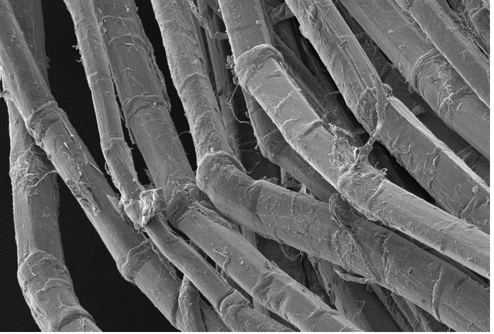
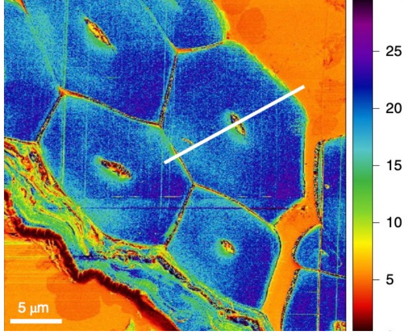
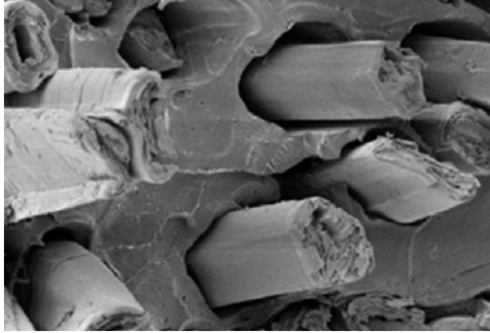






Prendre en compte les spécificités des fibres végétales pour un renforcement optimal des composites biobasés

Alain BOURMAUD

GDR FIBMAT – ENSTA Brest, le 13 novembre 2024

AGENDA OF THE PRESENTATION

- **Brief historical context**
- **Value chain and retting stage**
- **A large diversity of plant fibres**
- **Development and ultrastructure of plant fibres**
- **Multiscale mechanical characterization of plant fibres**
- **Structural defects: a key specificity**
- **Biobased composites processing**

BRIEF HISTORICAL CONTEXT

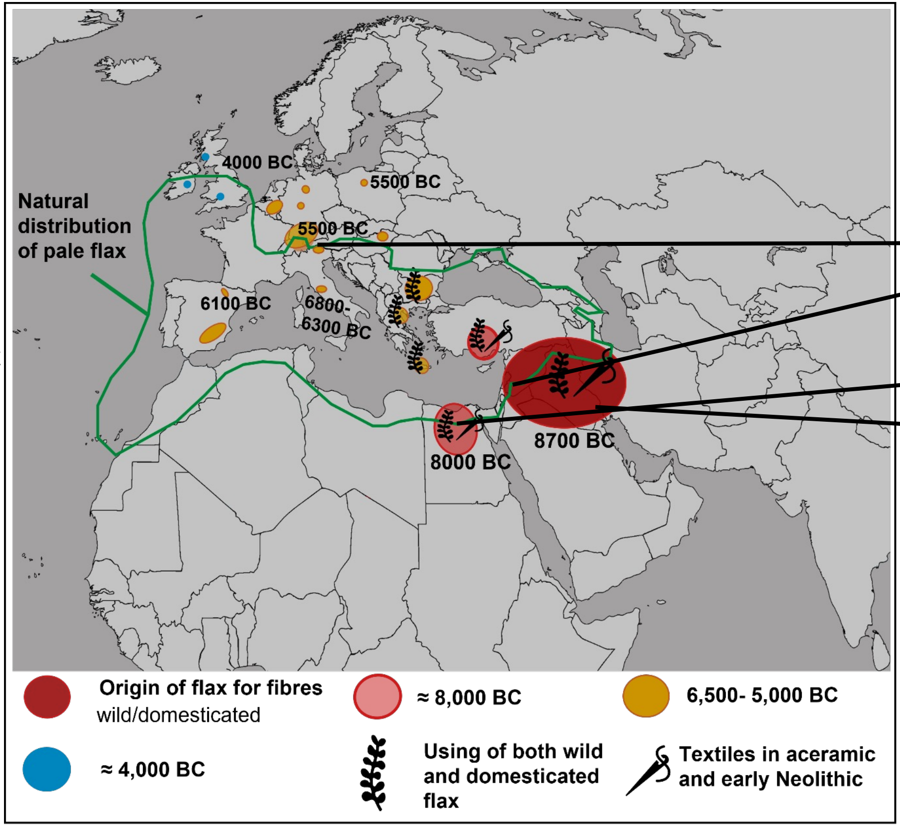
FIRST TRACES - DOMESTICATION

Image inspired by:

Karg, *Veg. Hist. Archaeobot.*, **2011**

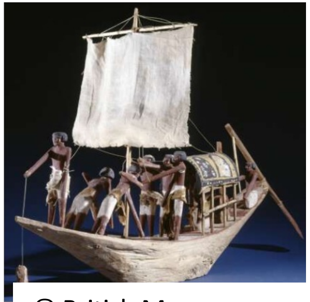
Harris, , in: *Textile Society of America 2014 Biennial Symposium Proceedings.*, **2014**

Melelli et al., under review



Sennedjem's tomb, Valley of the Artisans (Deir el-Medina)

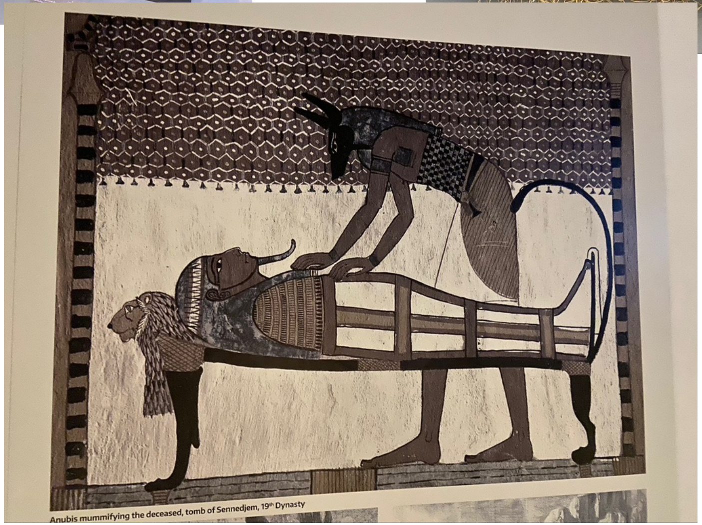
© Paolo Bondielli



© British Museum

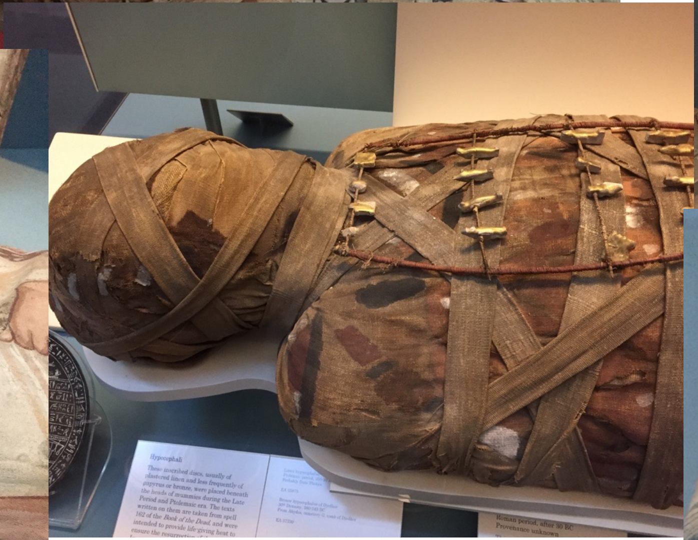
Landi & Hall, ***Stud. Conserv.***, 1979

IN EGYPT



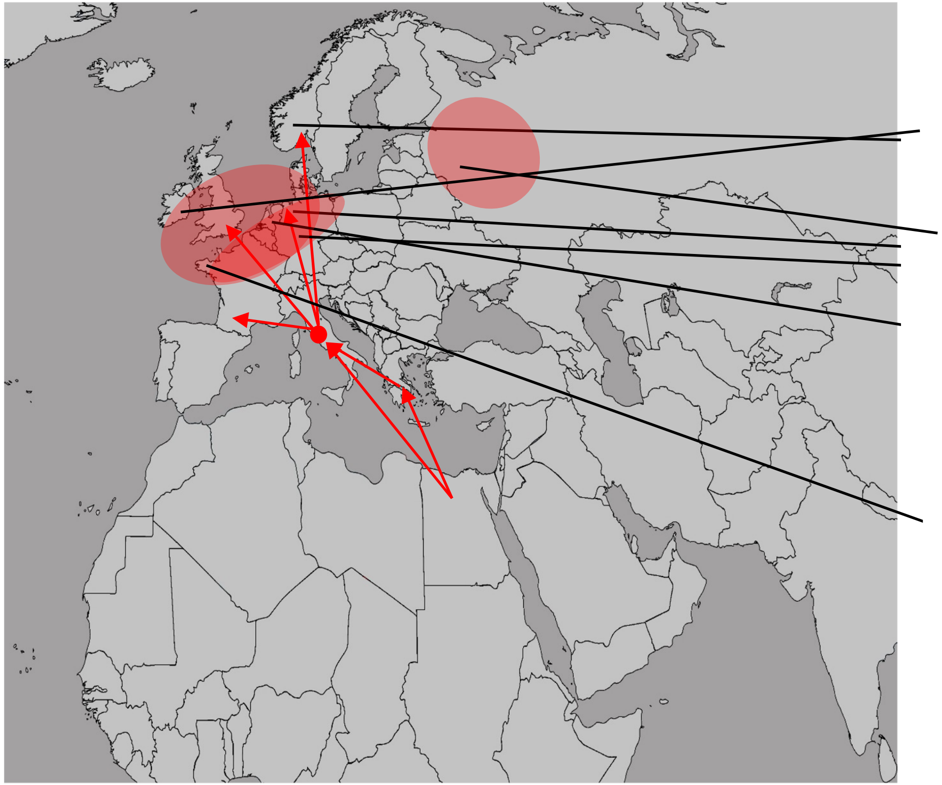
CIVILIZATIONS MUSEUM – CAIRO, 2022

IN EGYPT



BRITISH MUSEUM – LONDON, 2020

THE MAIN CULTIVATION CENTRES



Flax Combers, Fedot Sychkov, 1905

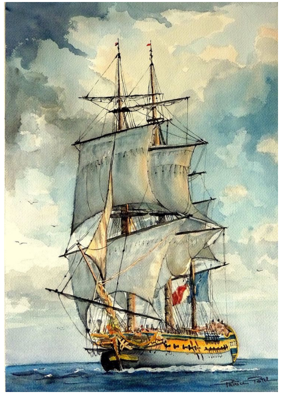
The processing of flax remained unchanged for centuries

IN EUROPE, MORE RECENTLY

Les Cahiers de Dourdon n° 5

In the 17th century, about 300,000 hectares of flax were cultivated in France (today 150,000)

About 20% of textiles are made of flax (less than 0.4% today!)

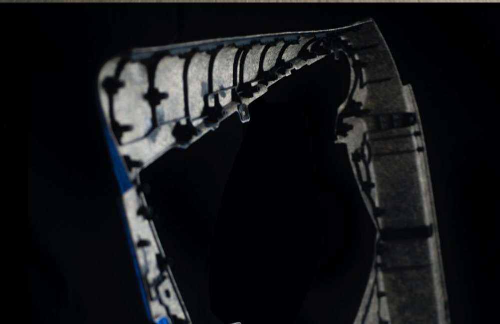


TODAY


LA FRENCH
FILATURE
UN 100% MADE IN FRANCE


Lemaitre Demeestere
1 8 3 5

TODAY

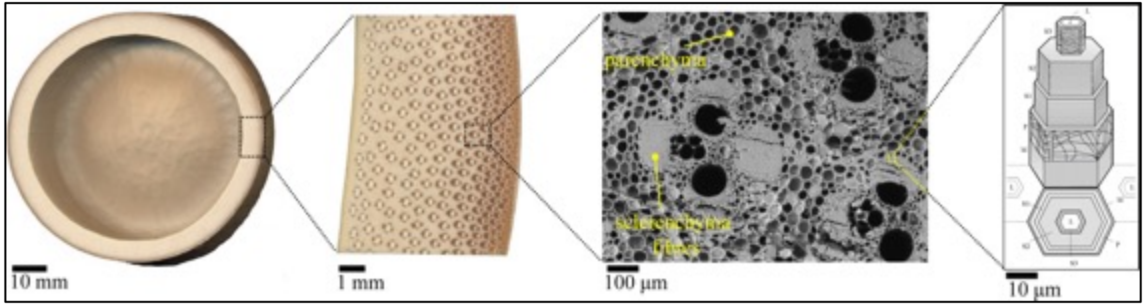


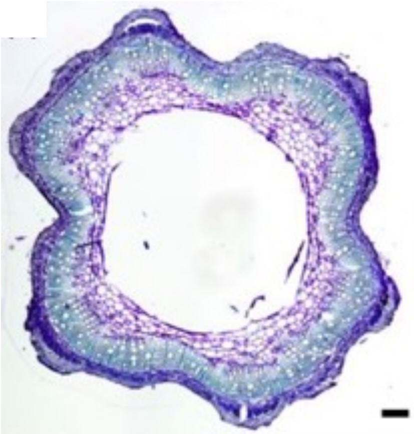
THE CHAIN VALUE

FIBRE EXTRACTION

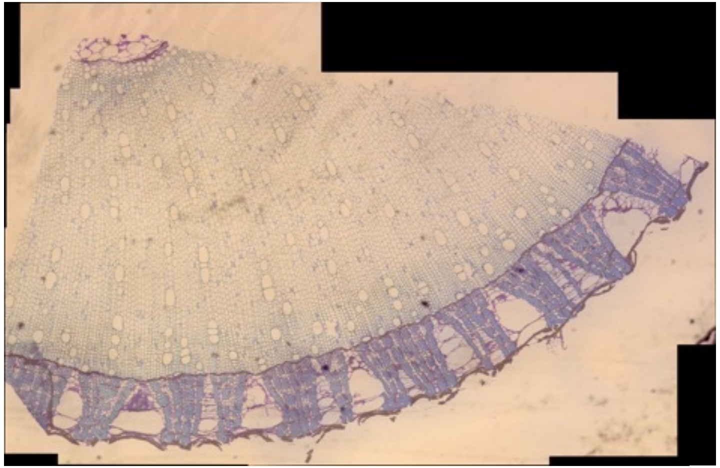
BAMBOO

FLAX



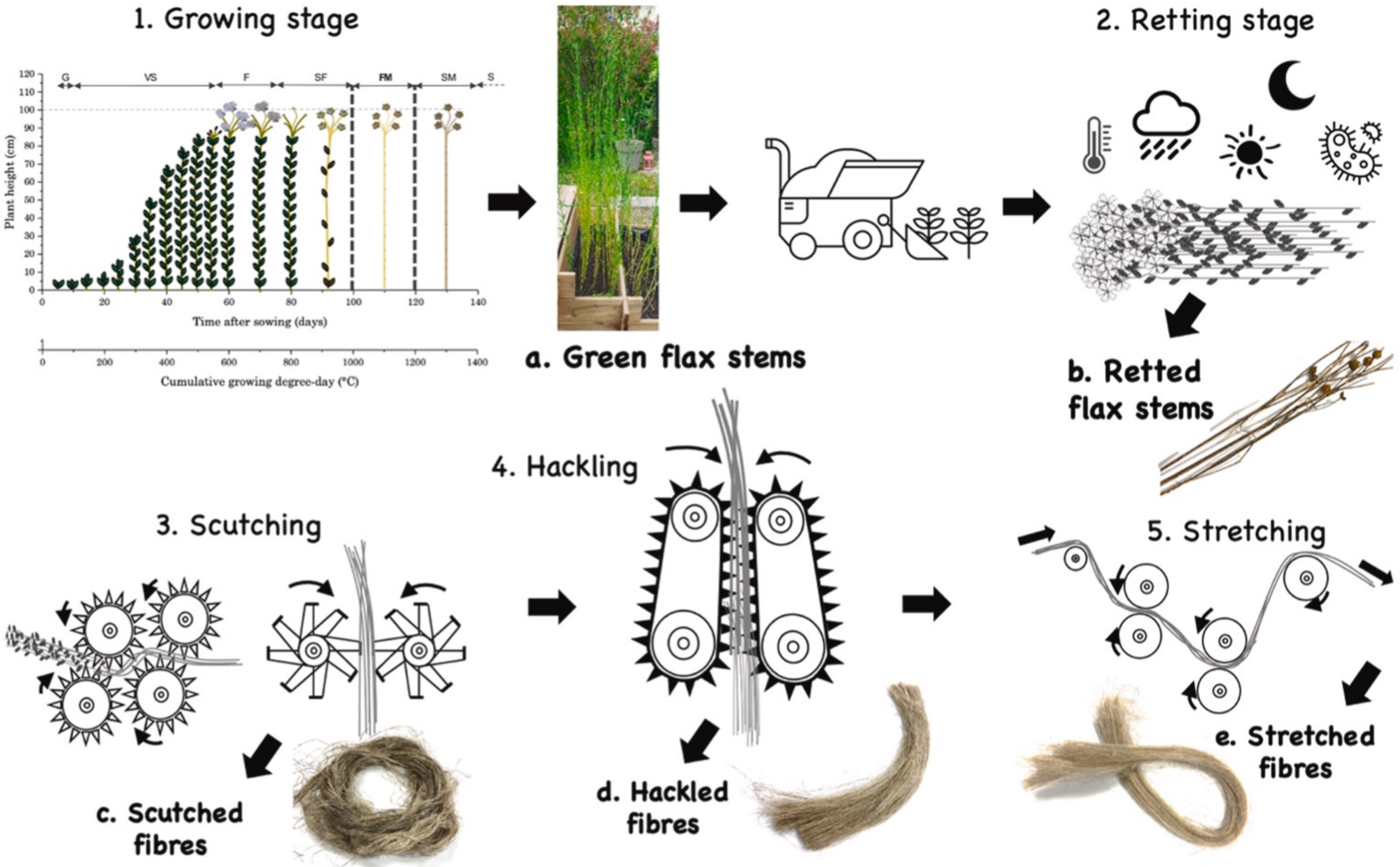


HEMP



JUTE

FIBRE EXTRACTION



FIBRE EXTRACTION

Field Sowing Flowers

Pulling-out Retting

Fibre balls

Scutching

Scutched fibres

FOCUS ON RETTING

FOCUS ON RETTING – EXAMPLE OF HEMP

FOCUS ON RETTING – EXAMPLE OF HEMP

Contents lists available at ScienceDirect

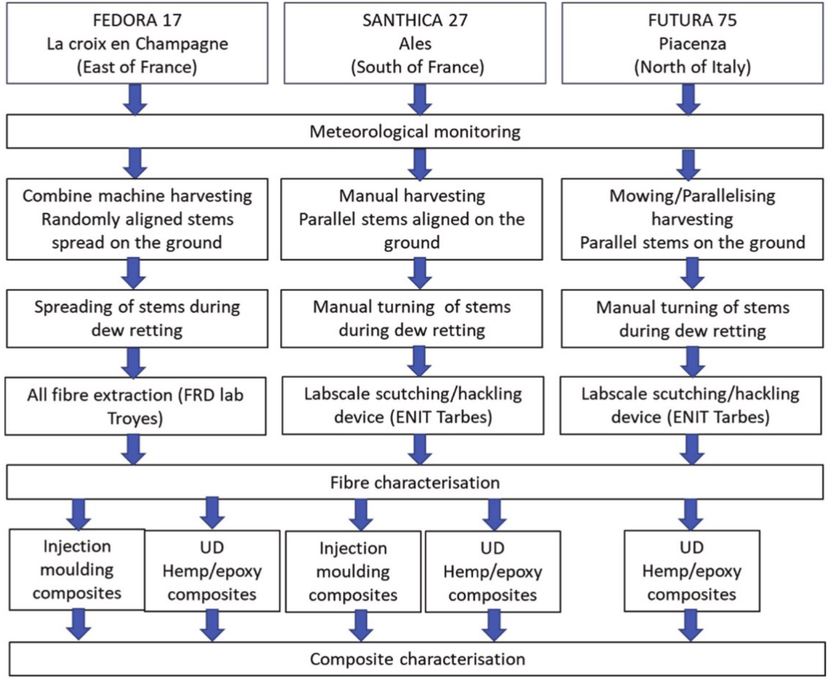
Industrial Crops & Products

journal homepage: www.elsevier.com/locate/indcrop

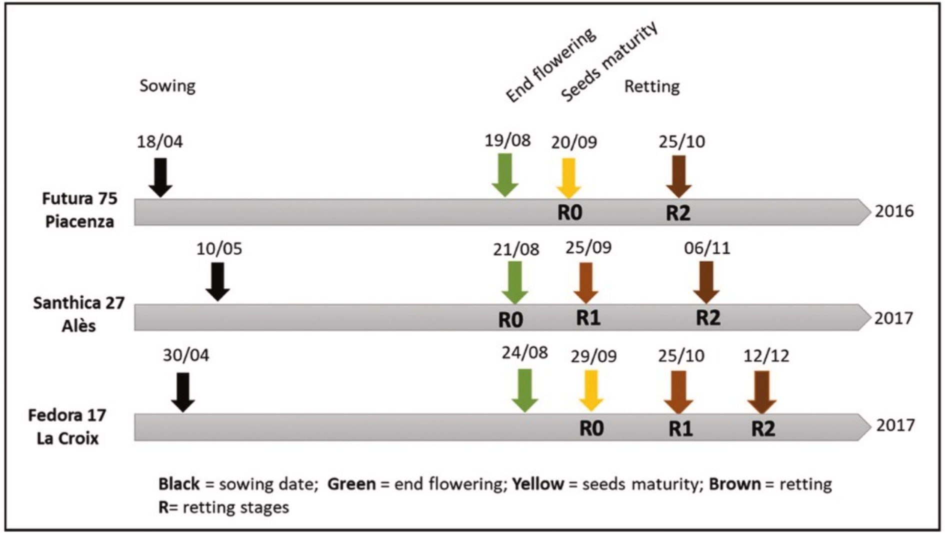
Exploring the dew retting feasibility of hemp in very contrasting European environments: Influence on the tensile mechanical properties of fibres and composites

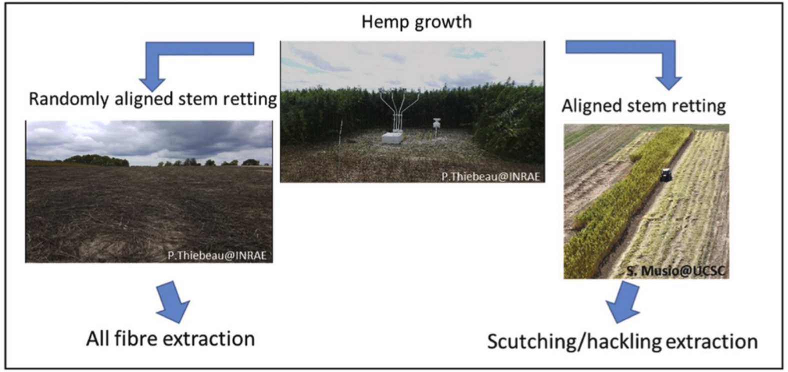
Samuel Réquilé [a], Brahim Mazian [b,c], Marie Grégoire [d], Salvatore Musio [e], Maxime Gautreau [f], Lucile Nuez [a], Arnaud Day [g,h], Pascal Thiébeau [i], Florian Philippe [i], Brigitte Chabbert [i], Anne Chamussy [j], Darshil U. Shah [k], Johnny Beaugrand [f], Vincent Placet [l], Jean-Charles Benezet [b], Antoine le Duigou [a], Mahadev Bar [d], Luc Malhautier [c], Emmanuel De Luycker [d], Stefano Amaducci [e], Christophe Baley [a], Anne Bergeret [b], Alain Bourmaud [a], Pierre Ouagne [d,*]

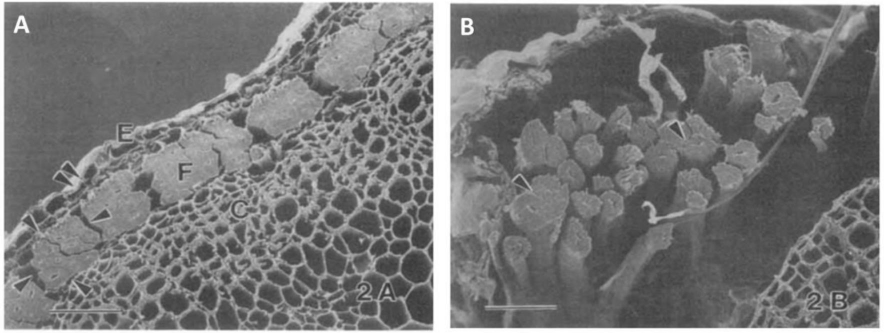
[a] Univ. Bretagne Sud, UMR CNRS 6027, IRDL, F-56100 Lorient, France
[b] Polymers Composites and Hybrids (PCH), IMT Mines Ales, 6 Avenue de Clavieres, 30319, Ales Cedex, France
[c] Laboratoire des Sciences des Risques (LSR), IMT Mines Ales, 6 Avenue de Clavieres, 30319, Ales Cedex, France
[d] Laboratoire Génie de Production, LGP, Université de Toulouse, INP-ENIT, Tarbes, France
[e] Department of Sustainable Crop Production, Università Cattolica del Sacro Cuore, Piacenza, Italy
[f] UR1268 Biopolymères Interactions Assemblages, INRAE, Nantes, France
[g] Fibres Recherche Développement, Technopole de l'Aube en Champagne, Hôtel de Bureaux 2 – 2 rue Gustave Eiffel, CS 90601-10901 Troyes Cedex 9, France
[h] CNRS, UMR8576, UGSF- Unité de Glycobiologie Structurale et Fonctionnelle, Unité de Lille, 59000 Lille, France
[i] Université de Reims Champagne Ardenne, INRAE, FARE, UMR A 614, 51097 Reims, France
[j] La Chanvrière, Rue du Général de Gaulle, CS 20602, 10200, Bar-sur-Aube, France
[k] Centre for Natural Material Innovation, Dept. of Architecture, University of Cambridge, Cambridge CB2 1PX, United Kingdom
[l] FEMTO-ST Institute, UFC/CNRS/ENSMM/UTBM, Université Bourgogne Franche-Comté, Besançon, France



