mail address: vincent.placet@univ-fcomte.fr (V. Placet).



Fig. 1. Photographs of stinging nettle growing spontaneously and in a prevalent manner under short rotation coppice on metal-contaminated lands.



Fig. 2. Photographs of stinging nettle harvesting.

crops, such hemp, it was previously demonstrated that bast fibers and shives were not affected by TE contamination [5].

The present study investigates the potentialities of nettles growing spontaneously under SRC grown poplar for material application. The experimental site was contaminated by Hg due to a chlor-alkali process. Trace element contents were determined in soil and in the harvested biomasses. The bast fiber content was determined after mechanical processing using a Laroche (France) Cadette 1000 “all fiber” extraction device without preliminary retting. Finally, the morphological and mechanical properties bast fiber were characterized.

2. Materials and methods

2.1. Site description

The experimental site is located in Tavaux, France (Bourgogne Franche Comté region lat. 47° 5' 5.985" N – long. 5° 19' 44.0322" E). This site was exploited from the 1950 s to 2003 as a storage area for sediments originated from effluents produced during the electrolytic processes associated with a Hg cell chlor-alkali activity.

Four replicate field blocks were established in April 2011 using unrooted poplar cuttings Skado (*P. trichocarpa* × *P. maximowiczii* section Tacahamaca) and I214 (*P. deltoides* × *P. nigra* section Aigeiros) of 1.5 m length, planted at an initial spacing of 1.8 m × 2.75 m, with a final plant density of 2.200 trees ha⁻¹, each block therefore consisting in 48 trees (6 × 8). The dioecious nettle was observed to grow spontaneously in a prevalent manner in these poplar short rotation coppices (Fig. 1).

2.2. Nettle harvesting

A plot of approximately 50 m² was harvested on the 8th of June 2018 at the edge of the poplar SRC. Stems were cut at soil level using scythes and shears. The aerial parts of the cut plants were then immediately picked up by hand and baled, without any prior retting. The stems were kept aligned in the bales (Fig. 2.). The bales were picked up the same day and stored in indoor spaces for 4 months.

2.3. Stem length and diameter

Stems were analyzed in terms of dimensions by selecting randomly 30 stems in the bales. Stem diameters were measured for each stem in three positions along the stem height (top, middle and base parts of the stem) using a caliper with a precision to 0.01 mm. The length was measured using a graduated steel rule with a precision of 0.1 cm. A dozen of cross sections of stems were then analyzed under a microscope Keyence VHX 5000 series with a magnification of 20. On each cross section, the wall thickness and diameter of 20 fibers was measured.

2.4. Stalks processing and fiber extraction

The stalks were mechanically processed using a Laroche (France) Cadette 1000 “all fiber” extraction device. After mechanical processing, the fractions of the resulting matters were determined. These matters are divided into three categories: a fiber lap containing the extracted bast fibers, shives and dust.

2.5. Single fiber tensile properties

Some fibers (single fibers and possibly small bundles of fibers) taken from the fiber laps were tensile tested. Approximately 50 fibers with a mean diameter of about $28 \pm 6 \mu\text{m}$ were tested on a Bose Electroforce 3230 machine. The sample size (50 fibers) represents a compromise between the statistical processing requirements and time consumption. The fibers were first glued on a paper frame and then examined using an optical microscope to determine their average external width. The paper frame supporting each fiber was clamped onto the testing machine and then cut prior to the beginning of each test. The clamping length was 10 mm. Fibers were tested at a constant crosshead displacement rate of 0.01 mm s^{-1} . The sample elongation and the load were recorded continuously. The strain was calculated as the elongation divided by the initial fiber length. The stress was calculated using the applied force and the evaluated initial cross-section of the fiber. The effective cross-section was determined using the mean external width, assuming that the fiber was to be perfectly cylindrical and the lumen area neglected. The apparent tangent modulus of rigidity (E) was computed on a strain range comprised between 0.2% and the strain at failure%. The mean value and standard deviation of these tensile properties were then computed.

2.6. Determination of TE content

Before analyses, dried nettle stems and leaves were separated, air-dried and ground into a homogenous powder in a Mixer Mill, for 4 min at 30 Hz and 7 min at 30 Hz for leaves and stems, respectively. Fibers were also isolated. Hg was measured using the atomic absorption spectrophotometry AMA-254 cold vapor atomic absorption (CV-AAS) Hg analyzer (Altec Co., Czech Republic), using standard conditions (45 s drying, 150 s heating, 45 s cooling) and the certified reference material (CRM), i.e. Oriental Basma Tobacco leaves (INCT-OBTL-5). All the results are reported as concentrations in $\mu\text{g}/\text{kg}$ of the dry weight.