FOCUS ON RETTING – EXAMPLE OF HEMP



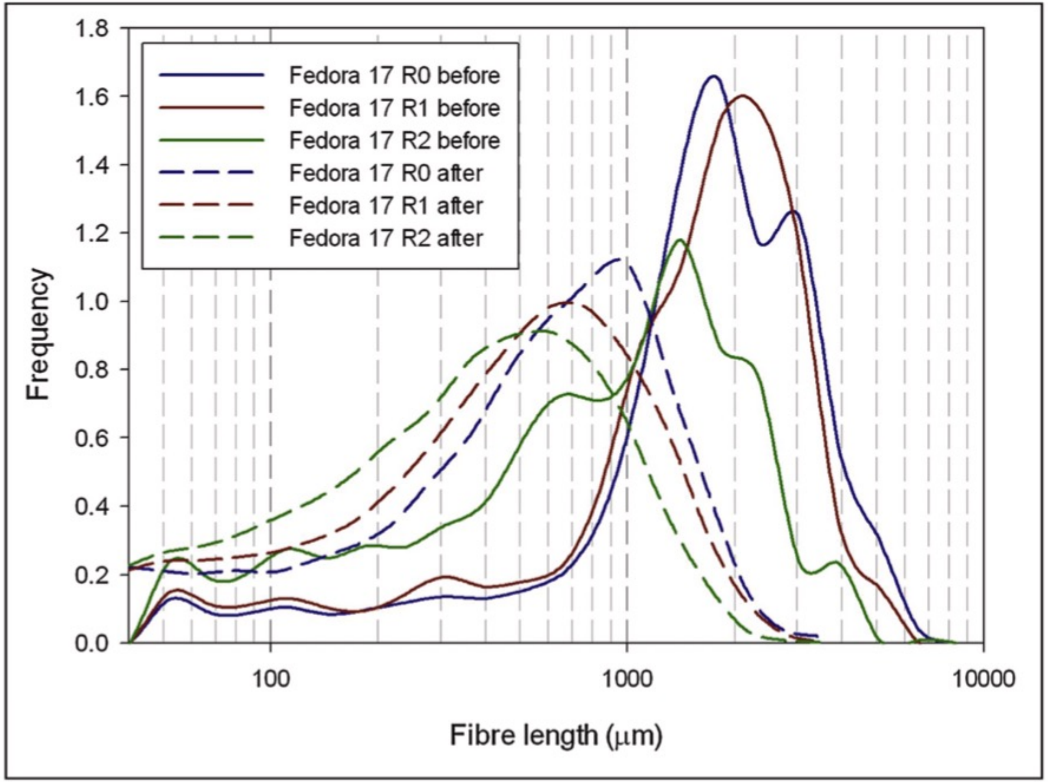


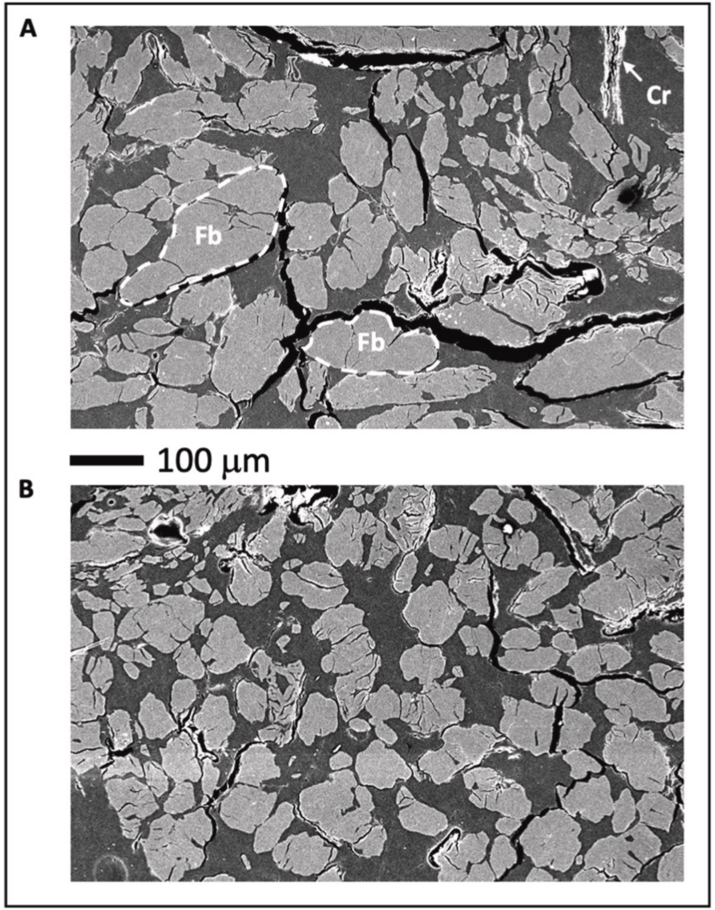


(a) Hemp extracted fibres at the Output of FRD line

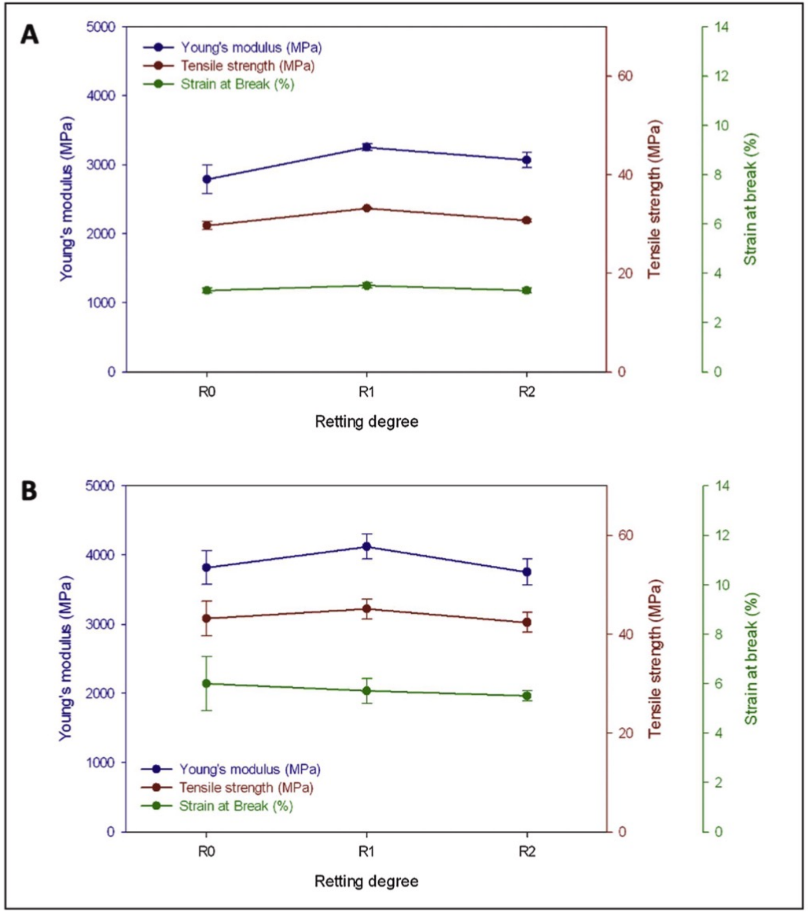
(b) Hemp extracted fibres at the output of the labscale scutching/hackling line

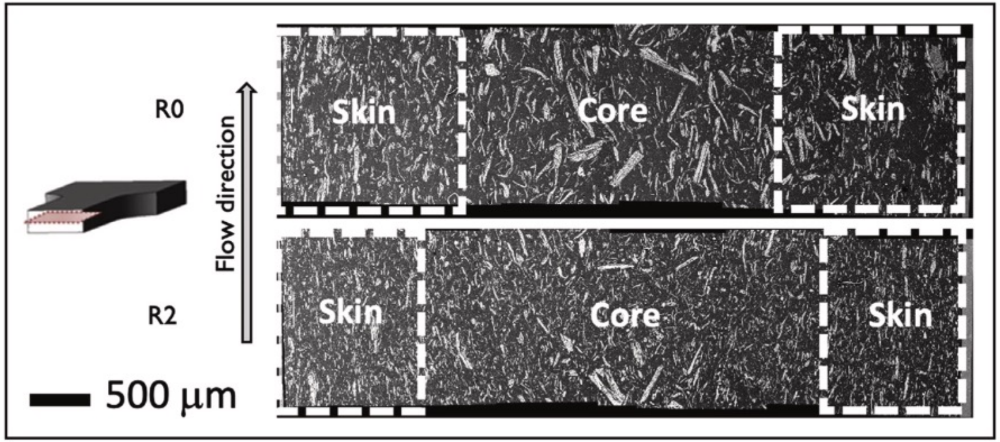
FOCUS ON RETTING – EXAMPLE OF HEMP





FOCUS ON RETTING – EXAMPLE OF HEMP





FOCUS ON RETTING – EXAMPLE OF FLAX

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

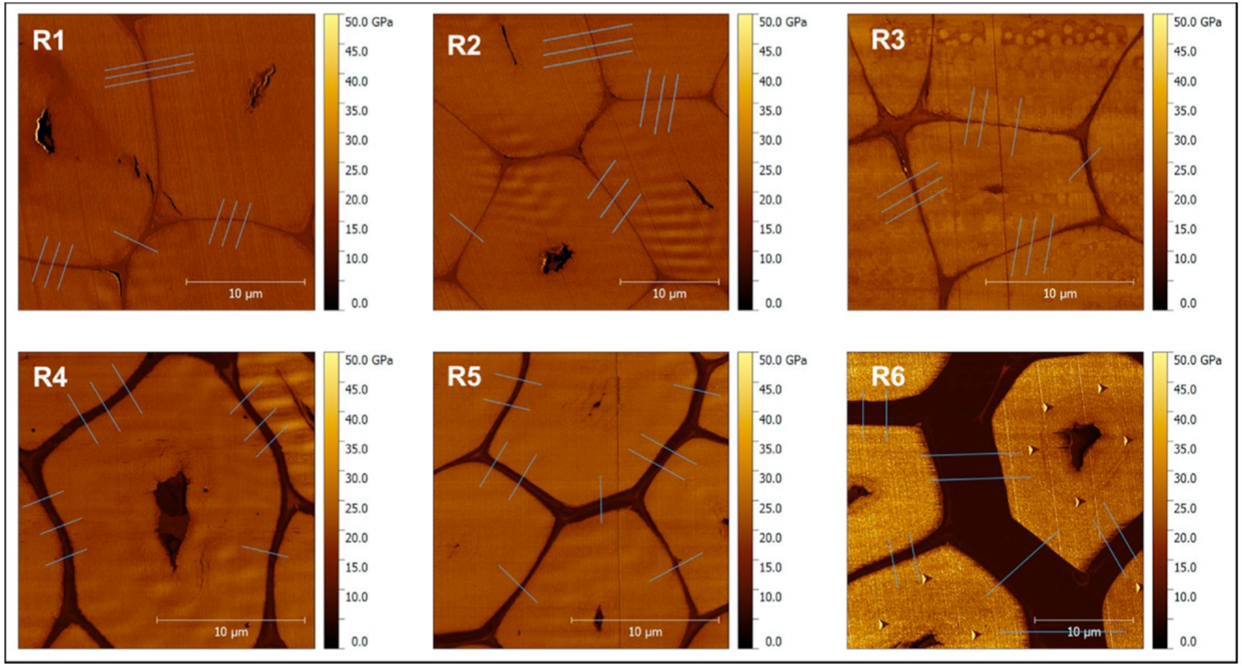
ELSEVIER

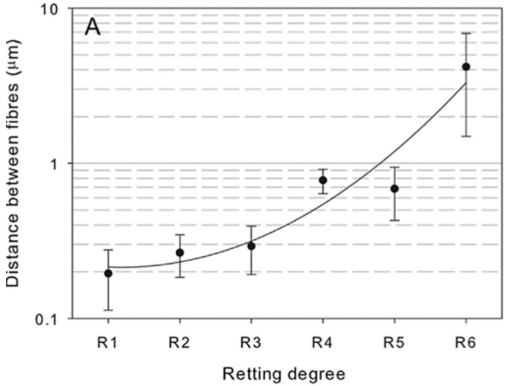
Evolution of flax cell wall ultrastructure and mechanical properties during the retting step

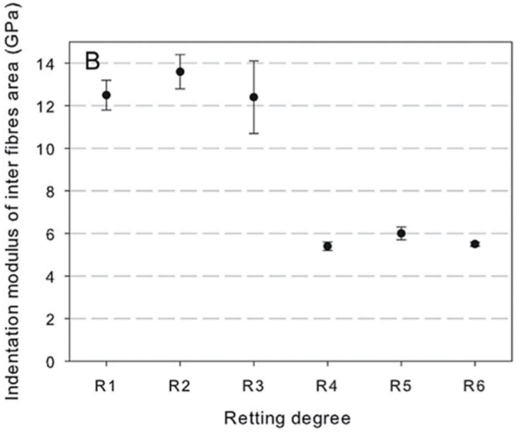
Alain Bourmaud[a,*], David Siniscalco[a], Loïc Foucat[b], Camille Goudenhooft[a], Xavier Falourd[b], Bruno Pontoire[b], Olivier Arnould[c], Johnny Beaugrand[b], Christophe Baley[a]

[a] *IRDL, Université Européenne Bretagne, CNRS, UMR 6027, Lorient, France*
[b] *UR1268 Biopolymères Interactions Assemblages, INRA, Nantes, France*
[c] *LMGC, Université de Montpellier, CNRS, UMR 5508, Montpellier, France*

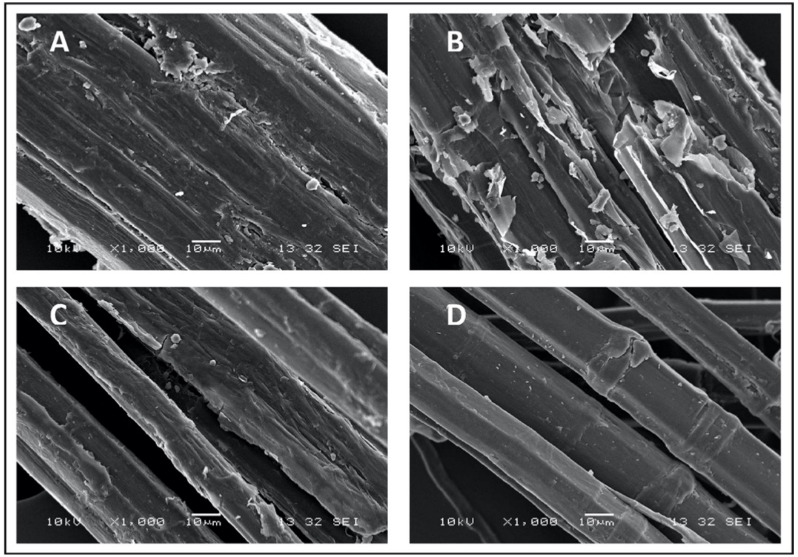
FOCUS ON RETTING – EXAMPLE OF FLAX

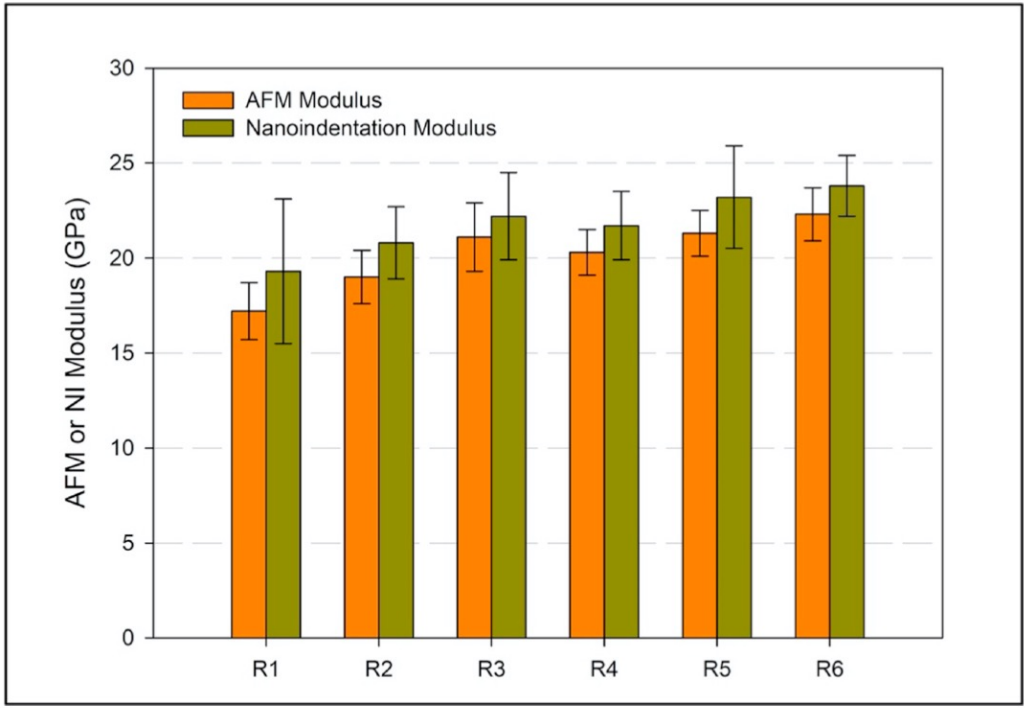




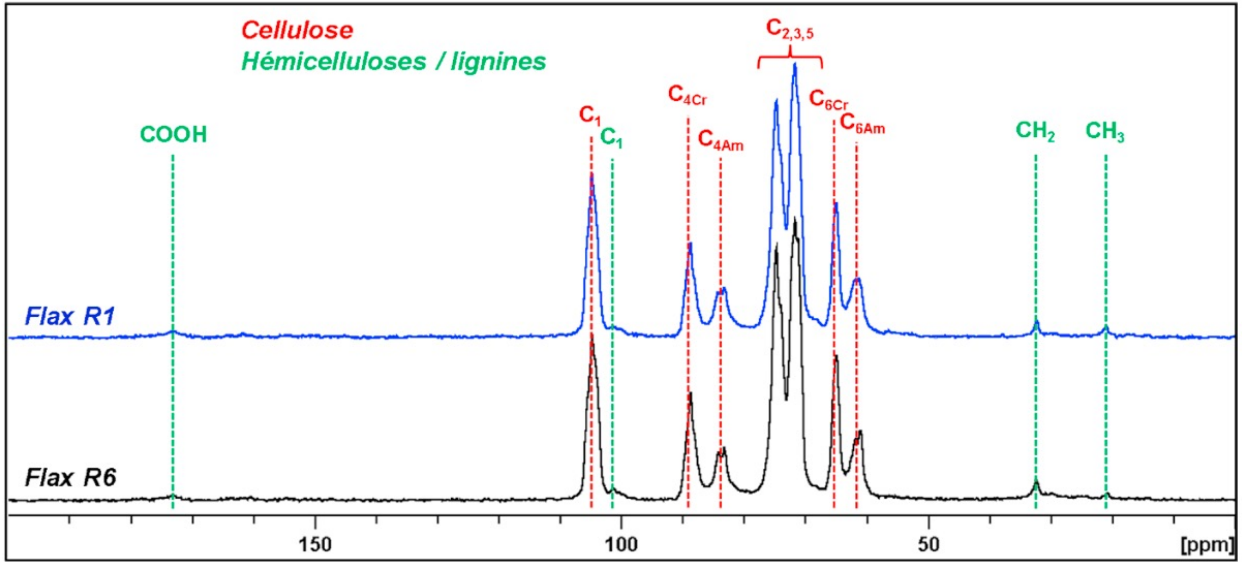


FOCUS ON RETTING – EXAMPLE OF FLAX



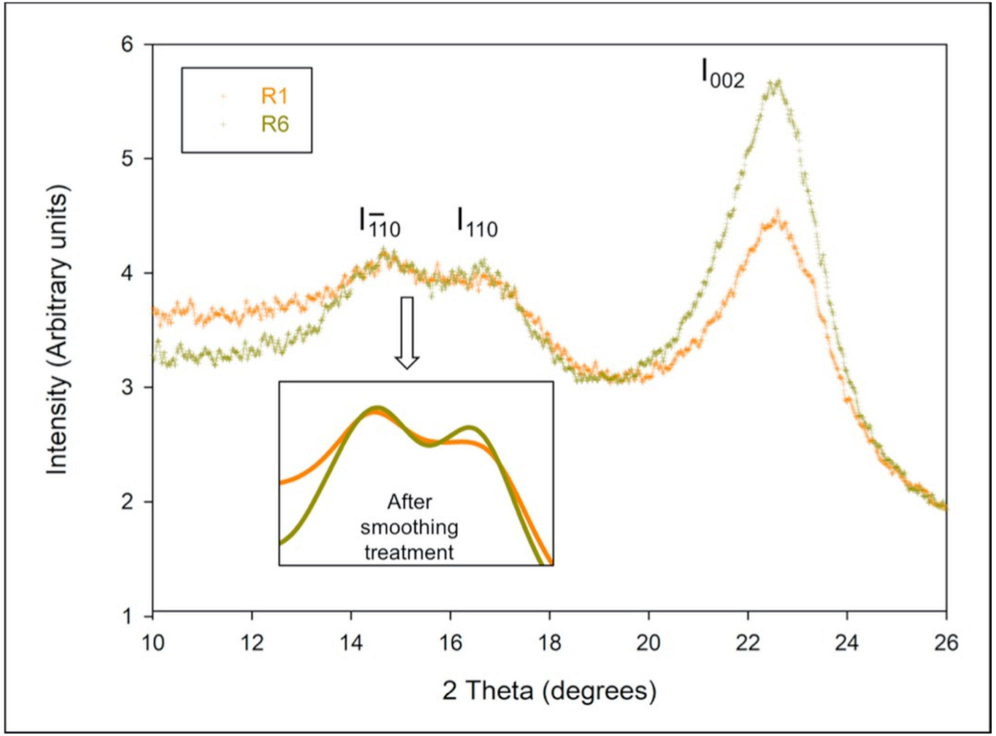


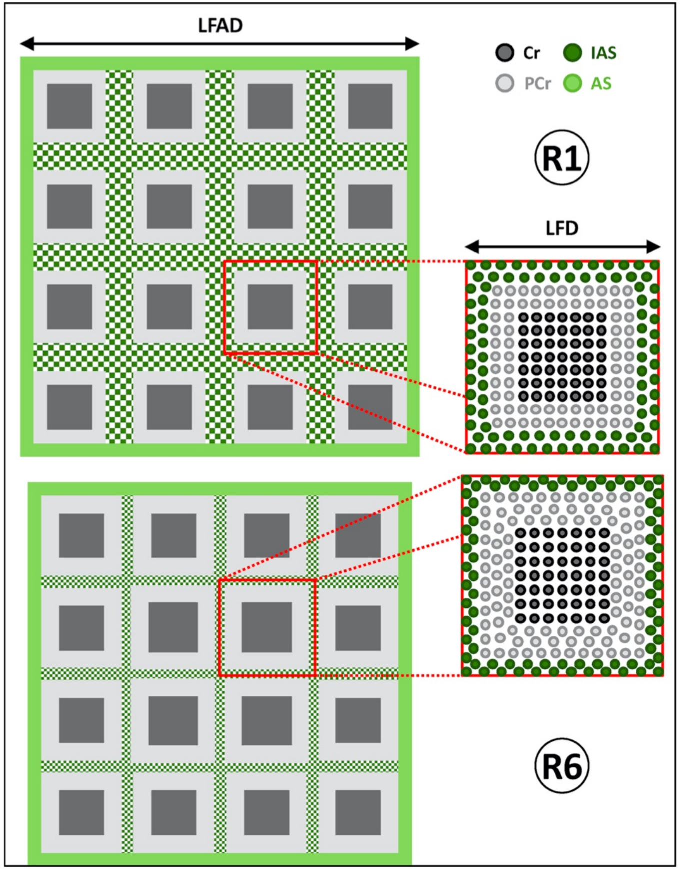
FOCUS ON RETTING – EXAMPLE OF FLAX



	Peak assignment							
	Cr(Iα)	Cr(Iα + β)	PCr	Cr(Iβ)	AS	IAS	AS	
R1	89.37	88.73	88.58	87.83	84.22	83.53	83.16	δ (ppm)
R6	89.38	88.70	88.54	87.68	84.14	83.68	83.16	
R1	81	60	199	90	98	482	86	FWHH (Hz)
R6	76	55	197	90	88	484	76	
R1	8	10	32	2	6	35	7	Normalized
R6	7	10	37	2	7	31	7	area (%)