3. Results and discussion

3.1. Straw yields and bast fibers content

The yields of fresh matter were determined by weighing the nettle bales just after harvesting. Results are presented in Table 1. A yield of 3.5 t/ha of fresh matter was measured. After 4 months of drying the matter was weighed once more. The obtained dry matter yield was approximately 0.55 t/ha. These yields are lower than those mentioned in the open literature in the context of traditional agronomic practices. Indeed, literature [6–9] reports dry matter yields comprised between 2.3 and 15.4 t/ha depending on the fertilization conditions. These yields are nevertheless extremely interesting since, in this agro-forestry system, nettle grows naturally and spontaneously, without chemical inputs. The savings associated to the lack of planting/sowing, crop maintenance and inputs may motivate harvesting lower yielding plots.

After mechanical extraction, a fiber yield of approximately 9.1% of the initial mass of nettle stalks was measured. This results in

Table 1
Straw yield and bast fiber content after mechanical processing on a Laroche-Cadette 1000 equipment.

Fresh biomass yield (t/ha) (stalks and leaves)	Dry straw yield (t/ha) (stalks)	Bast fiber content (%)
3.5	0.55	9.1

accordance with the literature [10] which reports fibers yields between 3 and 8%.

3.2. Nettle TE contents

The soil of the site used in this study was contaminated by Hg, with concentrations around 6 mg/kg D Wt [11]. Table 2 shows that the translocation of Hg to nettle tissues remained low, as Hg contents in leaves did not exceed 0.03 mg/kg D Wt. The Hg content in fibers was below the limit of quantification of the device. These Hg contents are far below the tolerable threshold for agronomics crops (0.2 mg/kg D Wt [12]). So, the industrial use of such fibers in material applications should not be a problem.

3.3. Morphological features of stems and fibers

Table 3 summarizes the morphological features of the nettle stems after drying. An average stem height of approximately 107 cm was measured for a diameter varying between 4.33 and 1.42 cm, on average, from the base to the top part of the stem. Bacci et al. [6] reported for the German Nettle clone 13 a mean stem height of 170 cm and a basal diameter of 8.1 mm. This result shows that the stem dimensions of the native nettle are lower than the cultivated ones.

Concerning the fiber features, the mean diameter is about $37 \mu\text{m}$. This value is in agreement with the mean diameter measured by Di Virgilio et al. [13] for cultivars. Fig. 3 shows the transverse cross-section of a nettle stem. It can be observed that the bast fibers present a quite large lumen area and a thin wall thickness of about $6.5 \mu\text{m}$, when compared to other European crops such as hemp and flax. Anyway, these features are dependent on the plant maturity. In this study, the harvest period of the native nettle was not determined with regard to the plant maturity.

So, for this study, it can be concluded that even if the crop yield for the native nettle growing spontaneously is lower than for cultivars and traditional agronomic practices, the fiber yield and features are similar. Before being generalized, this conclusion requires to be confirmed and averaged using complementary harvests over several years and at different geographical sites.

3.4. Mechanical properties of nettle fibers

Fig. 4 shows the stress/strain curves obtained from the 50 single fiber with a mean diameter of about $28 \pm 6 \mu\text{m}$ which were tensile

Table 2
Hg content in mg/kg D Wt.

Hg	
<i>Tavaux site</i>	
Nettle stems	0.01 ± 0.004
Nettle fibers	NA
Nettle leaves	0.03 ± 0.01
<i>Control site</i>	
Nettle leaves and stems	0.006 ± 0.003

Table 3
Morphological features of nettle stems and fibers.

	Mean value \pm standard deviation
Stem length (cm)	106.6 ± 13
Stem diameter (cm)	
Top	1.42 ± 0.63
Middle	3.2 ± 0.64
Base	4.33 ± 0.87
Fiber diameter (μm)	37 ± 11
Fiber wall thickness (μm)	6.5 ± 2

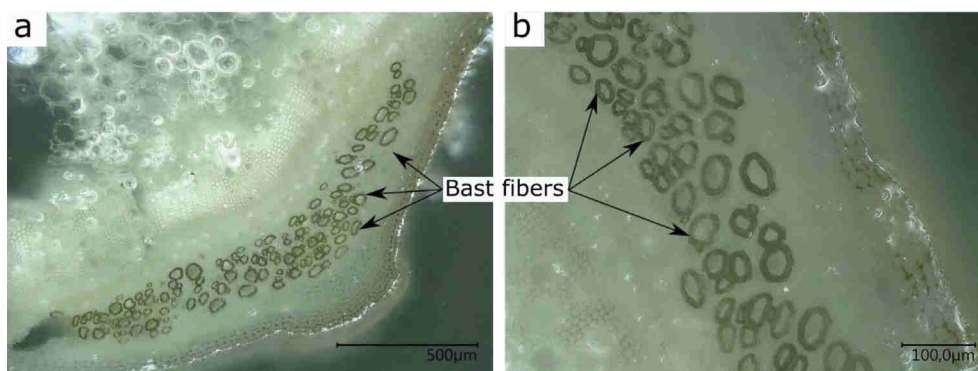


Fig. 3. Micrographs of the transverse cross-section of stinging nettle stems.