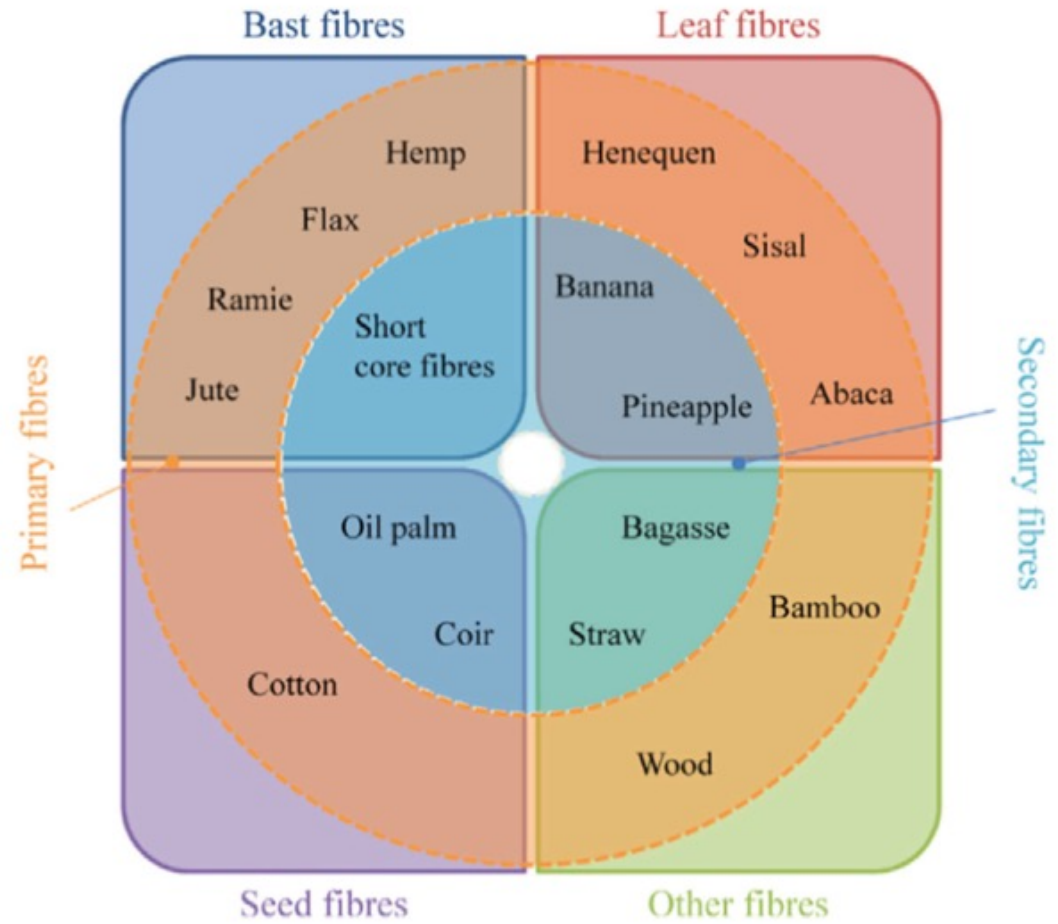
FOCUS ON RETTING – EXAMPLE OF FLAX





A DIVERSITY OF PLANT FIBRES

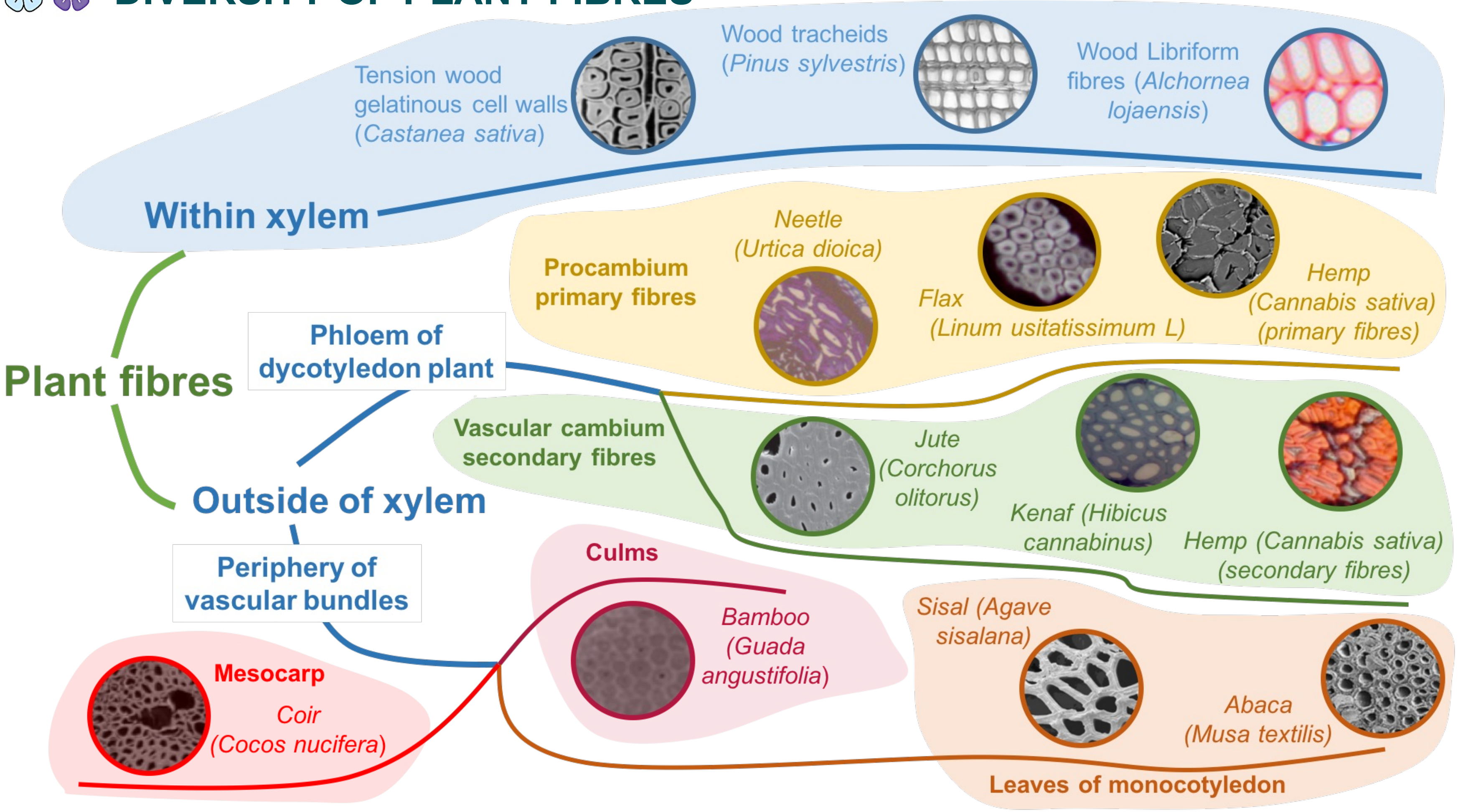
DIVERSITY OF PLANT FIBRES



Specific functions according to the fibre location within the plant

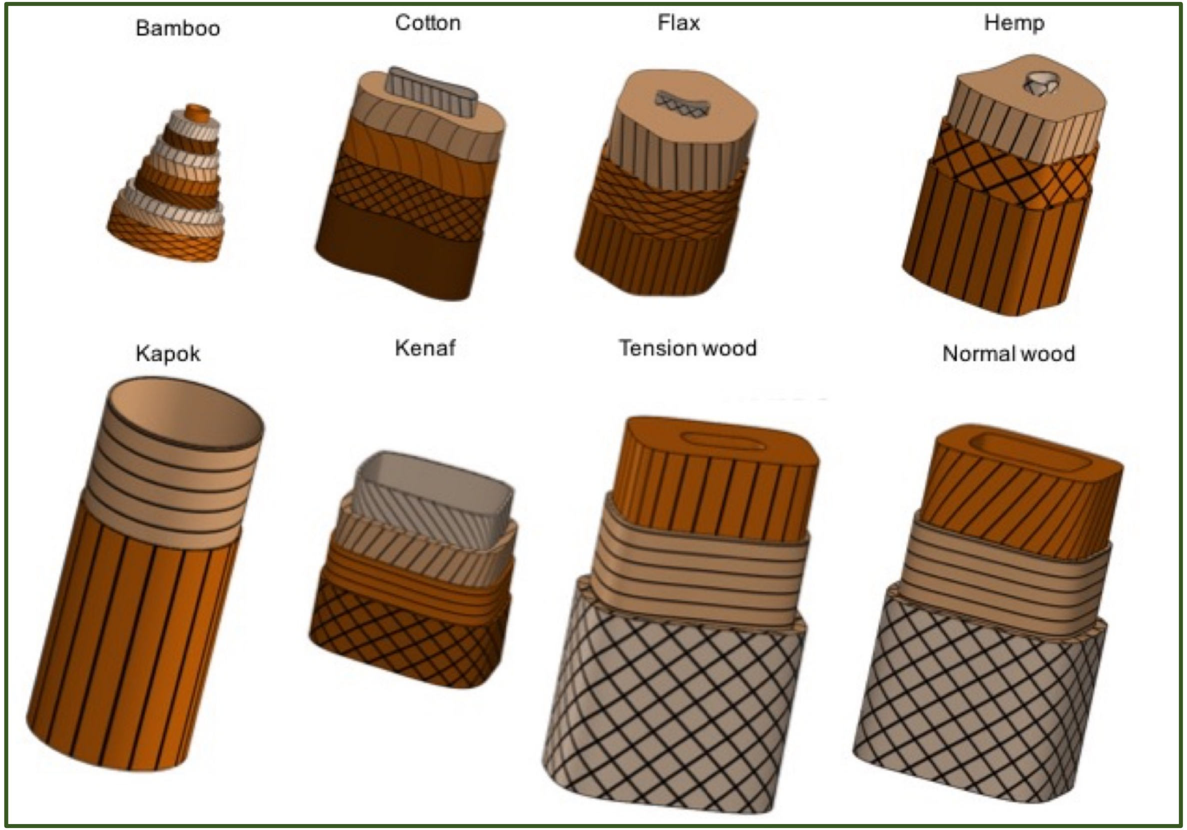
Shah, Materials & Design, 2014

DIVERSITY OF PLANT FIBRES



Bourmaud, Beaugrand, Shah, Placet, Baley, PMS, 2018

DIVERSITY OF PLANT FIBRES



Different morphologies

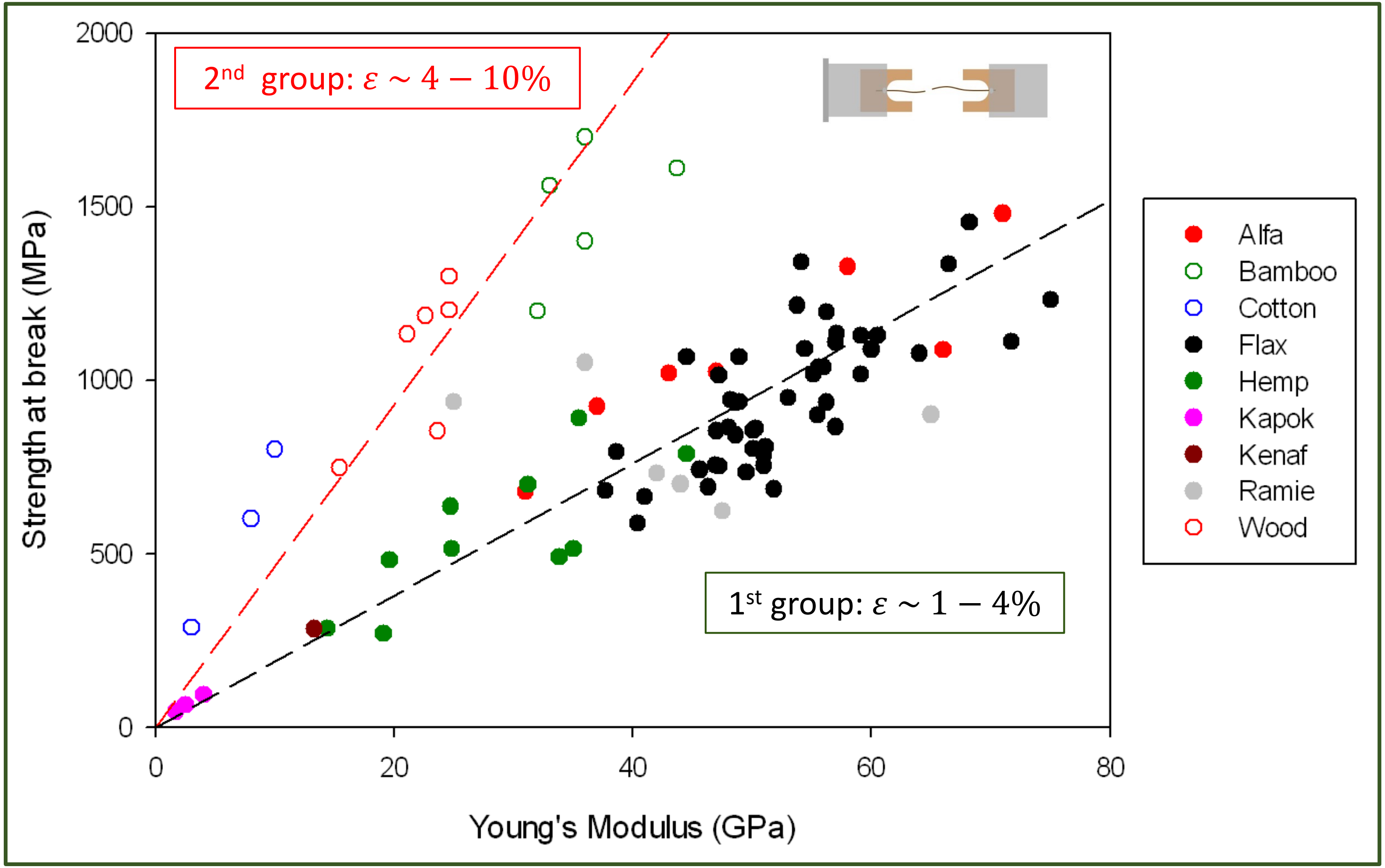
In link with function and cell wall development

Differences in length or stiffness

Filling rate, lumen size

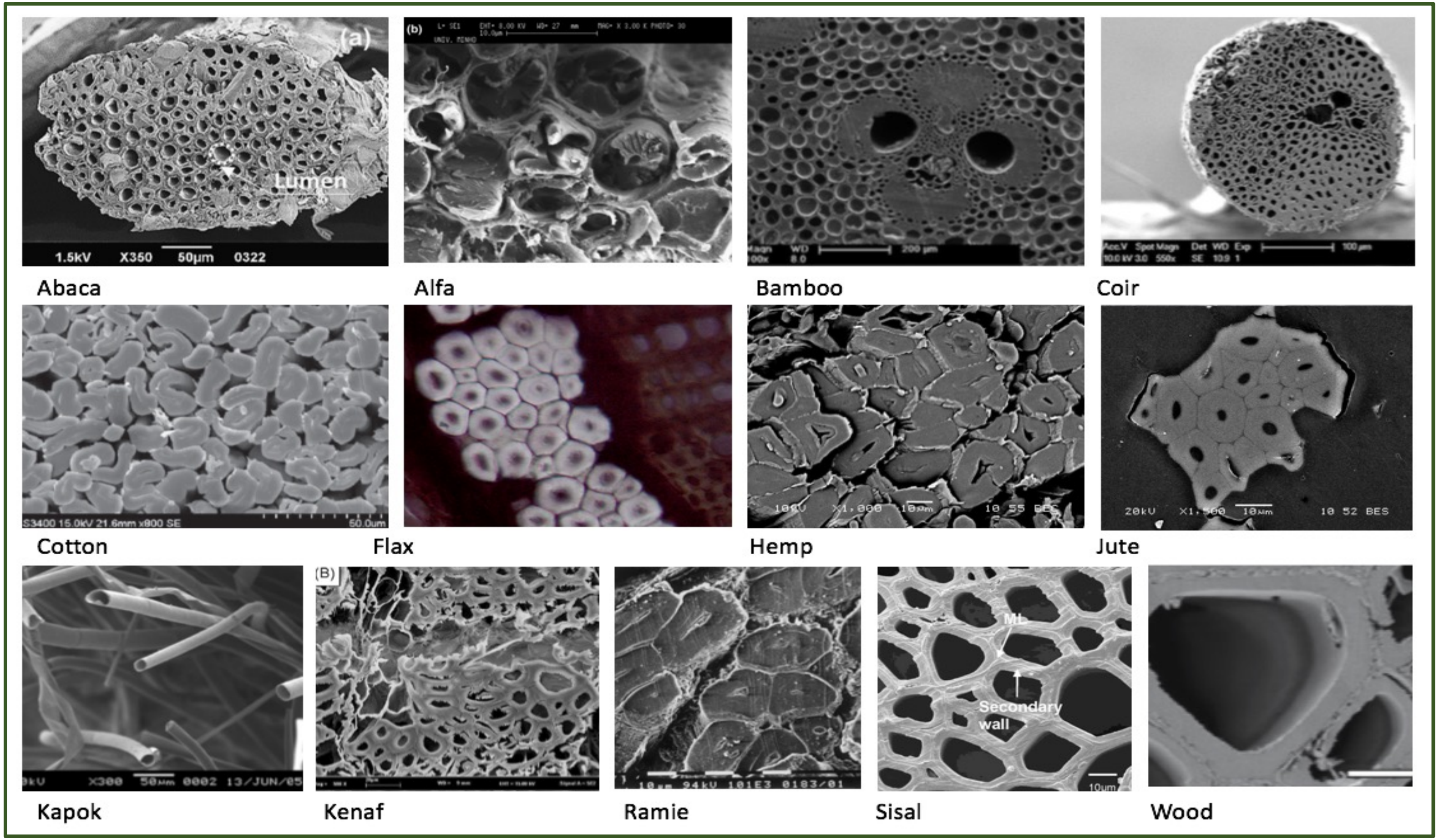
Bourmaud, Beaugrand, Shah, Placet, Baley, PMS, 2018

DIVERSITY OF PLANT FIBRES



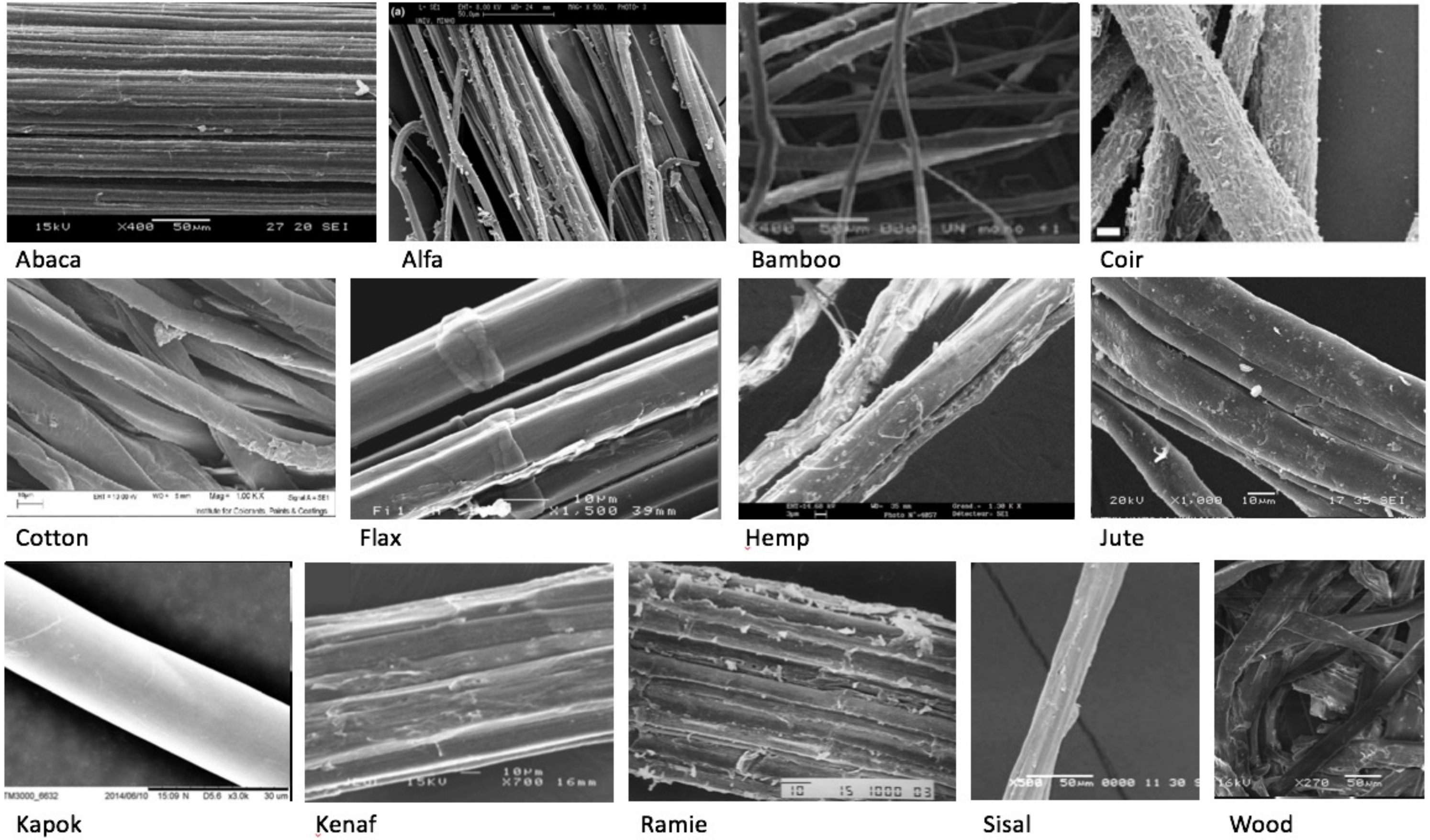
Bourmaud, Beaugrand, Shah, Placet, Baley, PMS, 2018

DIVERSITY OF PLANT FIBRES



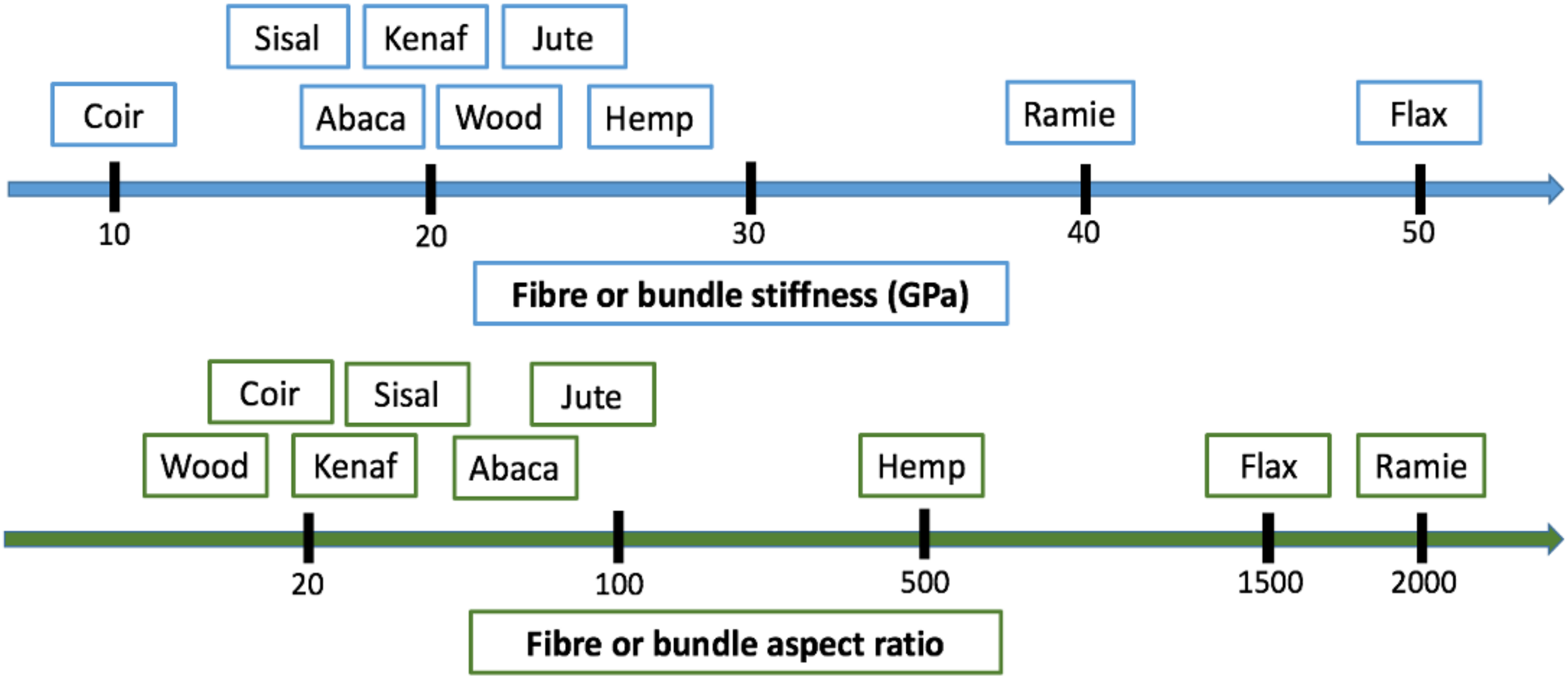
Bourmaud, Beaugrand, Shah, Placet, Baley, PMS, 2018

DIVERSITY OF PLANT FIBRES



Bourmaud, Beaugrand, Shah, Placet, Baley, PMS, 2018

DIVERSITY OF PLANT FIBRES



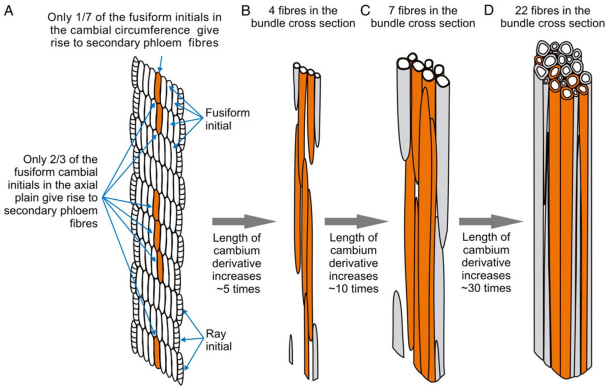
Bourmaud, Beaugrand, Shah, Placet, Baley, PMS, 2018