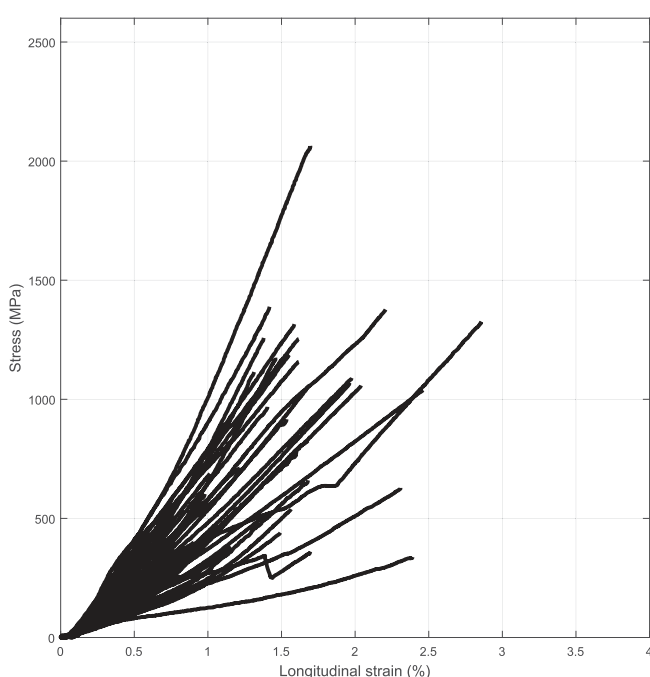


Fig. 4. Results of the single fiber tensile tests.

tested. A large scattering in the response is observed. This result is typical of plant fibers [14]. A significant non-linearity can be observed for most of the tested fibers. This contrasts with the results by Bodros and Baley [15] who reported a linear response for nettle fibers manually extracted from retted stems.

The mean tensile properties are presented in Table 4. The average properties are equal to 711 MPa, 1.37%, 53 GPa for the tensile strength, strain at failure and apparent rigidity, respectively. These values are lower than the ones reported in literature for native netted cultivars [15,16]. These differences can be due to several factors such as the harvesting period or related to the fiber preparation and processing. In literature, most of the studies

Table 4
Tensile properties of extracted bast fibers.

	Mean value \pm standard deviation
Elastic modulus (GPa)	53 \pm 24
Stress at failure (MPa)	711 \pm 427
Strain at failure (%)	1.37 \pm 0.53

provide results for fibers manually extracted. In this study fibers were extracted mechanically using an “all fiber” extraction device, which is well-known to be traumatic for fibers. Anyway, this value is representative of an industrial process and certainly more realistic in view of material engineering. These properties remain also very promising when compared to the best results obtained for the other European fiber crops, such as flax. Bensadoun et al. [17] reported for industrial flax fibers 57 GPa and 791 MPa for the tensile rigidity and strength, respectively, when determined using single fiber tensile test.

4. Conclusion

This study demonstrates that the use of trace-element contaminated soils is a relevant option to expand the material purpose-grown biomass and at the same time mitigate the land-use conflict between the needs of food and the increasing demand for plant based fiber raw materials. Results show in particular that bast fibers extracted from a native nettle growing spontaneously on a mercury contaminated soil present Hg contents far below the tolerable threshold for agronomics crops. Despite a crop yield lower than for cultivars and traditional agronomic practices, the content and features of the fiber extracted from this native nettle were observed to be similar. The tensile properties for the native nettle studied in this study are also lower than the ones generally reported in literature. They could be improved by optimizing the harvesting date and thus the stage of maturity and the associated fiber wall characteristics. Anyway, in this study we demonstrated that the tensile properties obtained after an extraction made with an industrial device are comparable to the properties of the best industrial flax fibers. This is really promising in view of a material use. Indeed, the savings associated to the lack of planting/sowing, crop maintenance and inputs may motivate harvesting lower yielding plots. The question of the mechanization of the harvesting, collecting and transportation and thus of the economic balance are however still opened.