DIVERSITY OF PLANT FIBRES

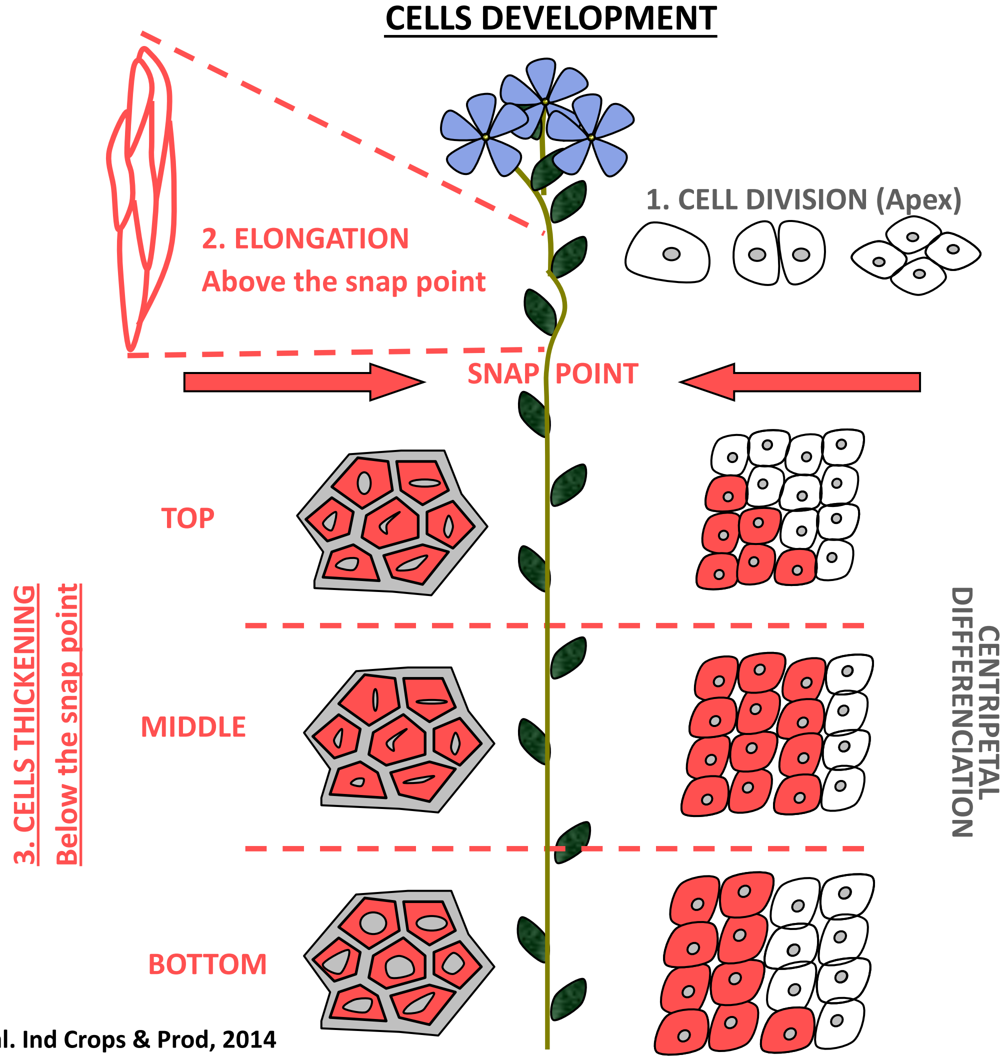
- Plant fibres are all different
- One plant fibre = specific intrinsec properties and potential different application
- One is not necessary better than another
- For their use, consider origin (LCA), competition with food, technical interest....
- Price and available volumes are also key-points

PLANT FIBRES DEVELOPMENT AND ULTRASTRUCTURE

CELL WALL DEVELOPMENT

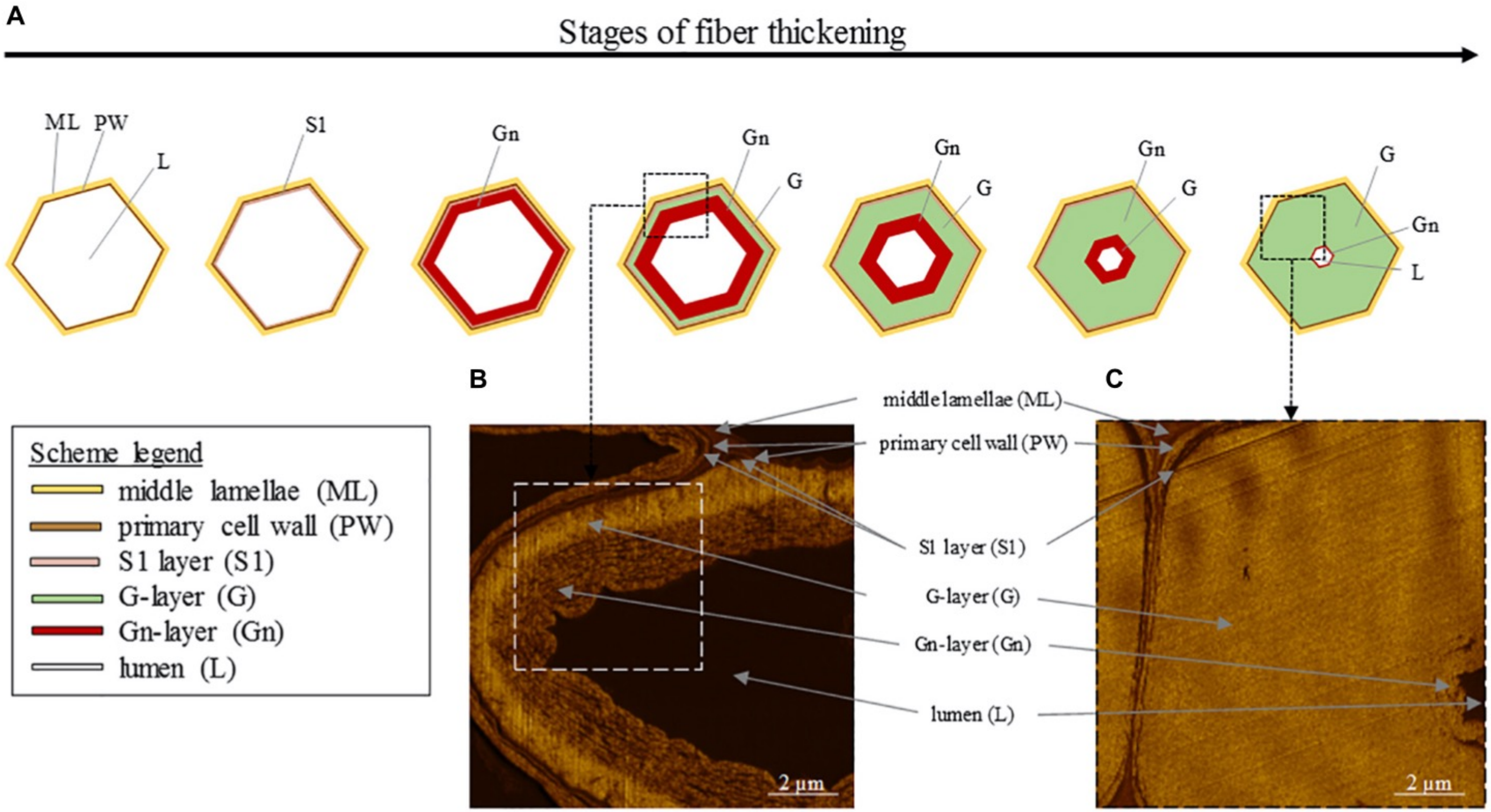


Snegireva 2015



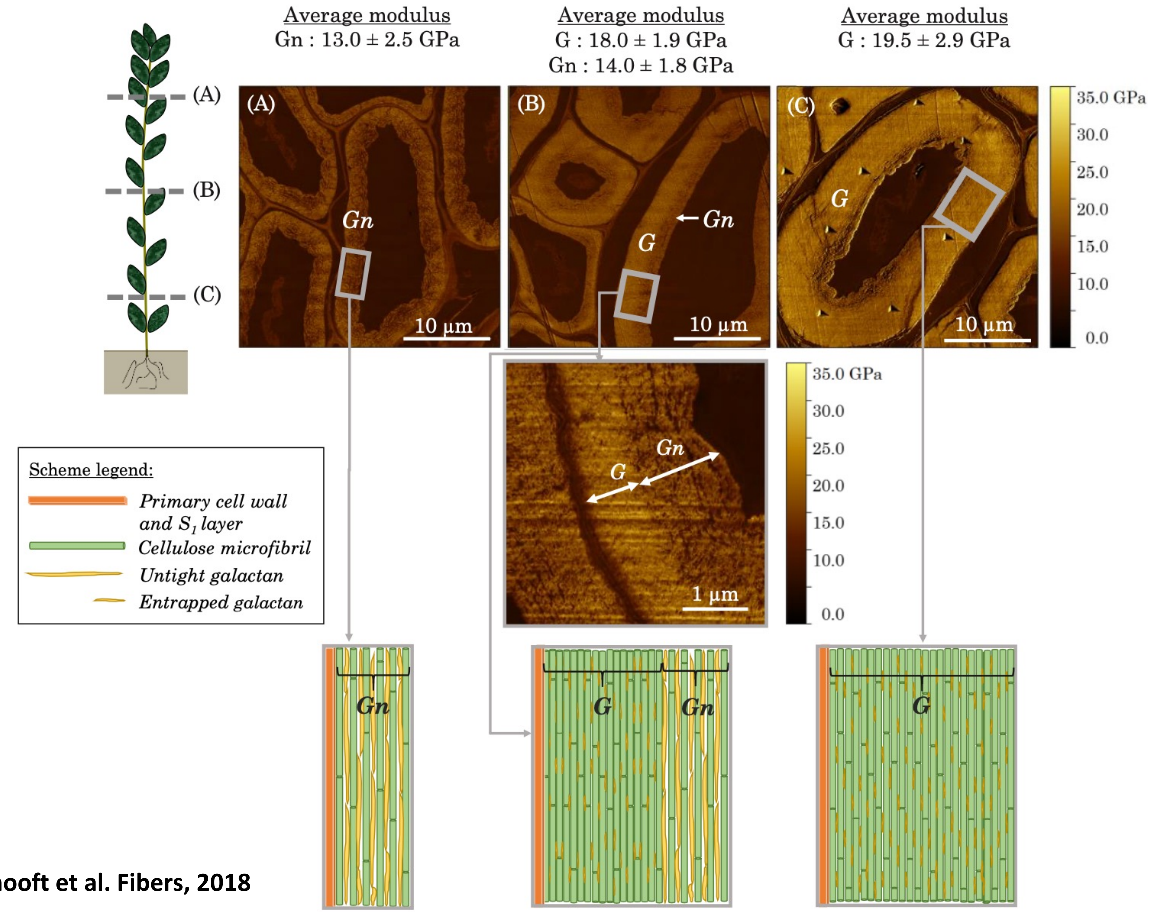
Bourmaud et al. Ind Crops & Prod, 2014

DEVELOPEMENT AND STRUCTURE OF FIBRES



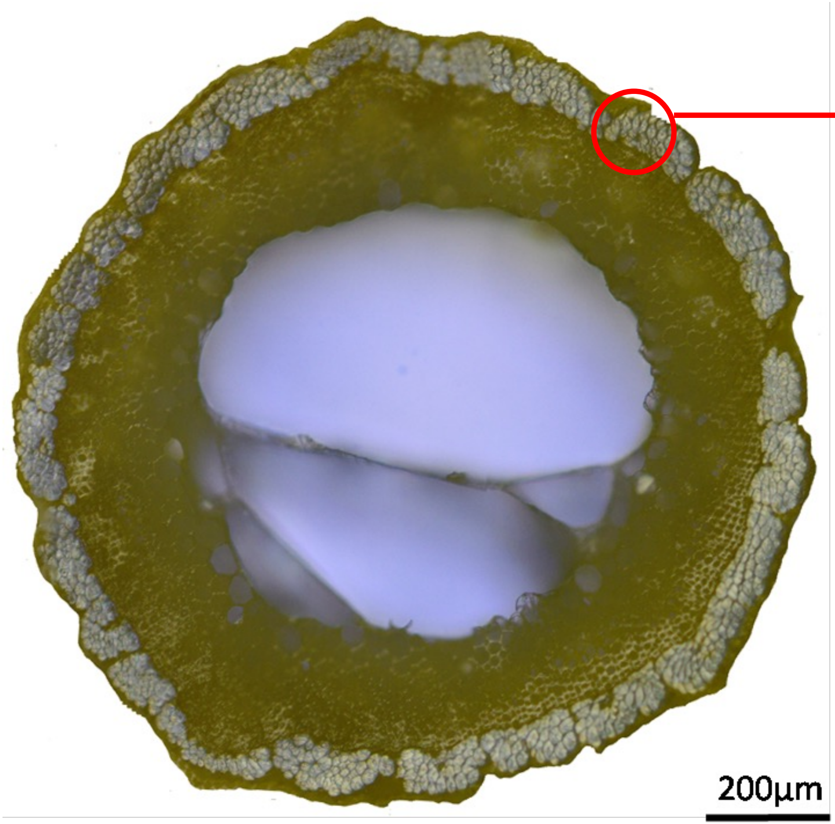
Goudenhooft et al. Frontiers in Plant Science, 2019

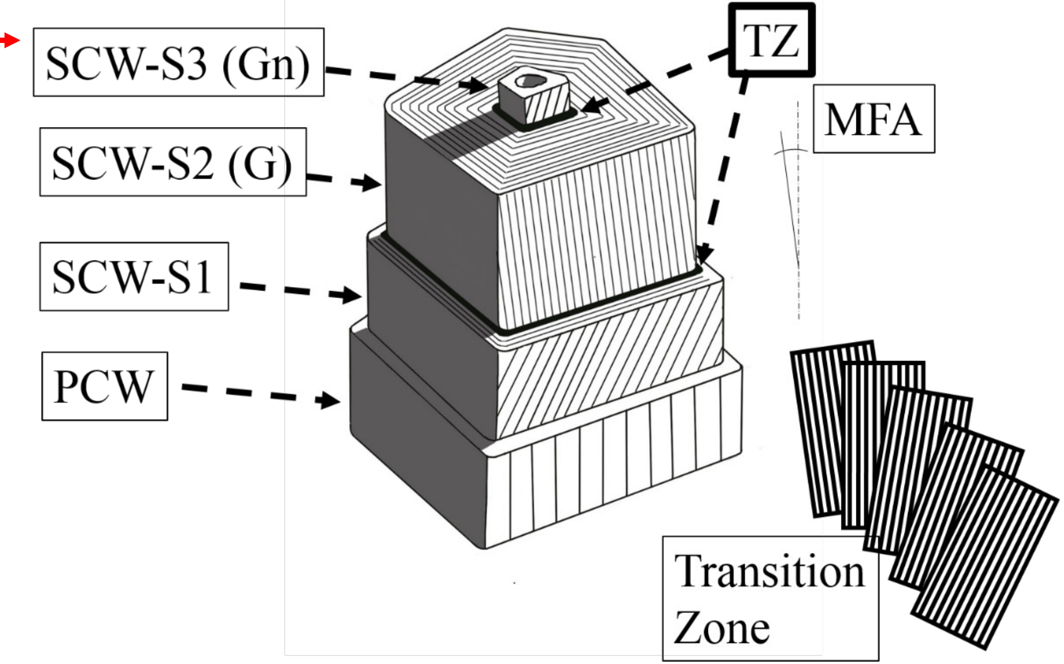
DEVELOPEMENT AND STRUCTURE OF FIBRES



Goudenhooft et al. Fibers, 2018

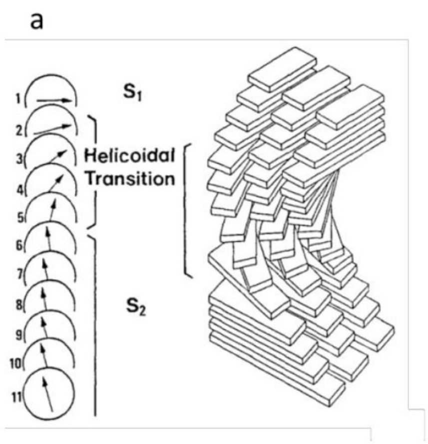
DEVELOPMENT AND STRUCTURE OF FIBRES

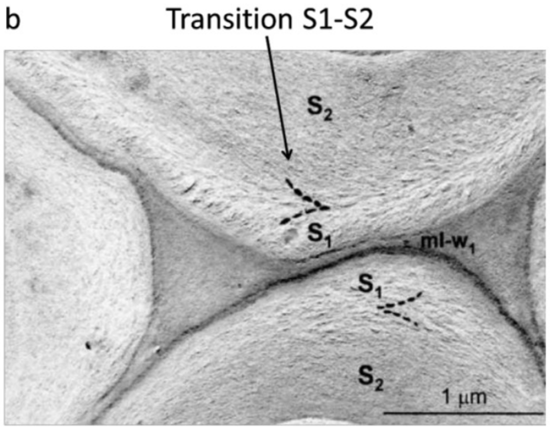


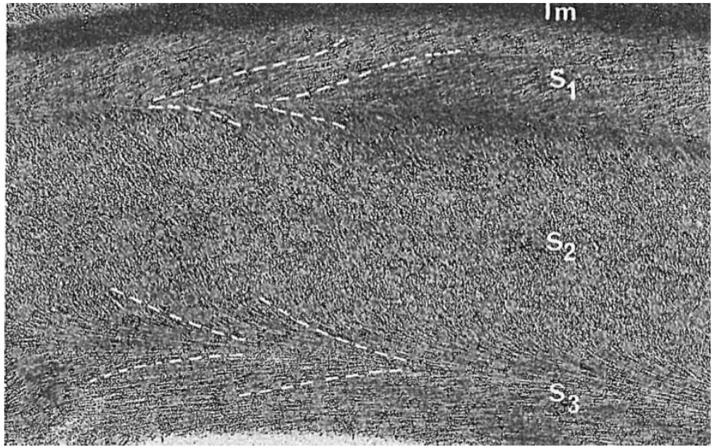


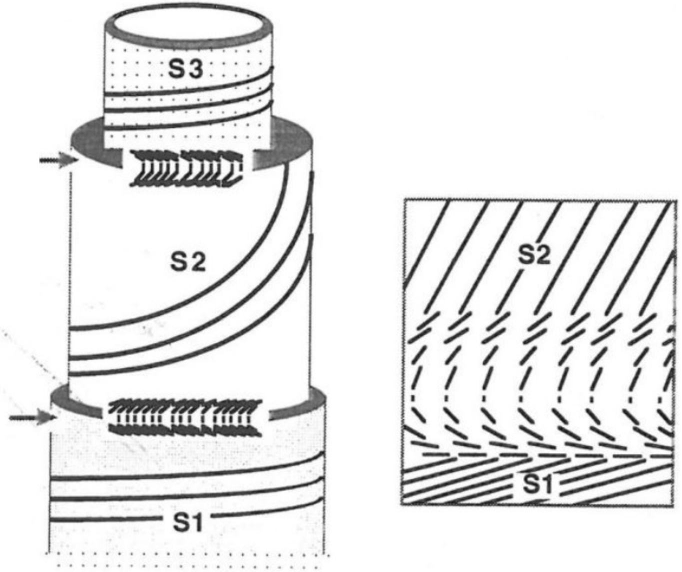
Baley et al. Bioinsp & Biom, 2018

DEVELOPMENT AND STRUCTURE OF FIBRES



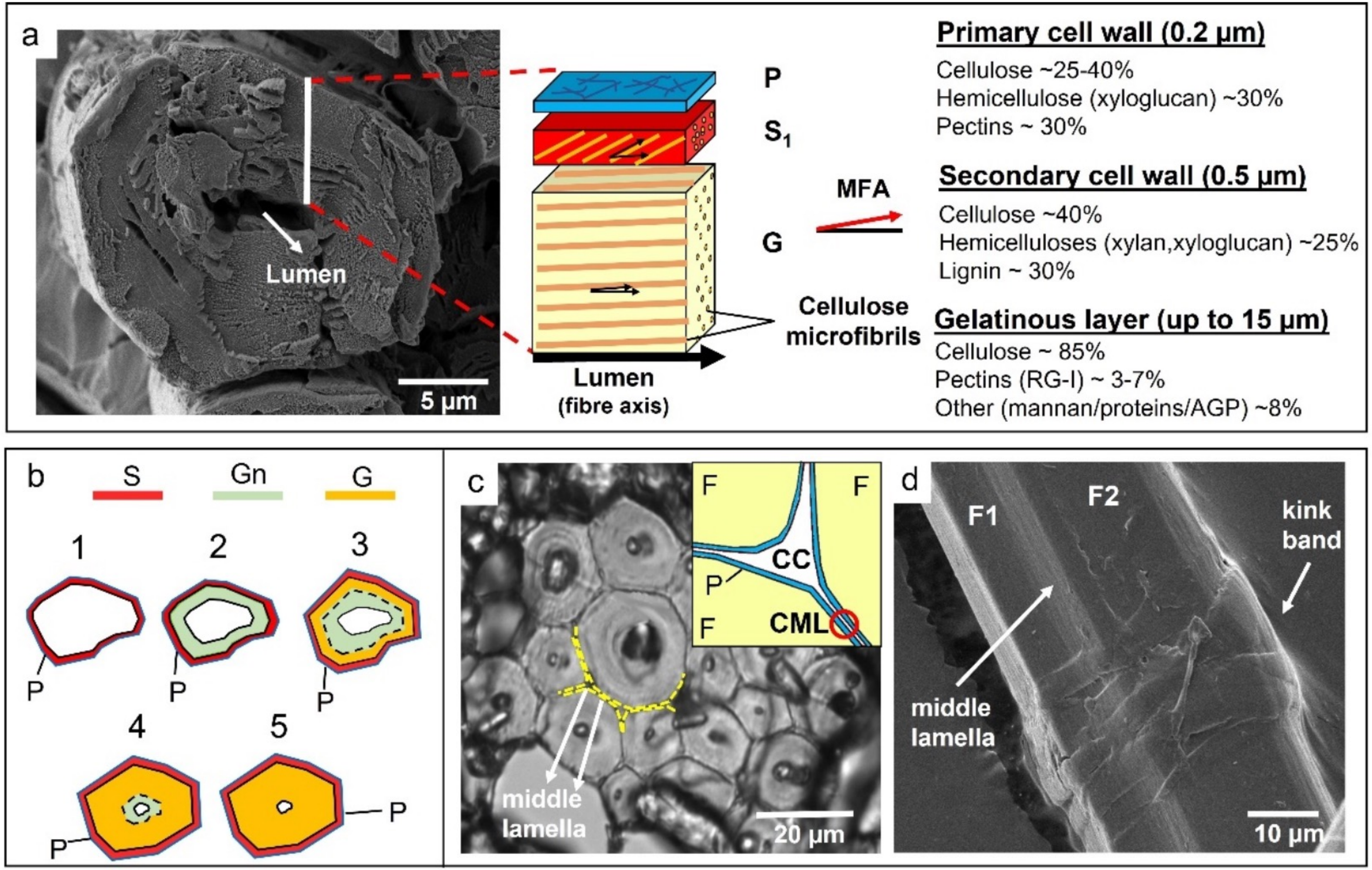


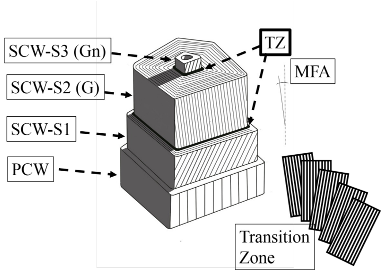




Roland et al. 1995

DEVELOPMENT AND STRUCTURE OF FIBRES



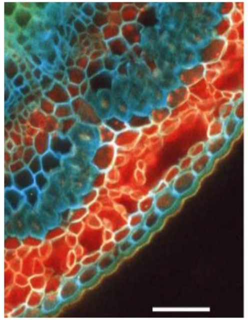


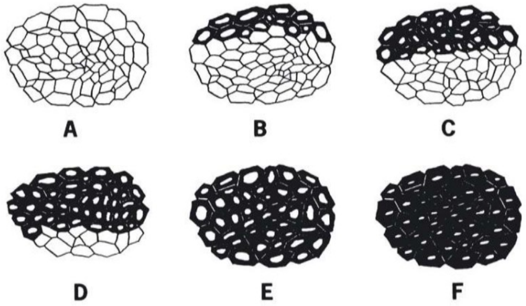
Baley et al. Bioinsp & Biom, 2018

Melelli et al, Carbo Poly, 2022

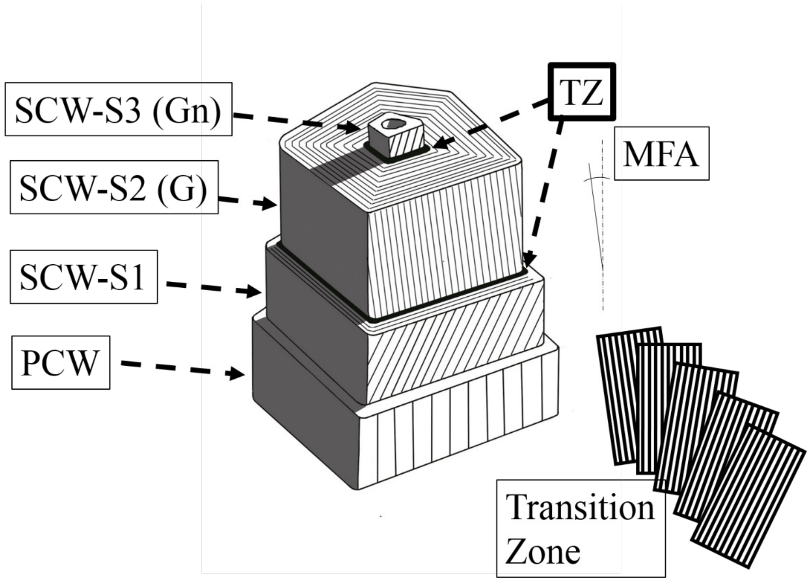
PLANT FIBRES: KEY POINTS

- Flax fibres are long and poly nucleated cells
- From 15 to 80 mm !
- Filled with cellulose (Approx 80%)
- Highly crystalline
- A porosity inside: 0-5%





BIOCHEMICAL ARCHITECTURE OF PLANT CELL WALLS



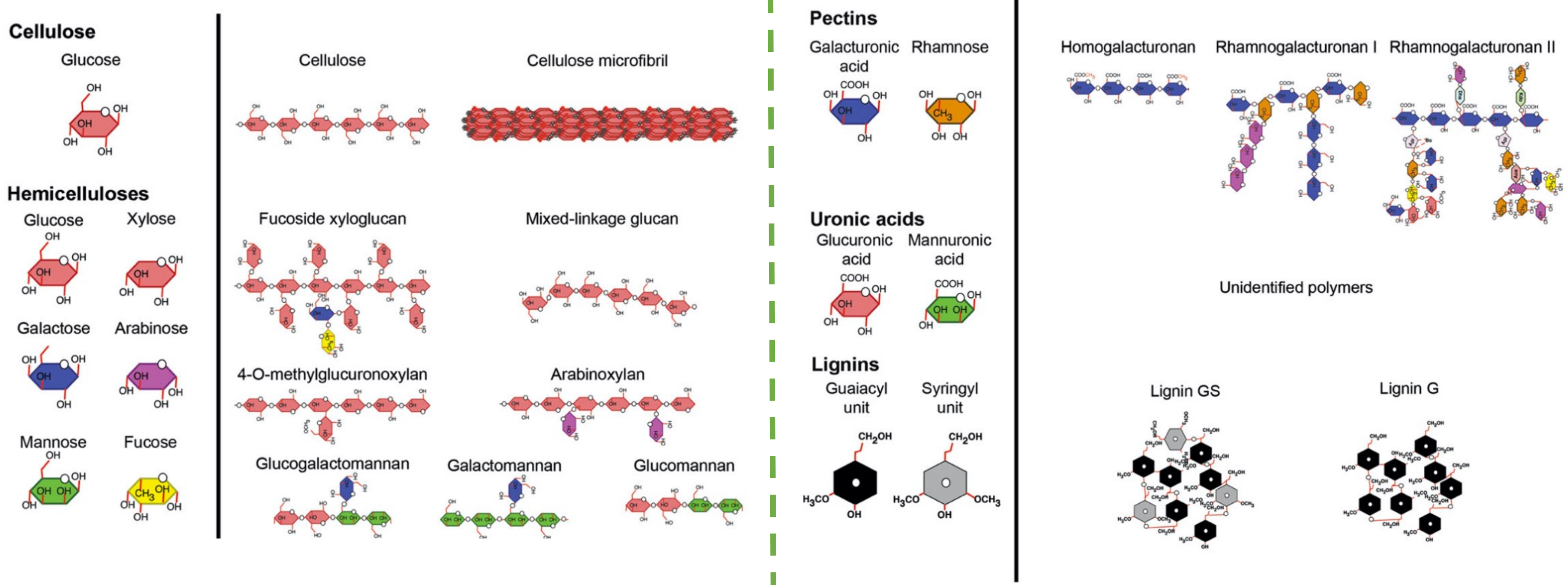
Cell wall layer	Average thickness	Microfibrils orientation	Approximate composition
PW	0.2 µm [87]	disperse orientation, preferentially 0° [70,72]	~25-40% cellulose ~30% hemicelluloses (mainly xyloglucan and lesser amounts of arabinoxylan) ~30% pectins (mainly homogalacturonan; possibly rhamnogalacturonan (RG) I; RG II and arabinogalactan) [53,70,83,88–90]
S1	0.5 µm [73,91]	60-80° [92]	~30-50% cellulose ~30% hemicelluloses (xylan, xyloglucan) ~5% pectins (homogalacturonan and RG I) ~10-20% lignin [70,72,78,89]
G	up to 15 µm or 90% of the total cell wall area at maturity [71,83]	8-10° [91,93]	~75-90% cellulose ~15-20% hemicelluloses (glucomanan) ~5-10% pectins (RG I) [53,71,72,83,89]
Gn	0.5-1 µm through thickening [79,91]	loosely packed as an heterogeneous structure [94]	cellulose hemicelluloses (glucomanan) pectins (nascent RG I (i.e. long galactan chains)) [71,72,78,94]

Baley et al. Bioinsp & Biom, 2018; Goudenhooft et al, Frontiers, 2019

BIOCHEMICAL ARCHITECTURE OF PLANT CELL WALLS

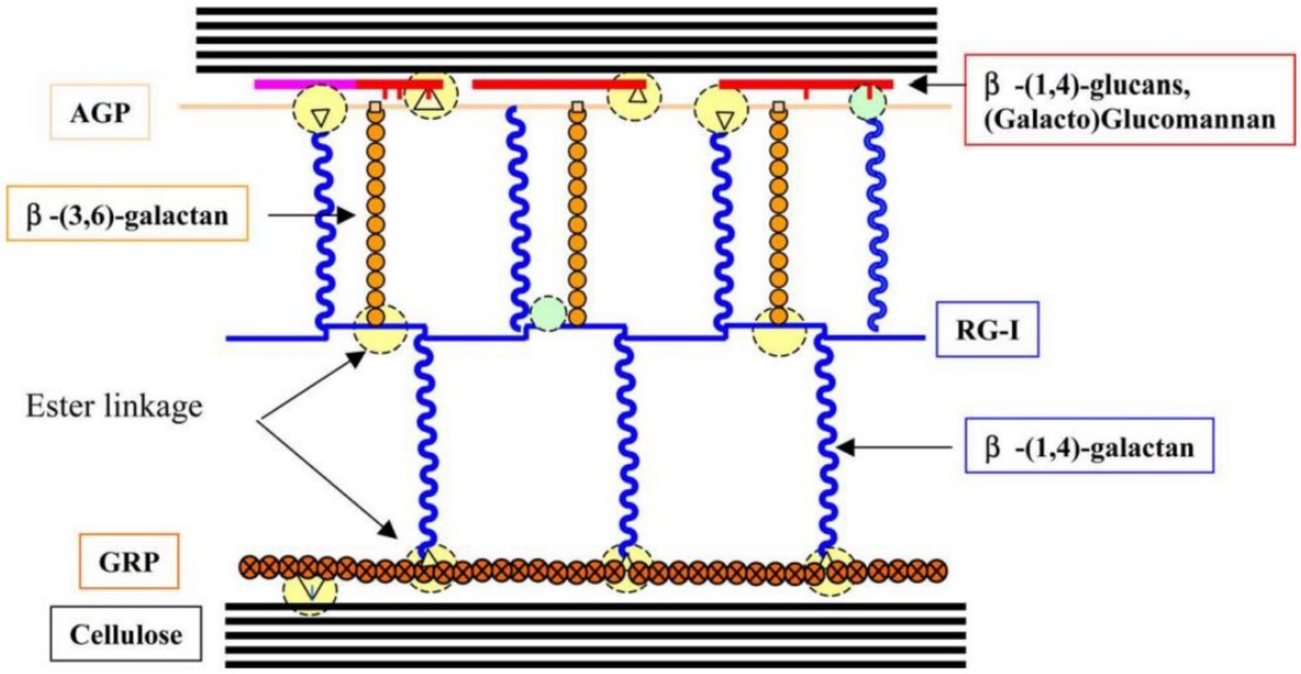
Cellulose / Hemicelluloses

Pectins, UA & Lignins



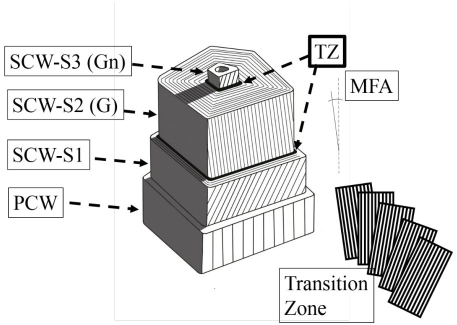
Sarkar et al. J Ex Bott, 2009

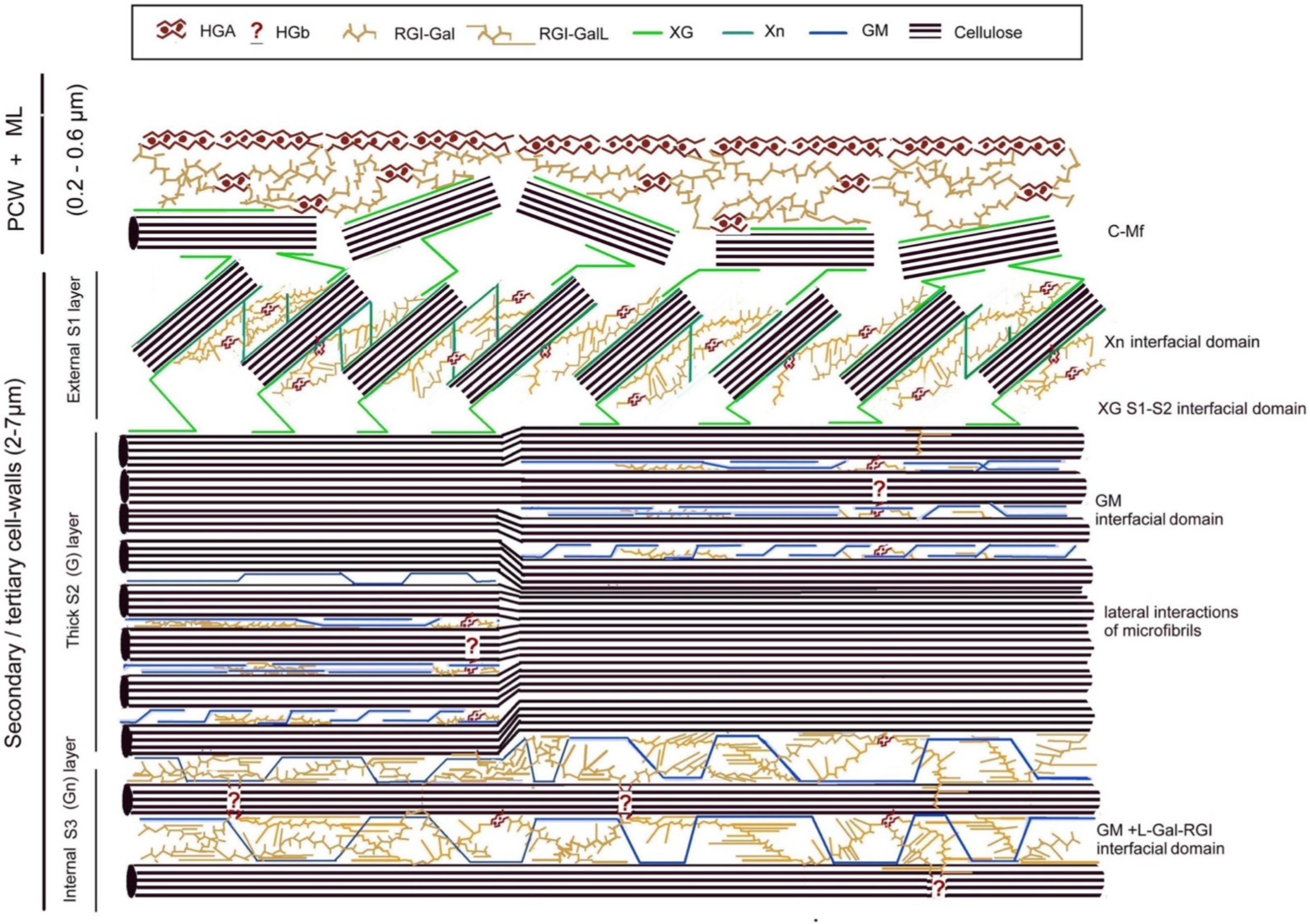
BIOCHEMICAL ARCHITECTURE OF PLANT CELL WALLS



Morvan et al., 2003

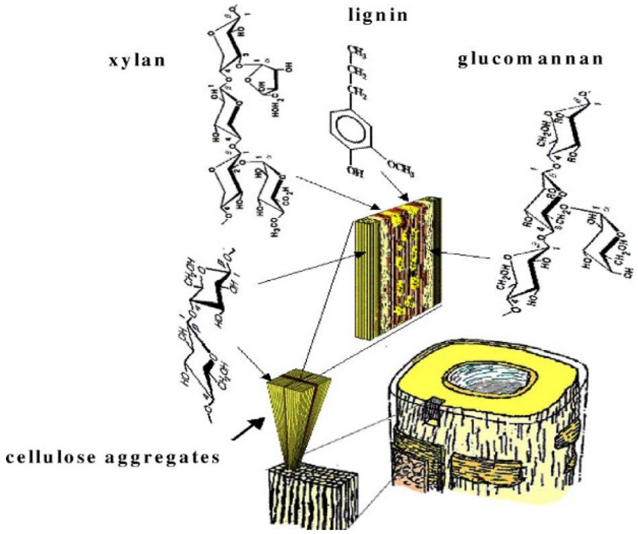
BIOCHEMICAL ARCHITECTURE OF PLANT CELL WALLS



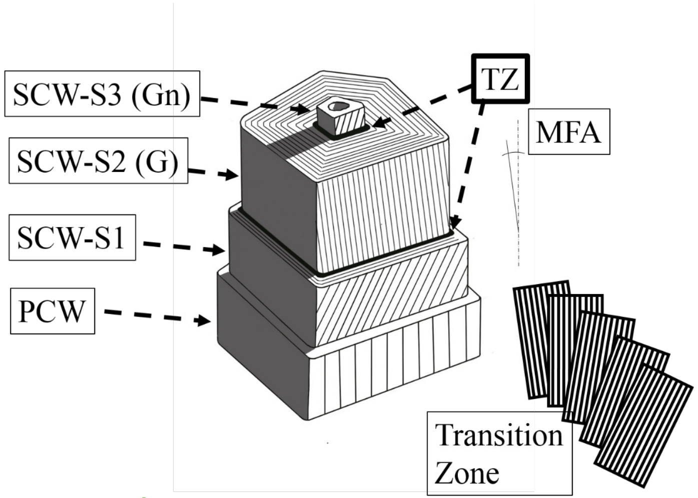


Rihouey et al., 2017

FLAX VS WOOD



Wood cell wall layer ultrastructure
[Salmén, *C.R. Biologies*, 2004]



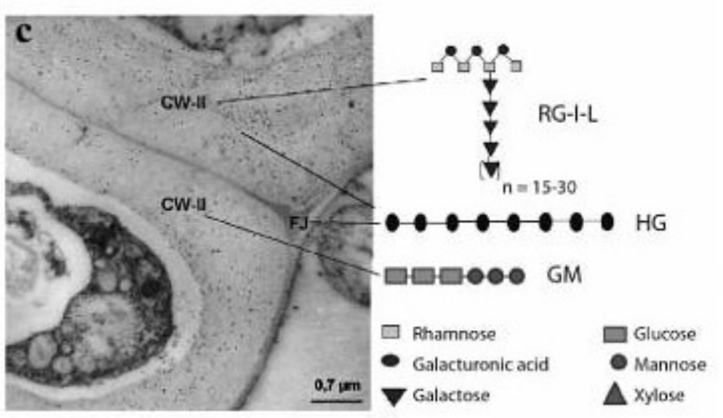
Wood S_2

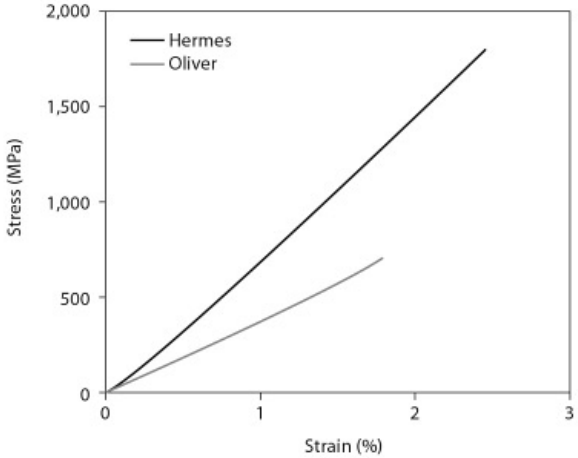
50% cellulose
25% hemicelluloses - pectines
25% lignin
+ extractives, ashes
MFA = (0) 8-40°

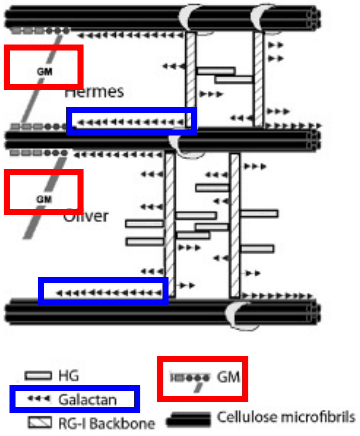
Flax G

80% cellulose
20% hemicelluloses - pectines
≈0% lignin
+ protein
MFA = (0) 5-10°

IMPACT ON STIFFNESS

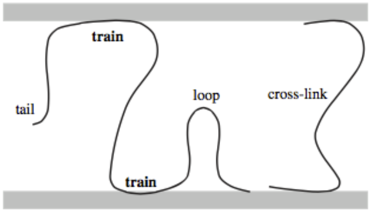






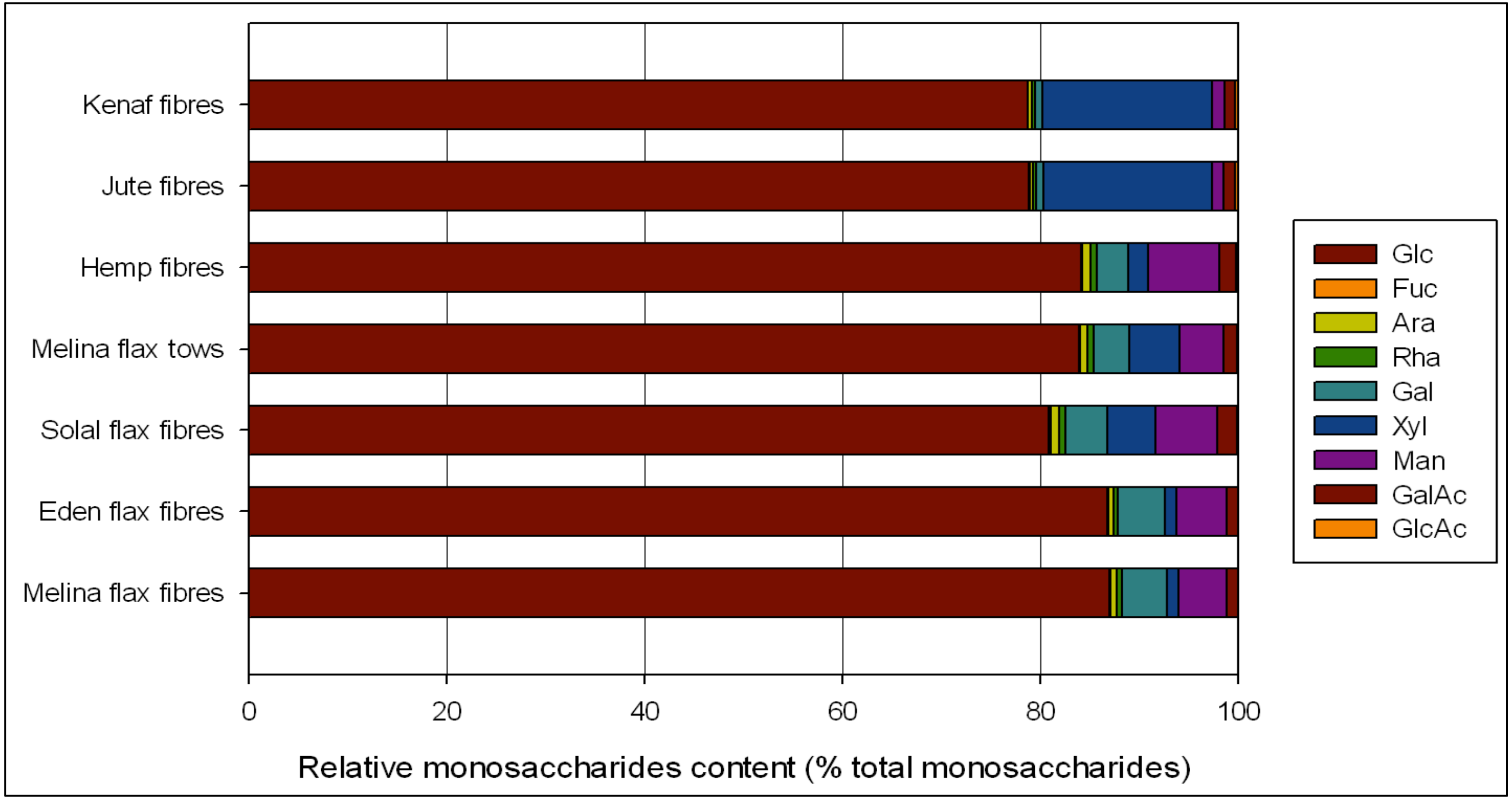
More structuring pectins in Hermes:
higher stiffness

Alix et al., Pectins et Pectinases, 2008



Zykwinska, J Exp Bot, 2007

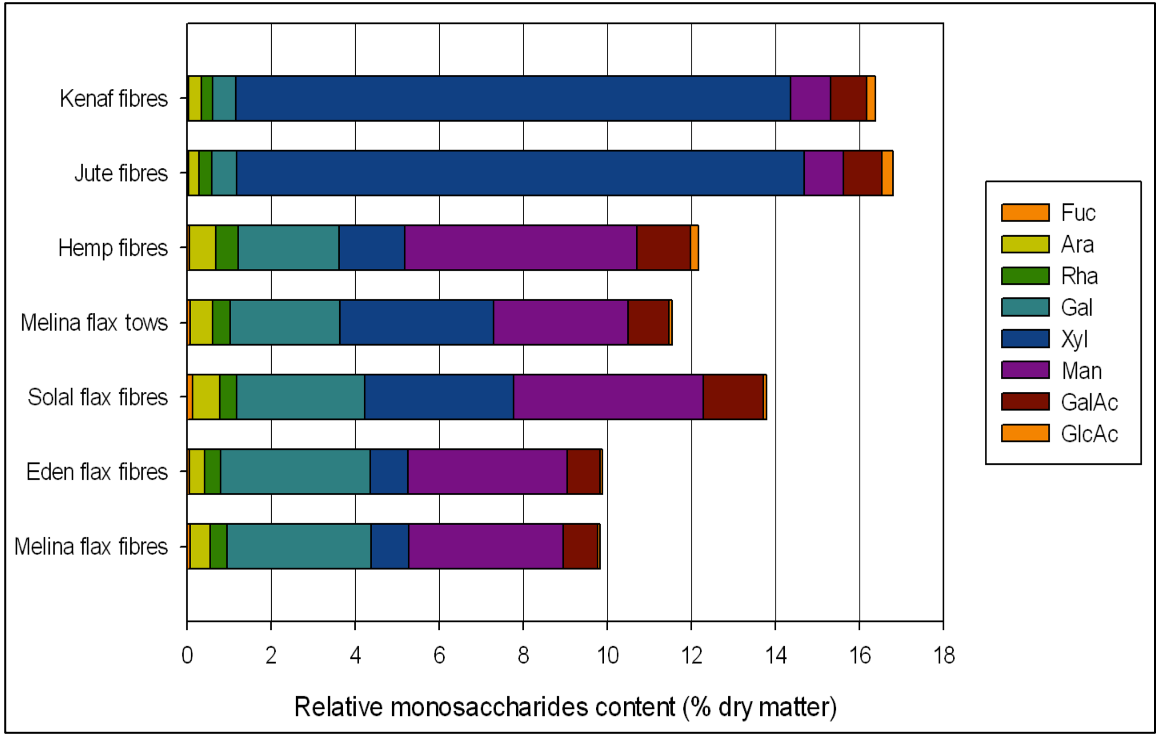
DIFFERENCES BETWEEN THE ORIGIN



More cellulose for scutched textile flax, Lower content for kenaf and jute

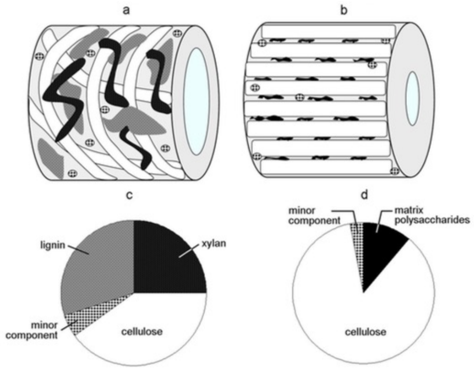
Bourmaud et al, Comp Part A, 2019

DIFFERENCES BETWEEN THE ORIGIN



3 main families:

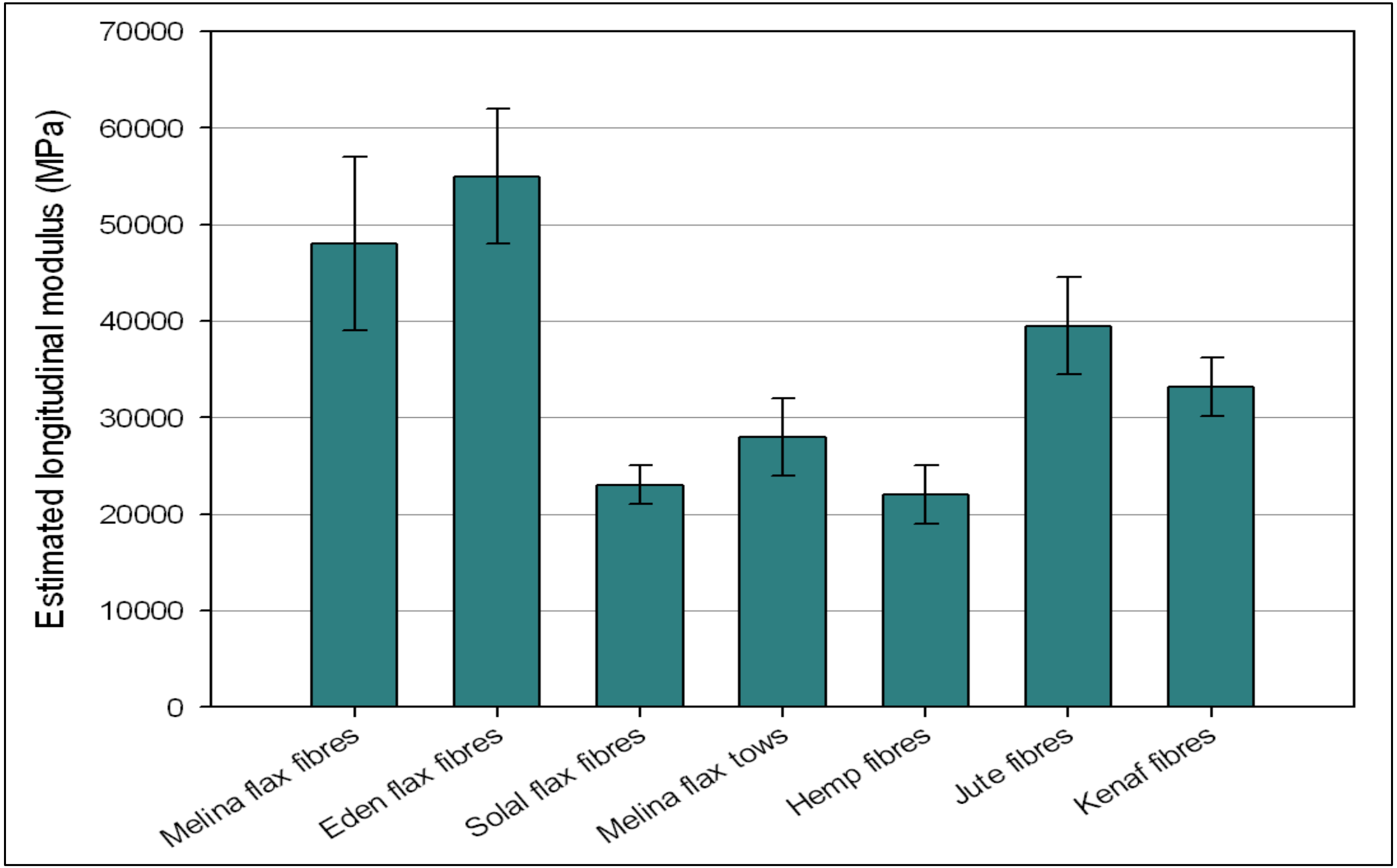
- Kenaf & jute
- Scutched textile flax
- Hemp, flax tows and oleaginous flax



Bourmaud et al, Comp Part A, 2019

Mikshina et al., INTECH, 2013

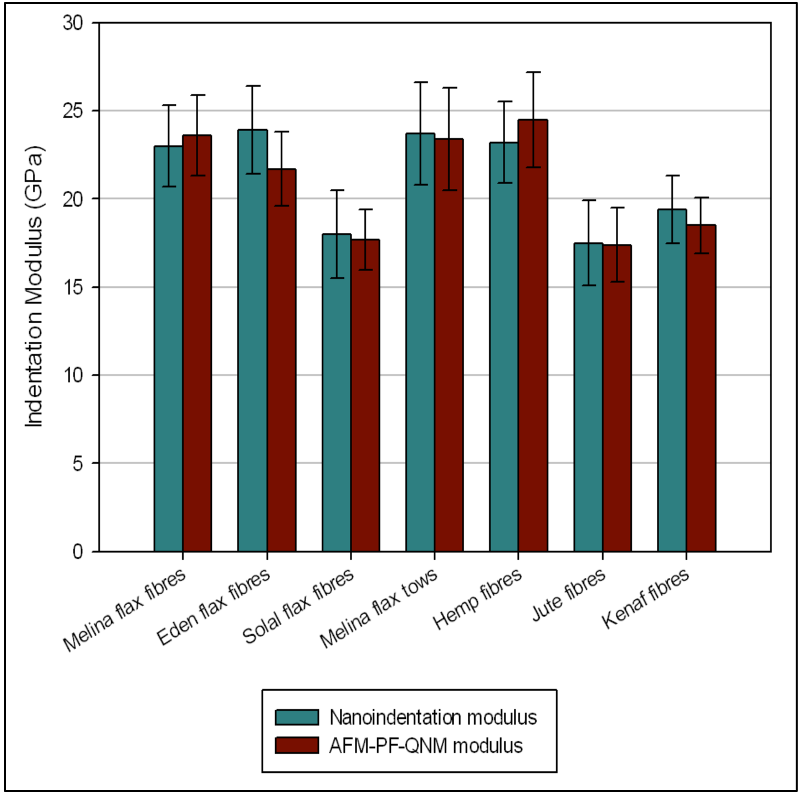
DIFFERENCES BETWEEN THE ORIGIN

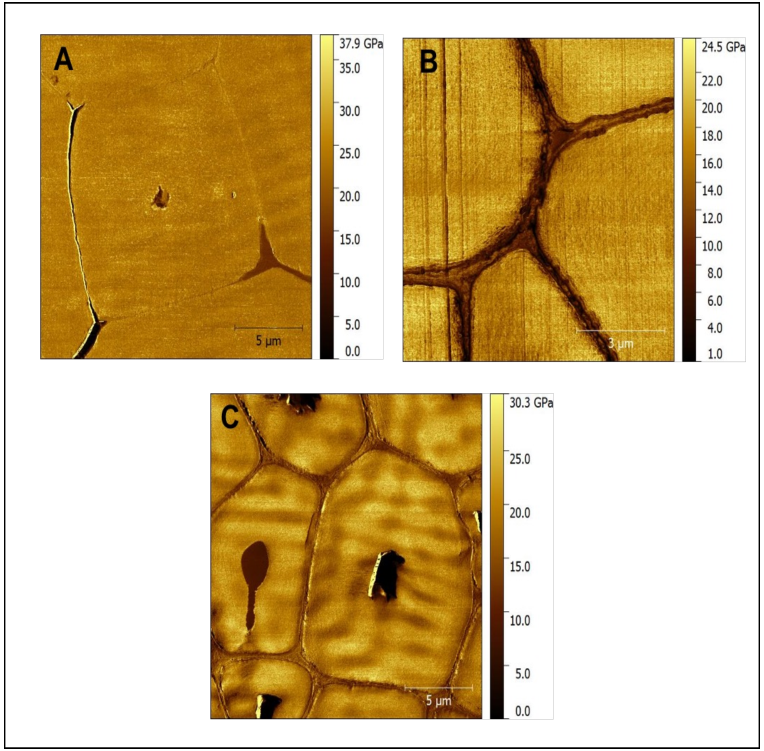


Use of ROM to estimate the fibre stiffness from epoxy-fibre UD composite

Bourmaud et al, Comp Part A, 2019

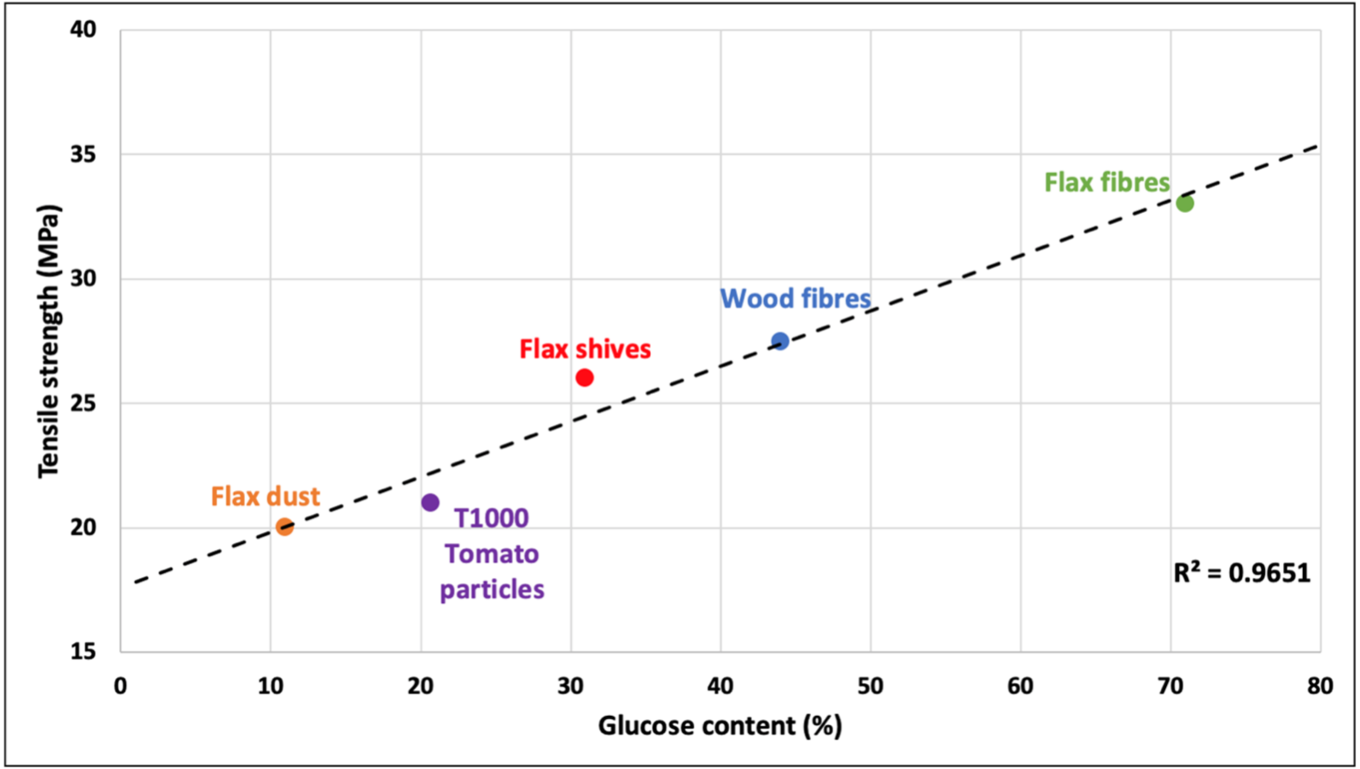
DIFFERENCES BETWEEN THE ORIGIN





Bourmaud et al, Comp Part A, 2019

DIFFERENCES BETWEEN THE ORIGIN



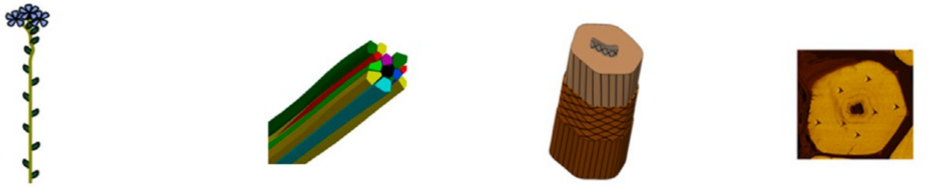
Injected PP-plant fibre composites – Same fibre volume fraction and same PP

Bourmaud et al, Polymers, 2023

MULTI SCALES MECHANICAL INVESTIGATIONS

FOUR MAIN SCALES

Data for Composite Engineer: the pertinent scale for the pertinent informations



Interest of this multi-scale approach:

- Need of understanding: Function, Relationship between biochemistry/mechanical properties
- Data for Engineer: both textile & composite

i) Textile: in our case: Elaboration of semi-products and preforms for composite reinforcement

ii) Composites: Short fibres for extrusion/injection

Mechanical properties at break

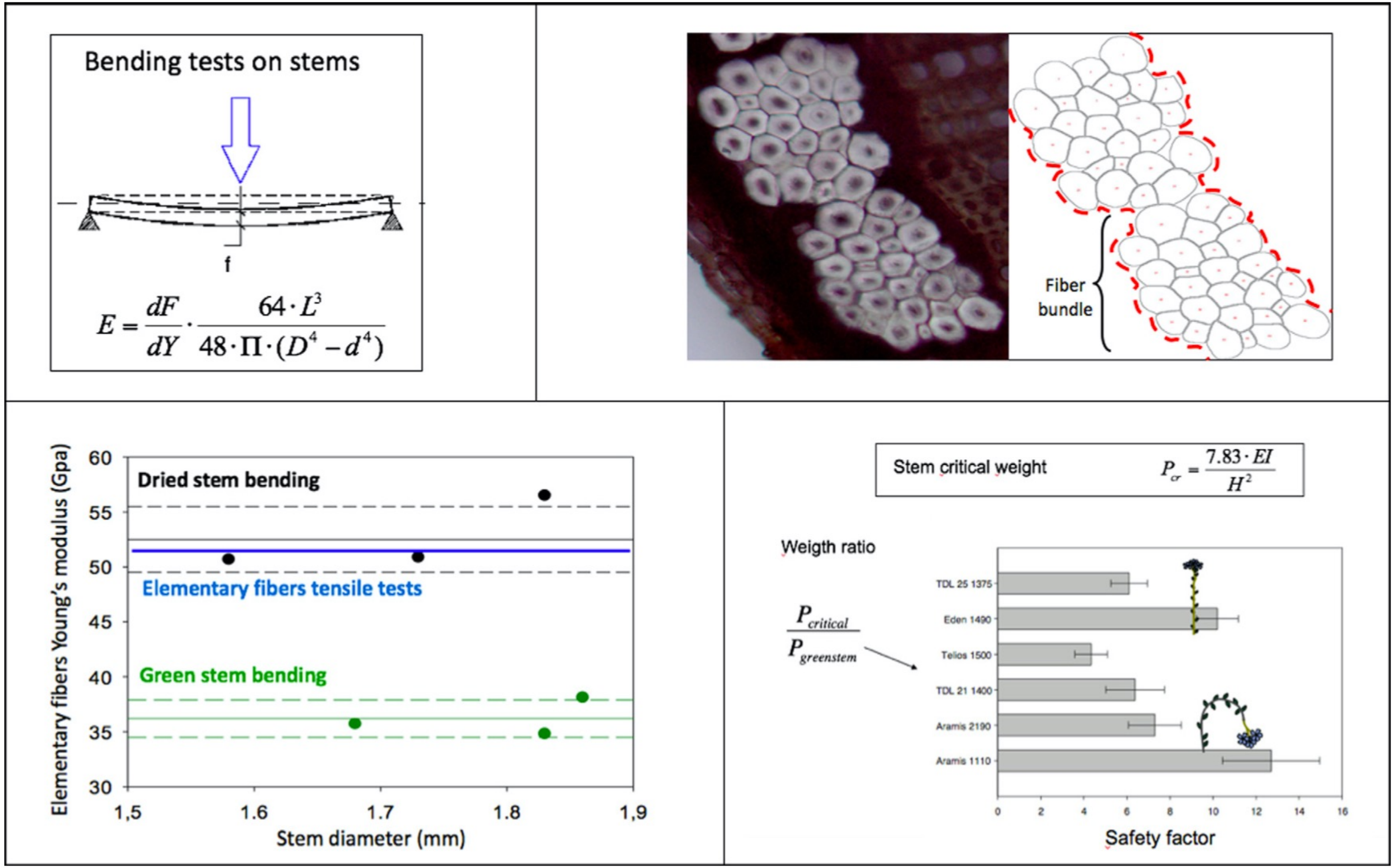
σf_T^+, σf_T^-, σf_L^+, σf_L^-, τ_{LT}, εf_L^+, εf_L^-, εf_T^+, εf_T^-

Mechanical properties of an anisotropic reinforcement

Ef_L, Ef_t, Gf_{lt}, ν_{LT}

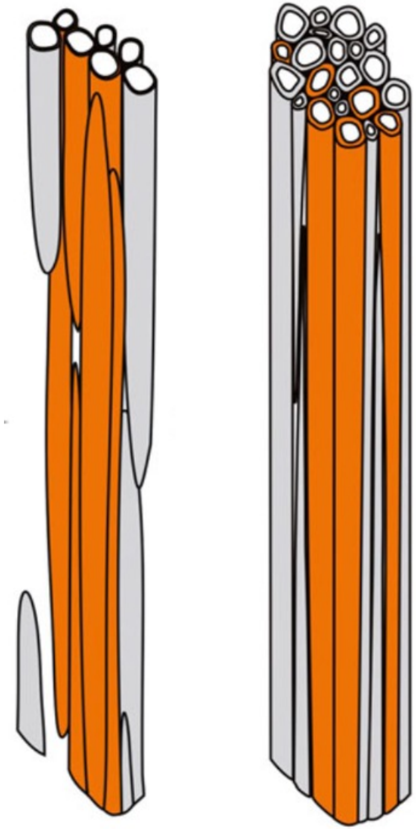
Bourmaud et al., Progress in Materials Science, 2018

STEM SCALE

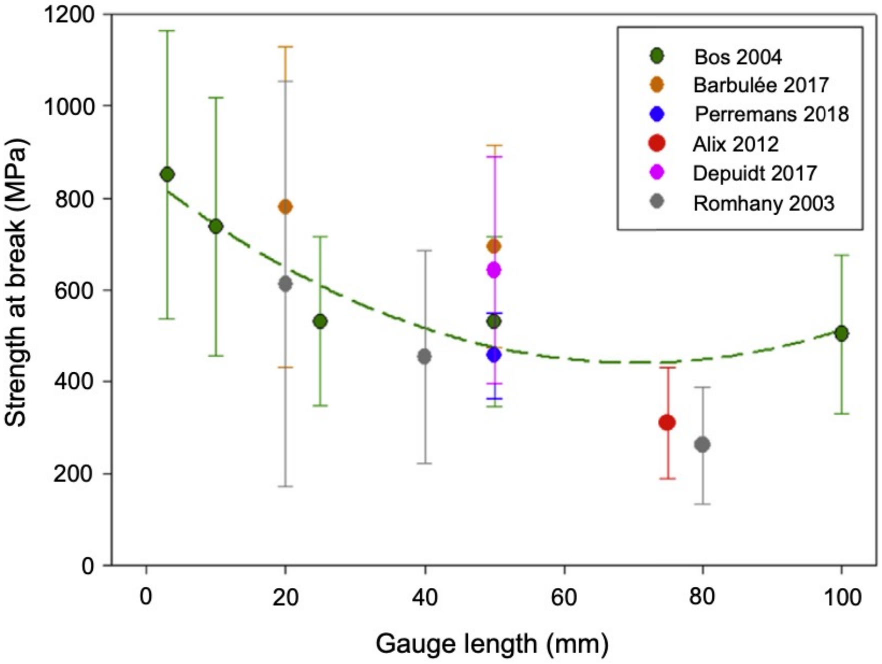


Bourmaud et al., Progress in Materials Science, 2018

BUNDLE SCALE

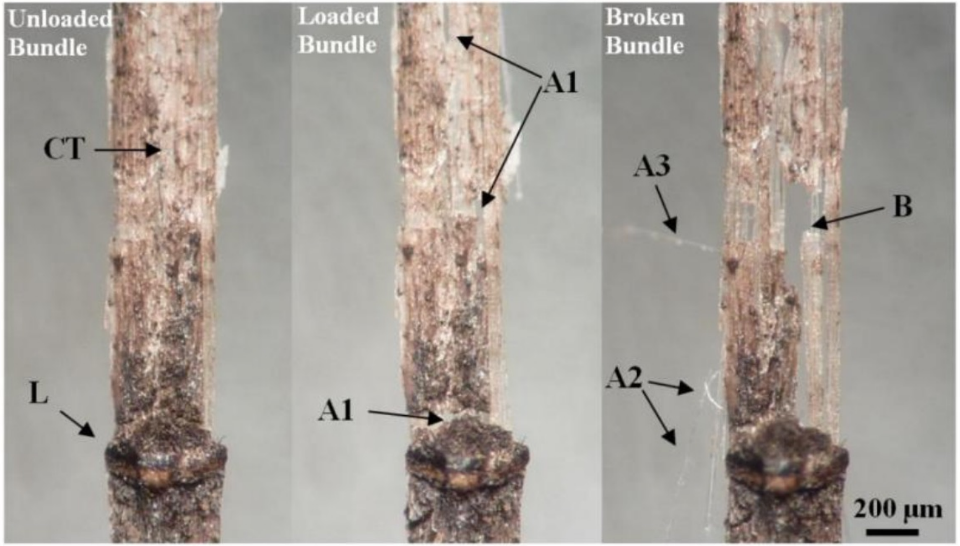


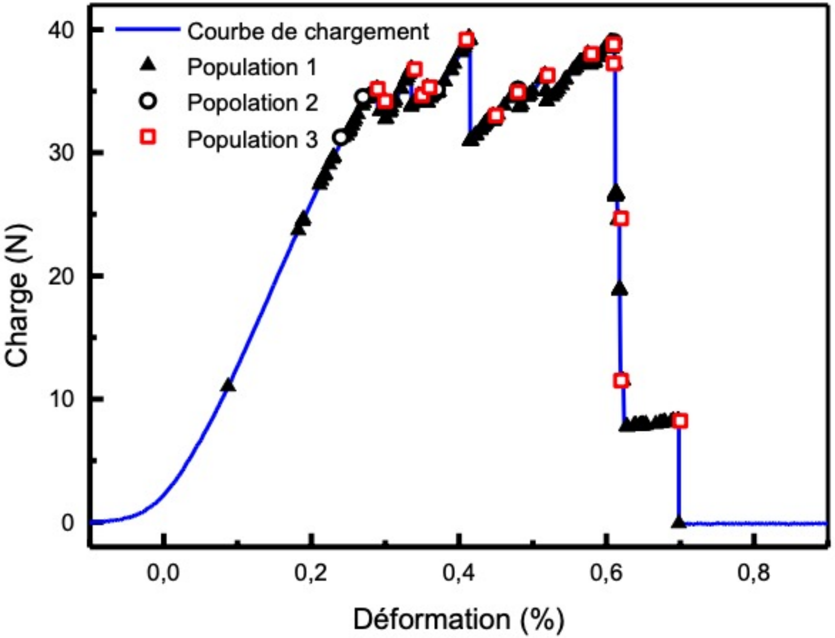
Snegireva 2015



Bourmaud et al., Handbook of Natural Fibres, 2020

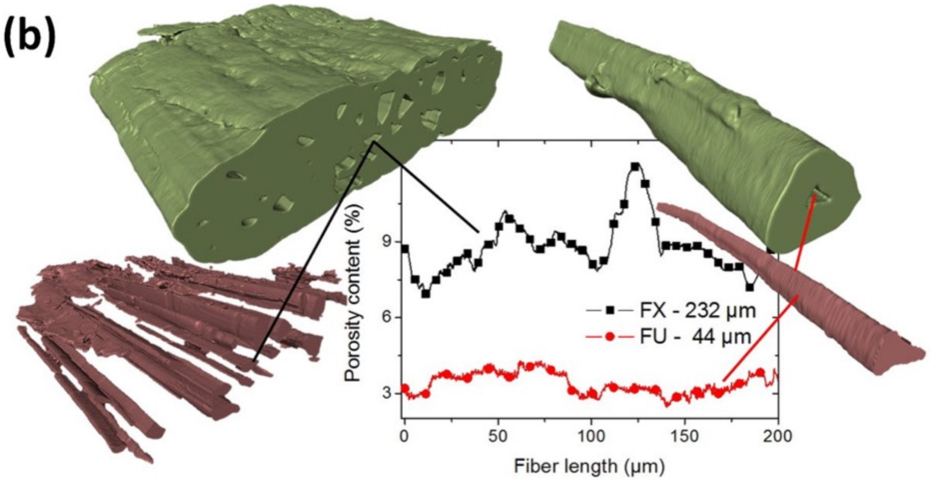
BUNDLE SCALE

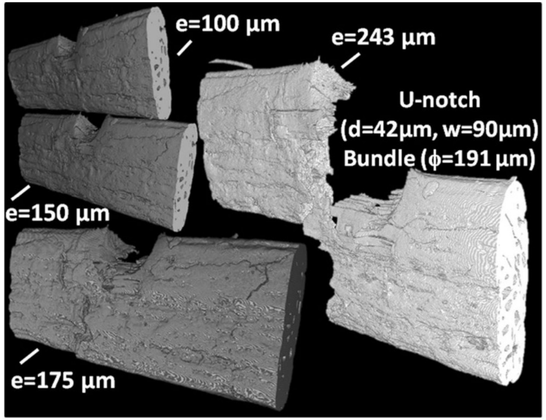


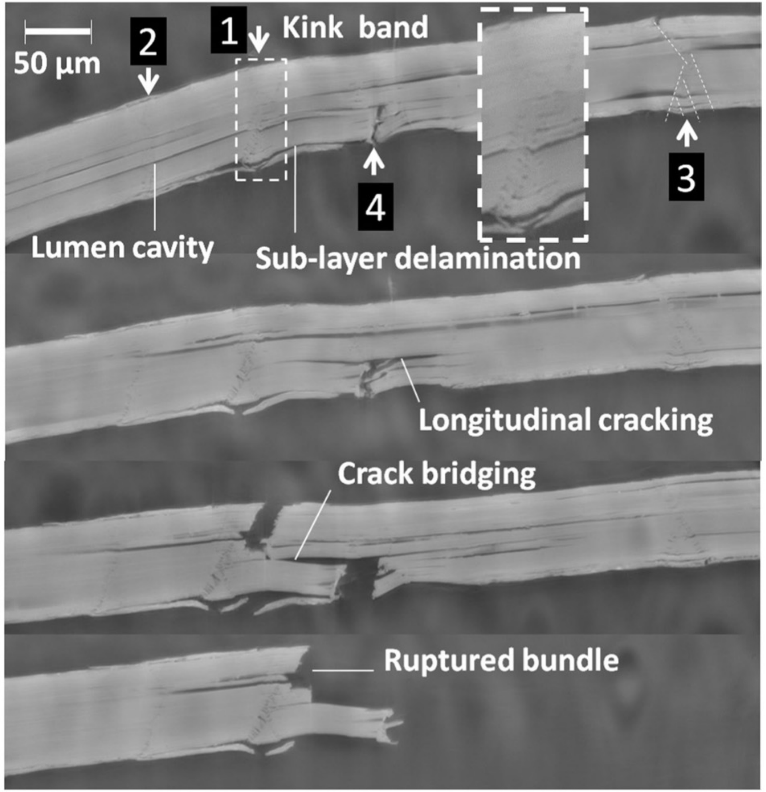


Bourmaud et al., Handbook of Natural Fibres, 2015

BUNDLE SCALE

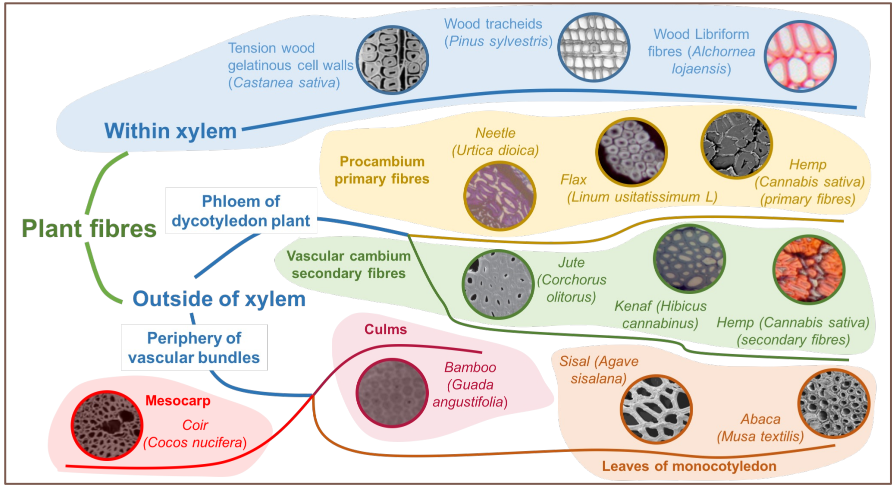


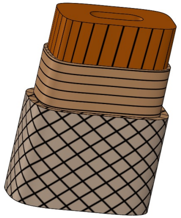




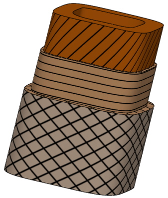
Beaugrand et al., Scientific Reports, 2017