CRedit authorship contribution statement

Thomas Jeannin: Investigation, Formal analysis, Visualization, Writing - original draft. **Loïc Yung:** Investigation, Visualization. **Philippe Evon:** Investigation, Resources. **Laurent Labonne:** Investigation. **Pierre Ouagne:** Resources. **Michael Lecourt:** Project administration, Funding acquisition. **David Cazaux:** Resources. **Michel Chalot:** Conceptualization, Funding acquisition, Supervision, Writing - review & editing. **Vincent Placet:** Conceptualization, Methodology, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The authors gratefully acknowledge the funding by ADEME, France, under grant no1772C0018, PHYTOFIBER project.

References

- [1] A.K. Mohanty, S. Vivekanandhan, J.-M. Pin, M. Misra, Composites from renewable and sustainable resources: challenges and innovations, *Science* 362 (6414) (2018.) 536–542.
- [2] E. E. Codling et K. L. Rutto, Stinging Nettle (*Urtica dioica* L.) Growth and Mineral Uptake from Lead-Arsenate Contaminated Orchard Soils, *J. Plant Nutr.*, vol. 37(3), pp. 393–405, févr. 2014.
- [3] F.M. Tack et M.G. Verloo, Metal contents in stinging nettle (*Urtica dioica* L.) as affected by soil characteristics, *Sci. Total Environ.*, vol. 192(1), pp. 31–39, Nov. 1996.
- [4] P. Phanthavongsa et al., Effect of mycorrhizal inoculation on metal accumulation by poplar leaves at phytomanaged sites, *Environ. Exp. Bot.* 143 (2017) 72–81.
- [5] P. Linger, J. Mussig, H. Fischer, J. Kobert, Industrial hemp (*Cannabis sativa* L.) growing on heavy metal contaminated soil: fibre quality and phytoremediation potential, *Ind. Crops Prod.* 16 (1) (2002) 33–42.
- [6] L. Bacci, S. Barontì, S. Predieri, N. di Virgilio, Fiber yield and quality of fiber nettle (*Urtica dioica* L.) cultivated in Italy, *Ind. Crops Prod.* 29 (2–3) (2009) 480–484.
- [7] J. Dreyer, G. Dreyling, F. Feldmann, Cultivation of stinging nettle *Urtica dioica* L. with high fibre and cellulose: qualitative and quantitative differentiation of ancient clones, *Angewandte Botanik (Germany)* (1996).
- [8] H. Francken-Welz, M. Scherr-Triebel, J. Léon. Ertragsund Qualitätsbildung von Lein, Hanf und Fasernessel in Abhängigkeit von Bestandesdichte und N-Düngung. *Mitteilungen der Gesellschaft für Pflanzenbauwissenschaften.* 12:177–178.
- [9] A. Hartl et C. R. Vogl, Dry matter and fiber yields, and the fiber characteristics of five nettle clones (*Urtica dioica* L.) organically grown in Austria for potential textile use, *Am. J. Altern. Agric.*, vol. 17(4), pp. 195–200, Déc. 2002.
- [10] J. Dreyer, Die Fasernessel als nachwachsender Rohstoff: Leistungsprüfung von Fasernesseln (*Urtica dioica* L., Große Brennessel) unter besonderer Berücksichtigung der phänotypischen Differenzierung anbauwürdiger Klone, Kovač (1999).
- [11] A. Durand, F. Maillard, J. Foulon, H.S. Gweon, B. Valot, M. Chalot, Environmental metabarcoding reveals contrasting belowground and aboveground fungal communities from poplar at a hg phytomanagement site, *Microb. Ecol.* 74 (4) (nov. 2017.) 795–809.
- [12] K. P. A et P. H, Trace elements in soils and plants. 2nd ed. 1992.
- [13] N. Di Virgilio, Stinging nettle: a neglected species with a high potential as multi-purpose crop, National Research Council of Italy. Institut of Biometeorology. Catania, Italy, vol. 23, 2013.
- [14] A. Bourmaud, J. Beaugrand, D.U. Shah, V. Placet, C. Baley, Towards the design of high-performance plant fibre composites, *Prog. Mater. Sci.* 97 (2018) 347–408.
- [15] E. Bodros et C. Baley, Study of the tensile properties of stinging nettle fibres (*Urtica dioica*), *Mater. Lett.* vol. 62(14), pp. 2143–2145, mai 2008.
- [16] G. Lanzilao, P. Goswami, R.S. Blackburn, Study of the morphological characteristics and physical properties of Himalayan giant nettle (*Girardinia diversifolia* L.) fibre in comparison with European nettle (*Urtica dioica* L.) fibre, *Mater. Lett.* 181 (2016) 200–203.
- [17] F. Bensadoun et al., Impregnated fibre bundle test for natural fibres used in composites, *J. Reinf. Plast. Compos.* 36 (13) (2017) 942–957.