SINGLE FIBRES AND CELL WALLS

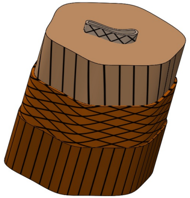




Tension wood



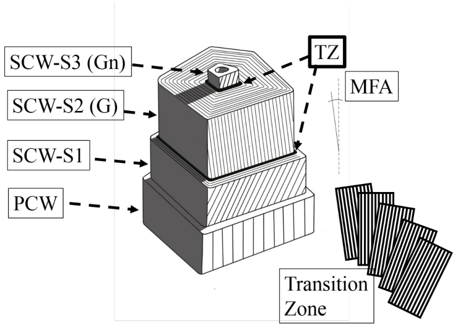
Normal wood

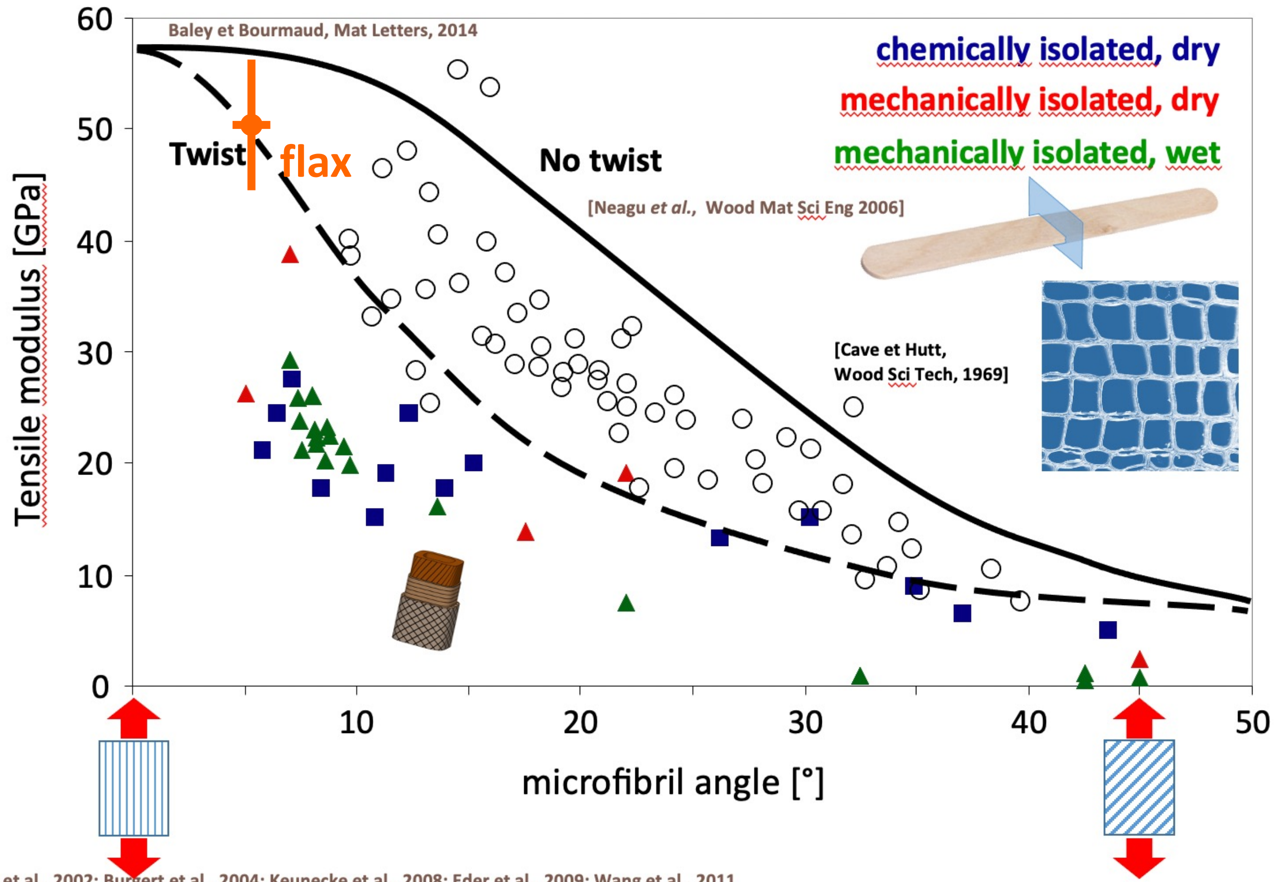


Flax

Bourmaud et al., Progress in Materials Science, 2018

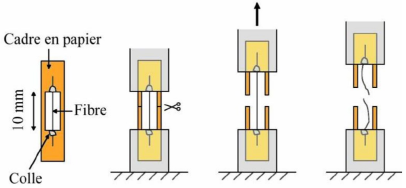
SPECIFIC MECHANICAL BEHAVIOUR OF PLANT FIBRES

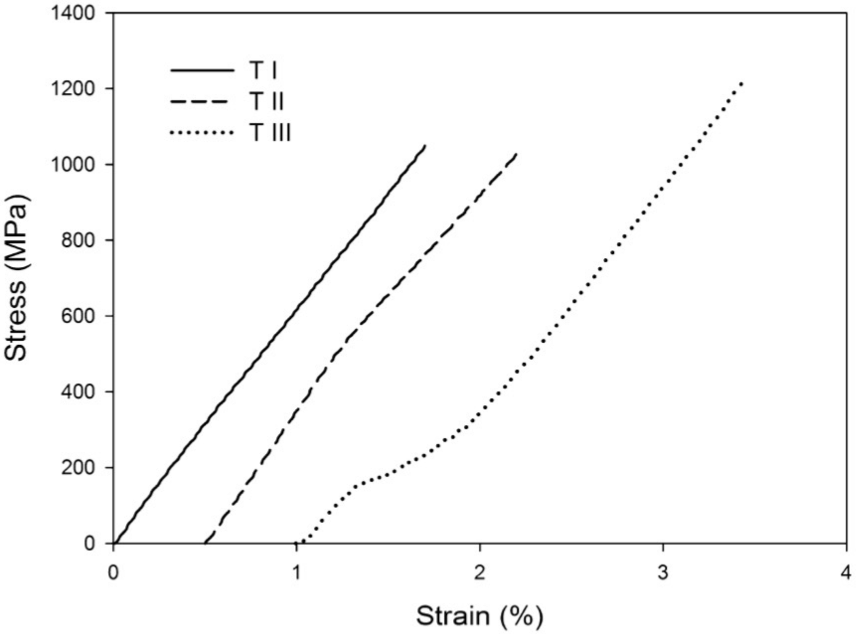


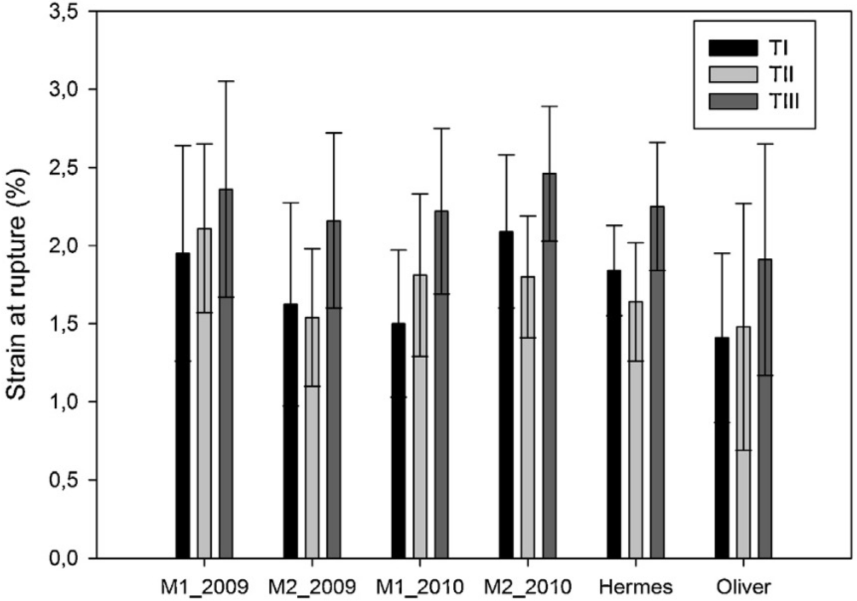


Mott et al., 2002; Burgert et al., 2004; Keunecke et al., 2008; Eder et al., 2009; Wang et al., 2011

SPECIFIC MECHANICAL BEHAVIOUR OF PLANT FIBRES

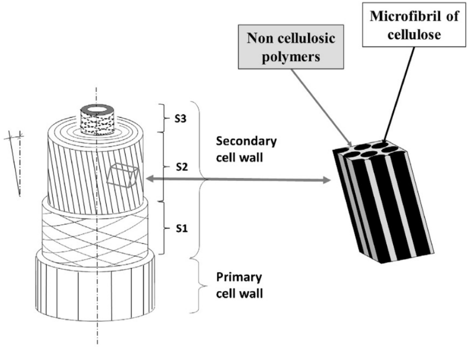


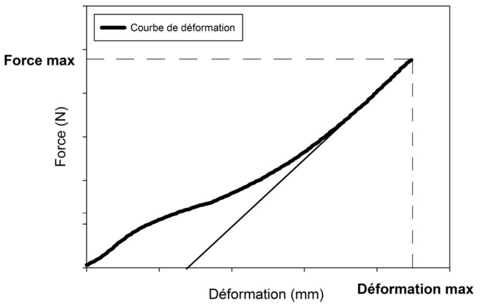


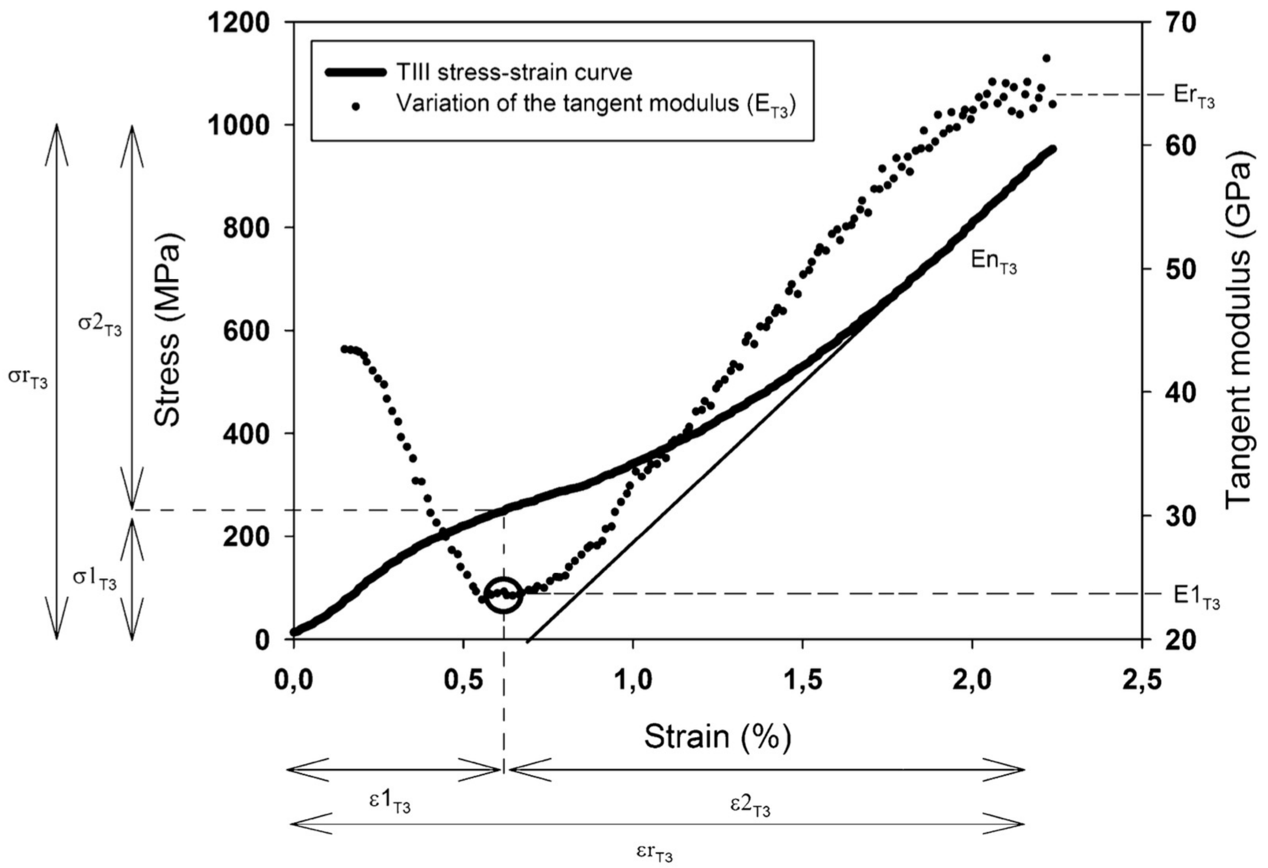


Lefeuvre et al, Ind Crops et Prod, 2014

SPECIFIC MECHANICAL BEHAVIOUR OF PLANT FIBRES

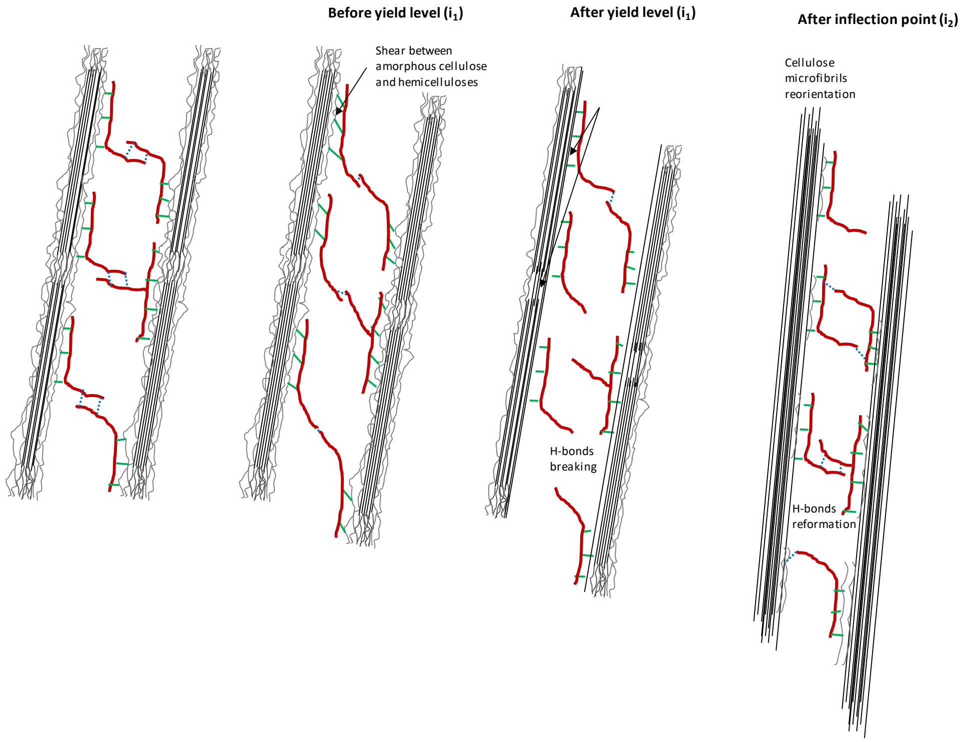






Lefeuvre et al, Ind Crops et Prod, 2014

SPECIFIC MECHANICAL BEHAVIOUR OF PLANT FIBRES



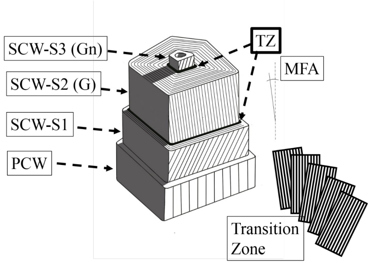
Possible scenario describing the various mechanisms contributing to the multiple nonlinearities of the stress–strain curve of hemp fibre.

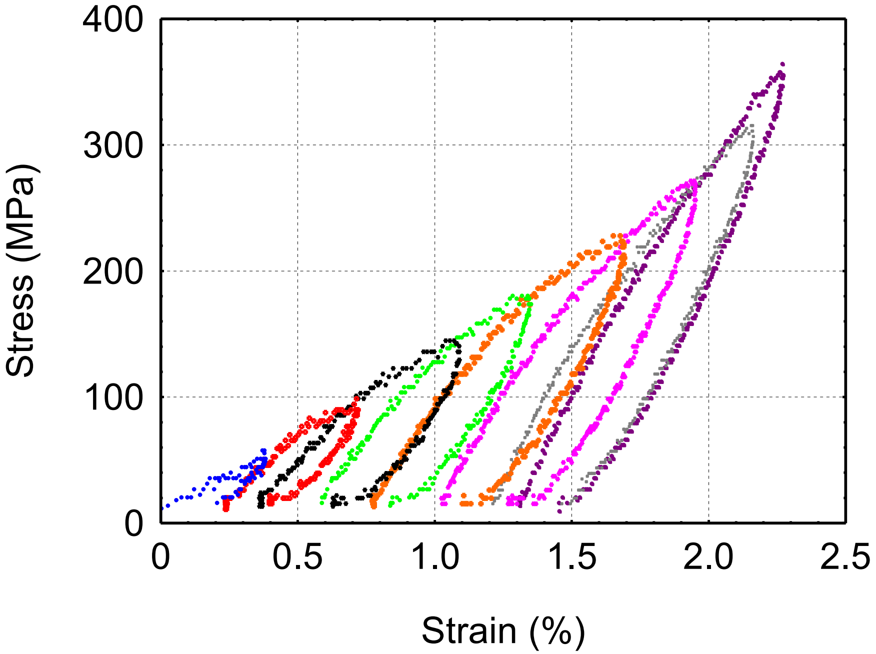
Segment (point) of the stress–strain curve	Observations	Possible mechanisms
I	Quasi-linear behaviour with slightly irreversible strain	• Elastic deformation of the cellulose microfibrils and amorphous polymers • Slight rotation of the microfibrils towards a more parallel orientation
i_1	Yield level	• Matrix flow threshold: bonds break in the amorphous matrix
II	Apparent decrease in fibre' stiffness	• Viscous flow of the amorphous components under shear strain and lock-in at a new position
	Quasi-linear behaviour, with significant irreversible deformations and fibre stiffening when the load is released or the fibre is re-loaded	• Stress-induced crystallisation of the para-crystalline cellulose • Spiral spring-like extension of the cellulose microfibrils in the amorphous matrix
i_2	Inflection point	• Maximum flow point of the matrix • Crystallisation saturation point
III	Quasi-linear or parabolic	• Deployment of cellulose microfibrils in dislocation areas • Decrease of the mean MFA • Interfacial rupture between crystalline cellulose and the amorphous matrix

Placet et al, Comp Part A, 2013

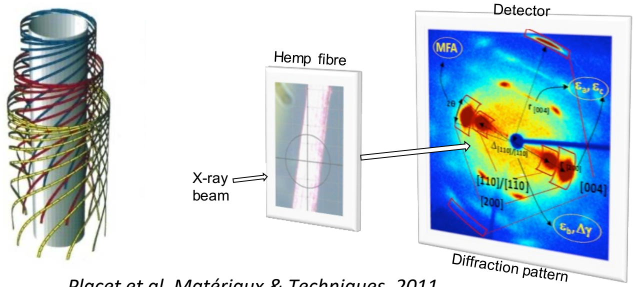
SPECIFIC MECHANICAL BEHAVIOUR OF PLANT FIBRES

- **Stiffening effect**



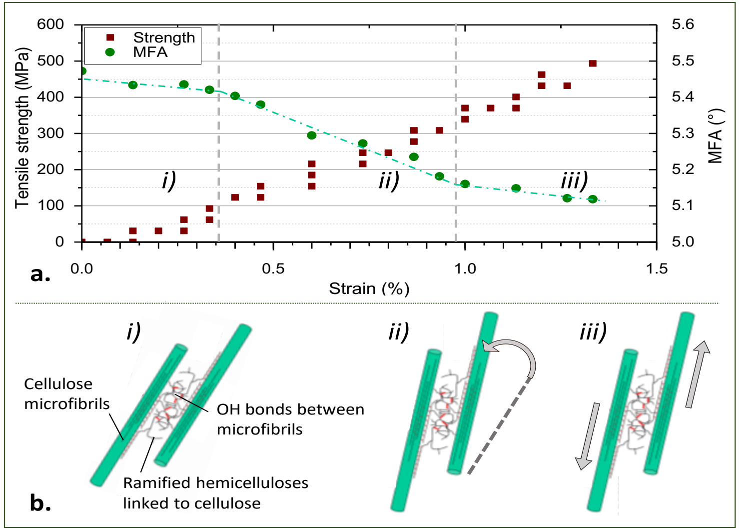


Placet et al. Compos Part A, 2014



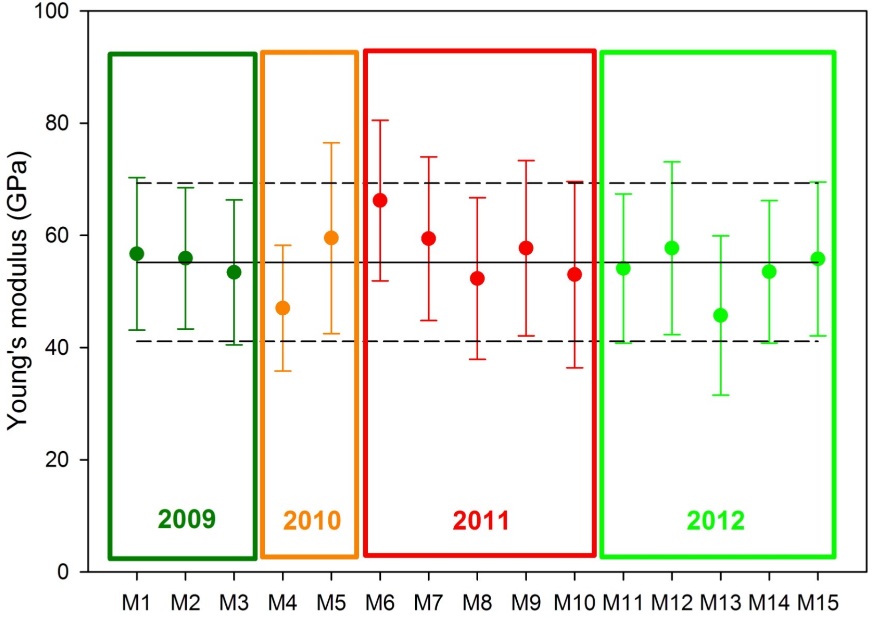
Placet et al. Matériaux & Techniques, 2011

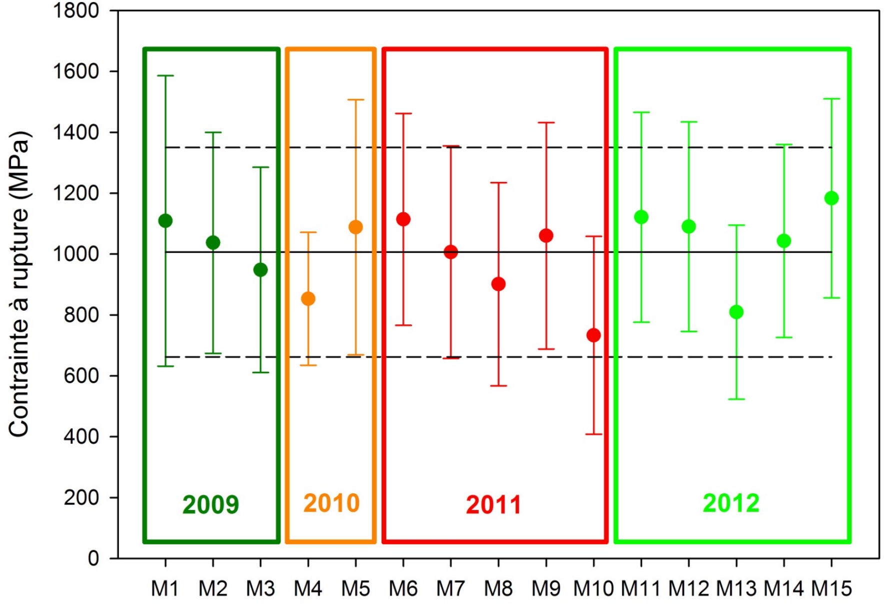
Bourmaud et al. Ind Crop Prod, 2013



Nuez et al.,Comp Part C, 2021

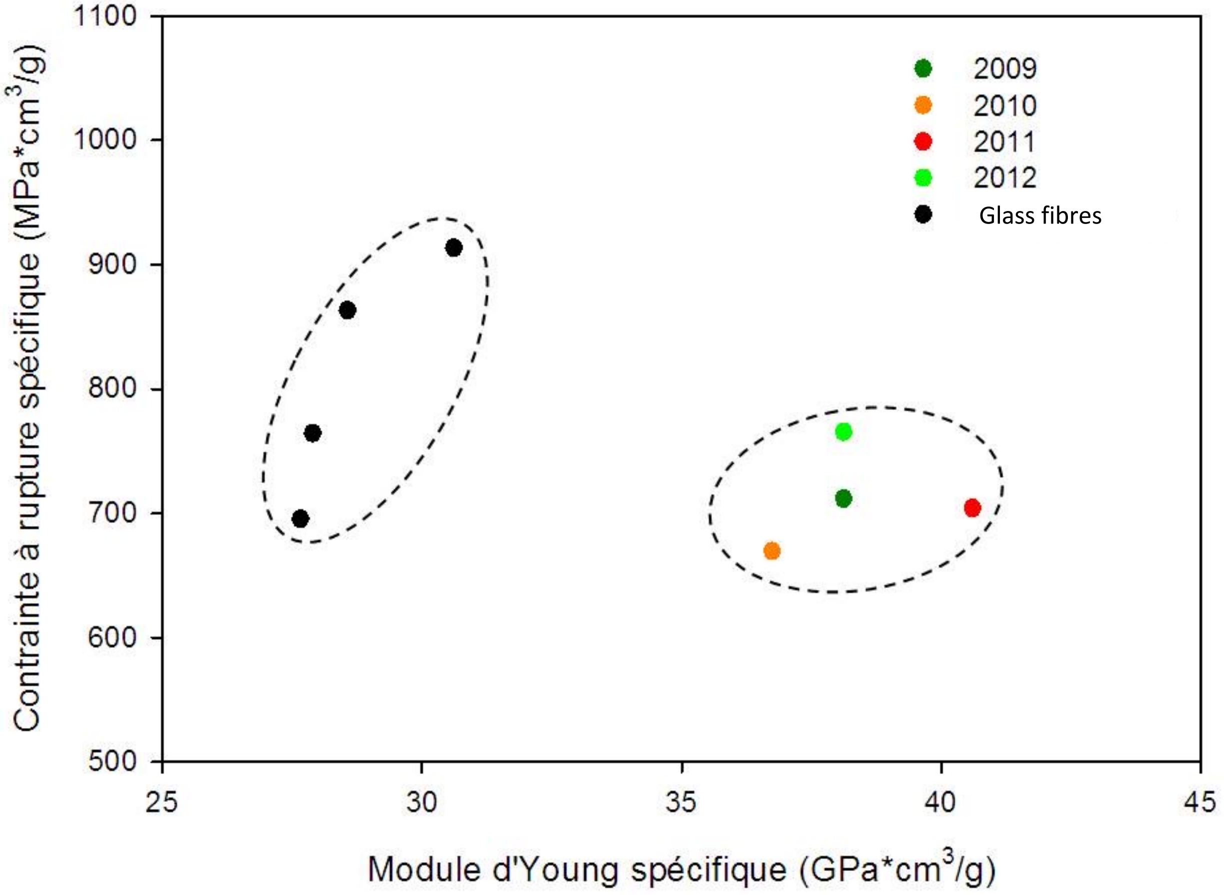
SPECIFIC MECHANICAL BEHAVIOUR OF PLANT FIBRES





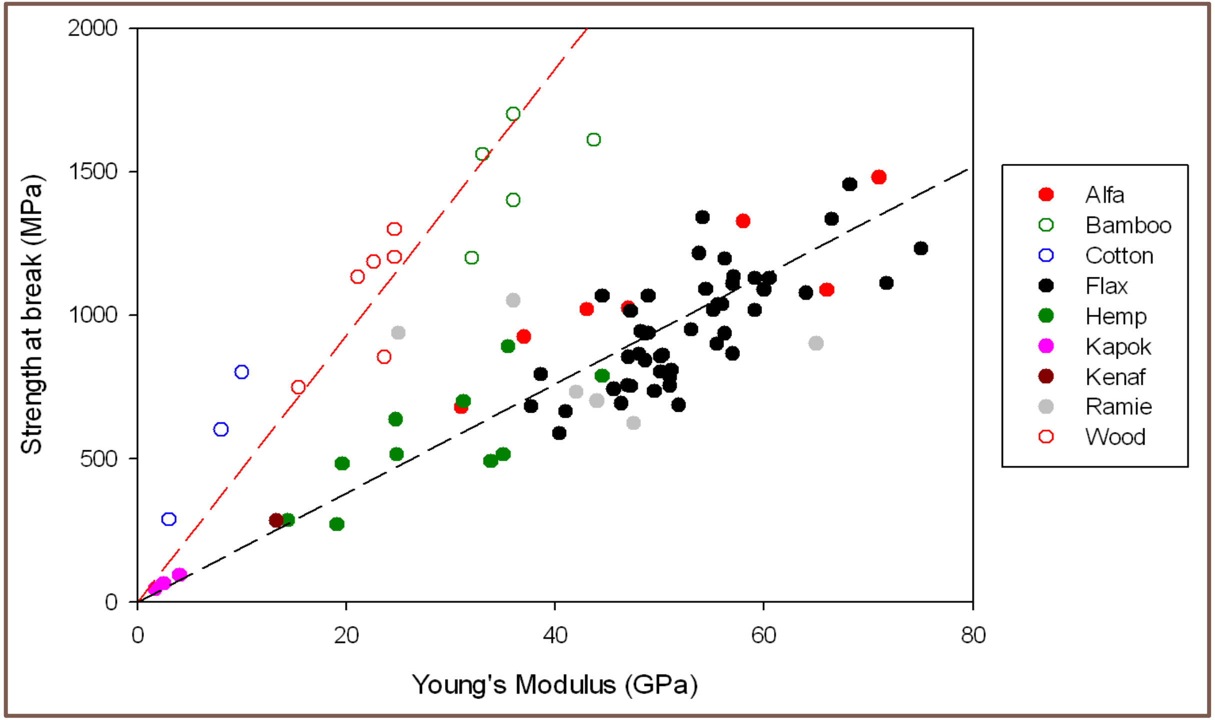
Lefeuvre et al, Ind Crops et Prod, 2014

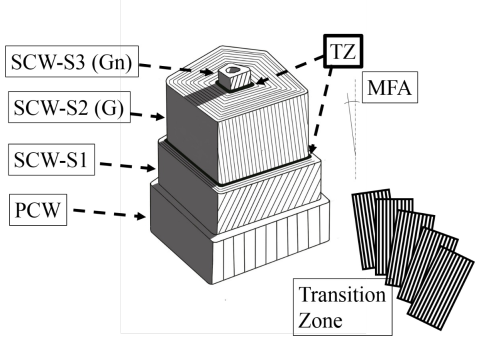
SPECIFIC MECHANICAL BEHAVIOUR OF PLANT FIBRES



Lefeuvre et al, Ind Crops et Prod, 2014

SINGLE FIBRES AND CELL WALLS

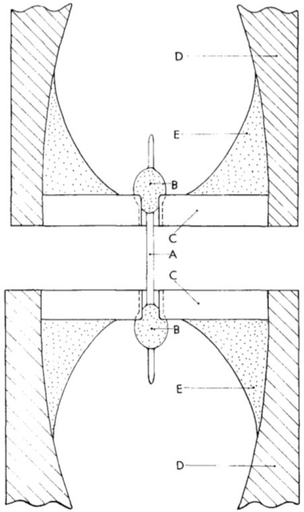


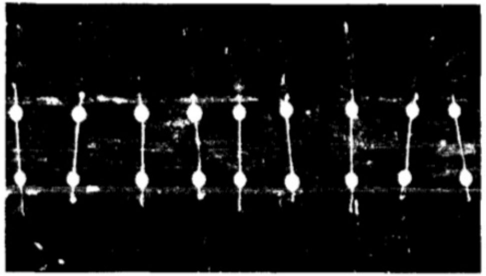


But strong limits, especially in link with the length of fibres for a range of species

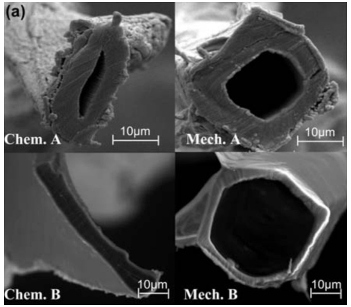
Bourmaud et al., Progress in Materials Science, 2018

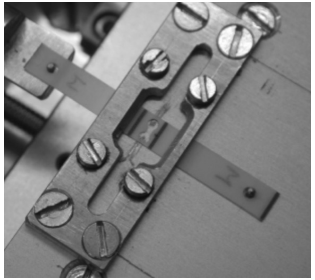
TENSILE TESTS ON SHORT FIBRES

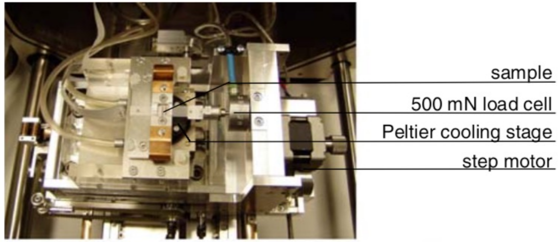


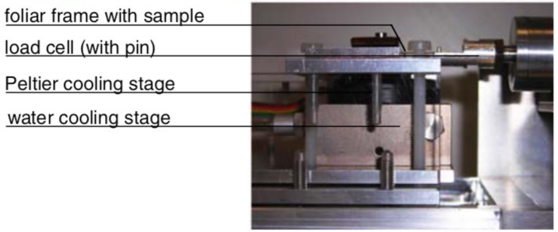


Kersevage, Wood Fiber Sci, 1973



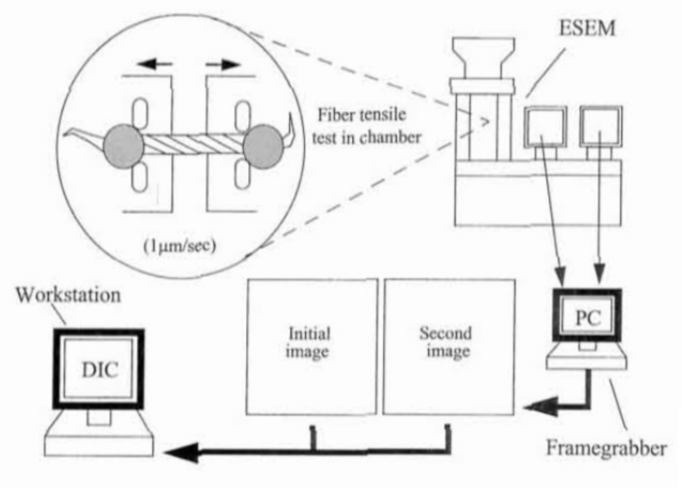


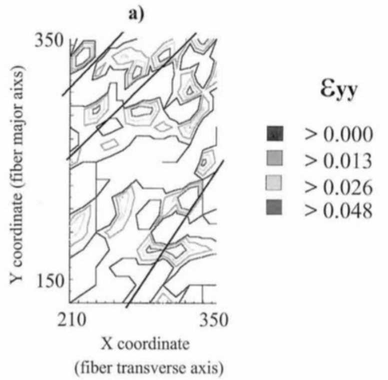




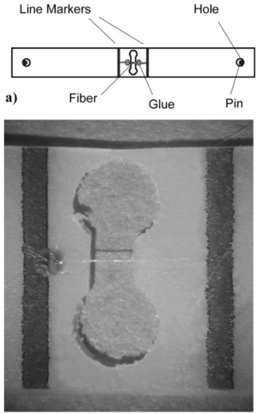
Eder, Wood Sci Technol, 2008

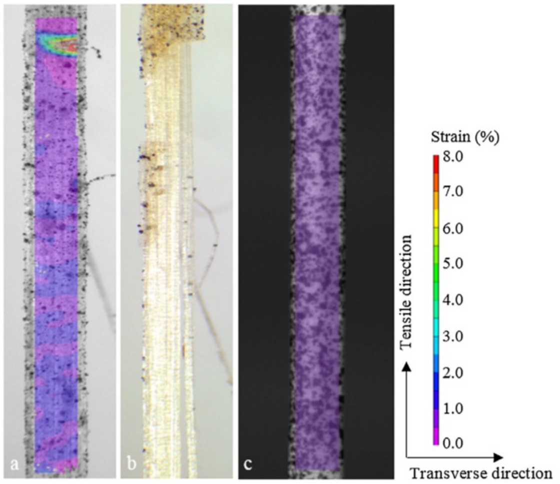
STRAIN MONITORING



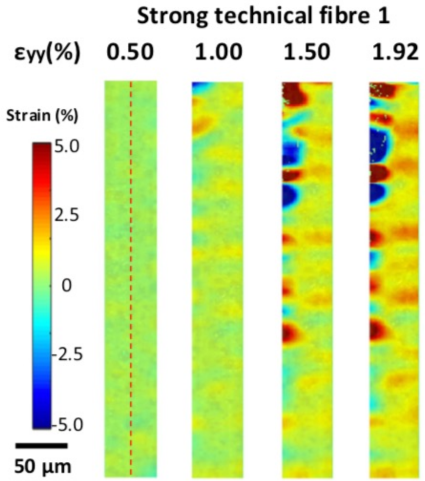


Mott, Wood Sci Technol, 1996





Burgert, Holzforschung, 2003

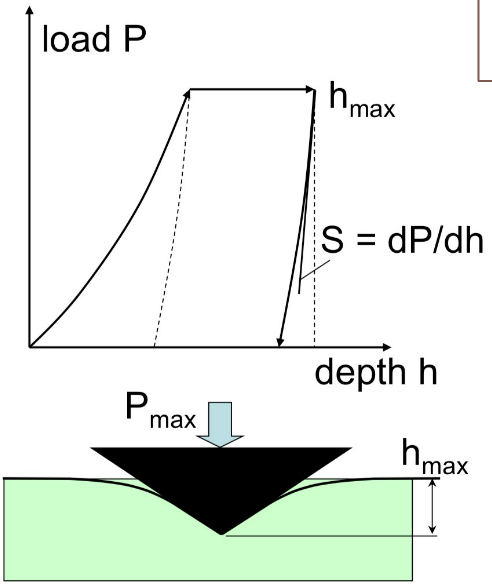


Fuentes, Comp Part A, 2017

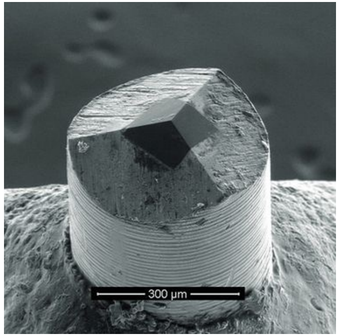
LIMITS OF TENSILE TESTING ON SINGLE FIBRES

- **Difficult to implement for a range of short plant fibres: wood, sisal, jute….**
- **Apparent mechanical properties: interesting for composite reinforcement but not at a lower scale.**
- **There is a need for mechanical investigations at the cell wall scale.**
- **Link between ultrastructure and mechanical properties: impact of MFA, multi layers arrangement……**

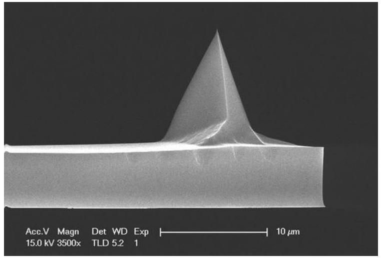
NANOINDENTATION & AFM PEAK FORCE MEASUREMENTS

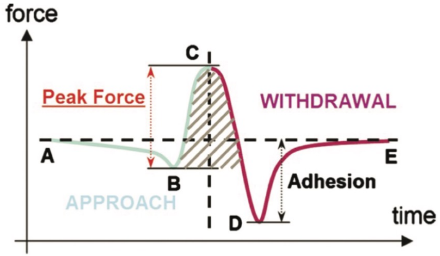


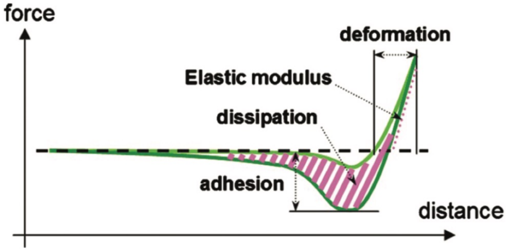
80-120 nm



2-3 nm



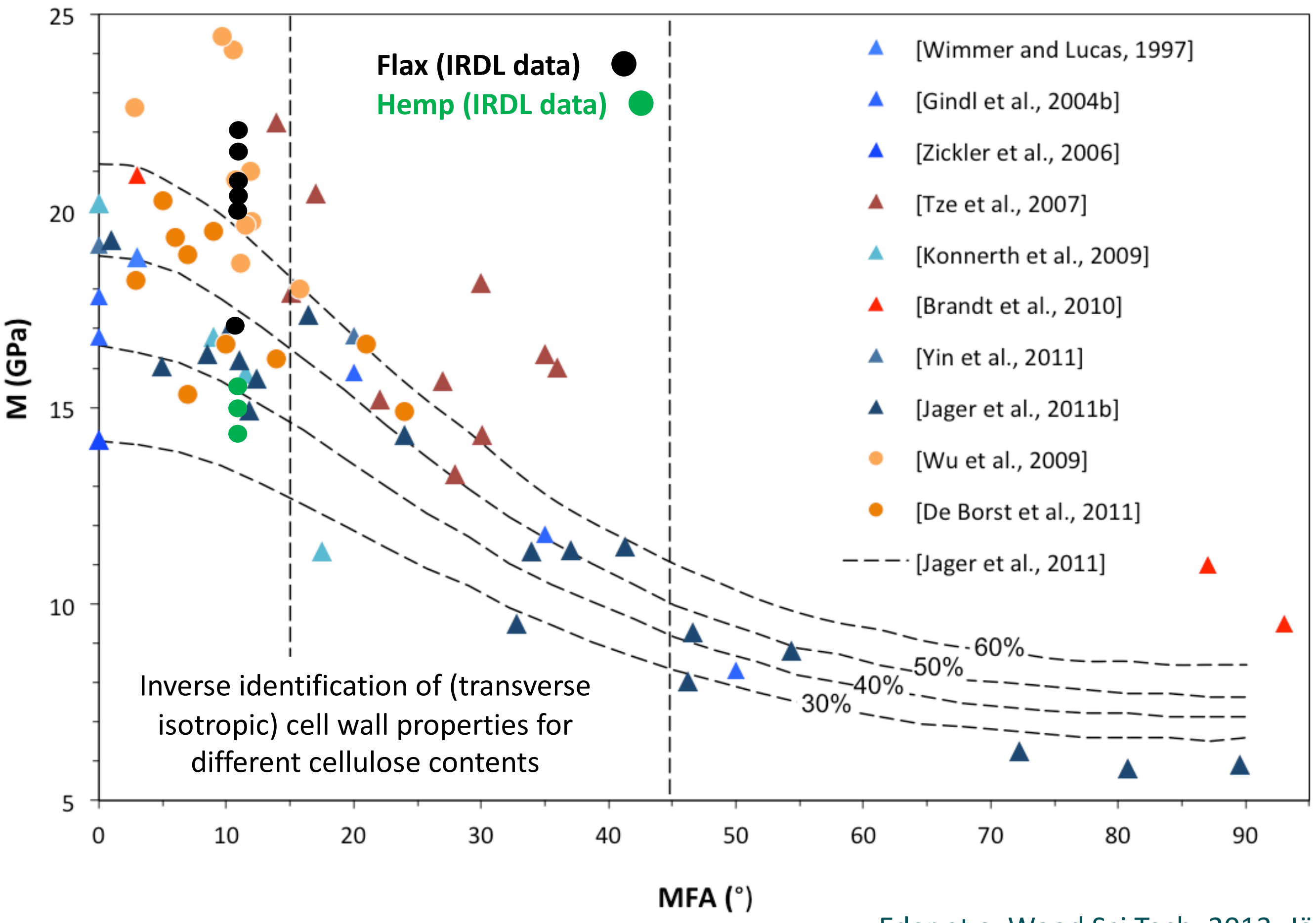




Lee et al. Intech, 2019

Konnerth (BOKU Institute) and BRUKER doc

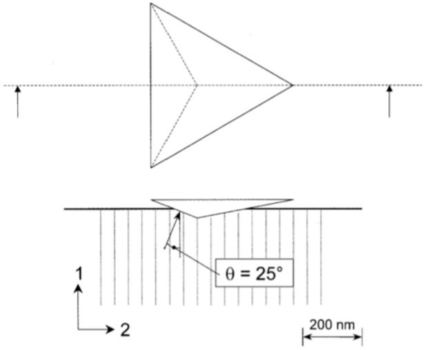
NANOINDENTATION MEASUREMENTS

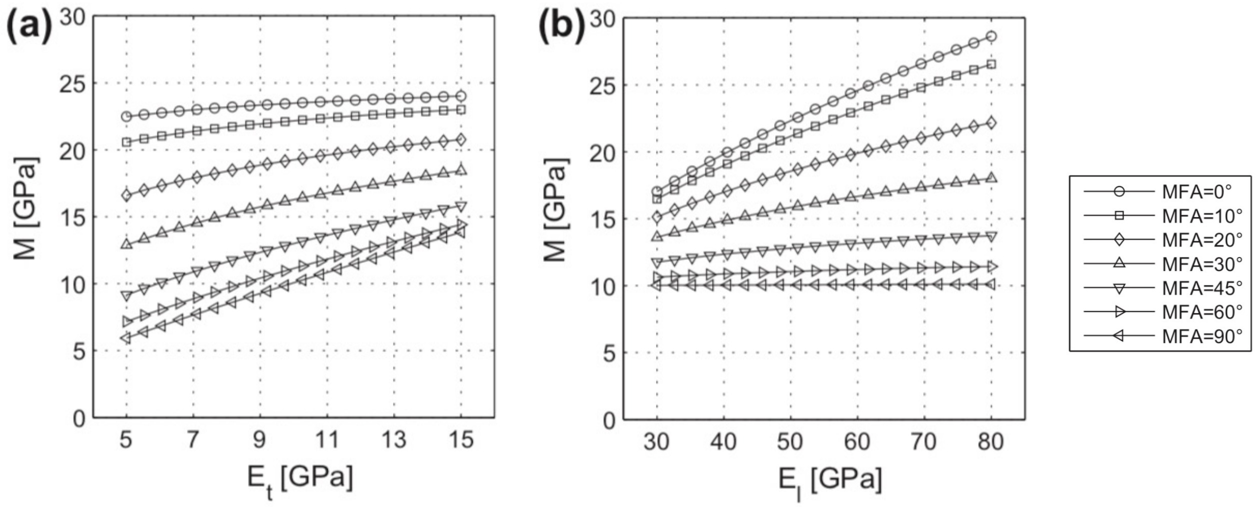


Eder et a. Wood Sci Tech, 2013; Jäger et al., Comp A, 2011

NANOINDENTATION Vs TENSILE TEST

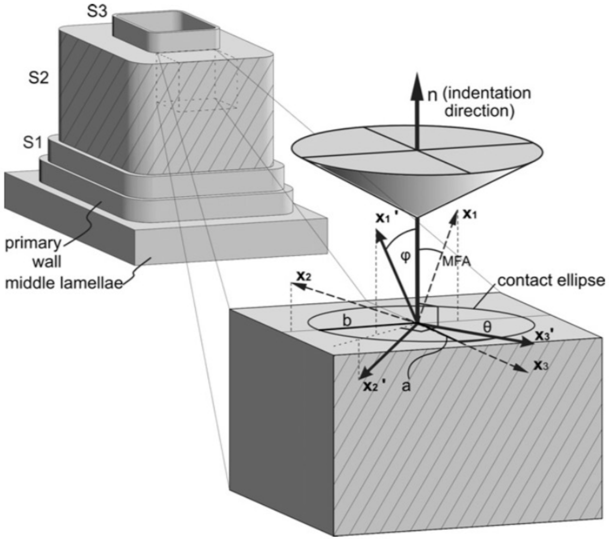
Usual NI models do not consider anisotropy of plant cell walls: Underestimation of M compared to E_{Lf}





Gindl, Comp Part A, 2004

NANOINDENTATION Vs TENSILE TEST



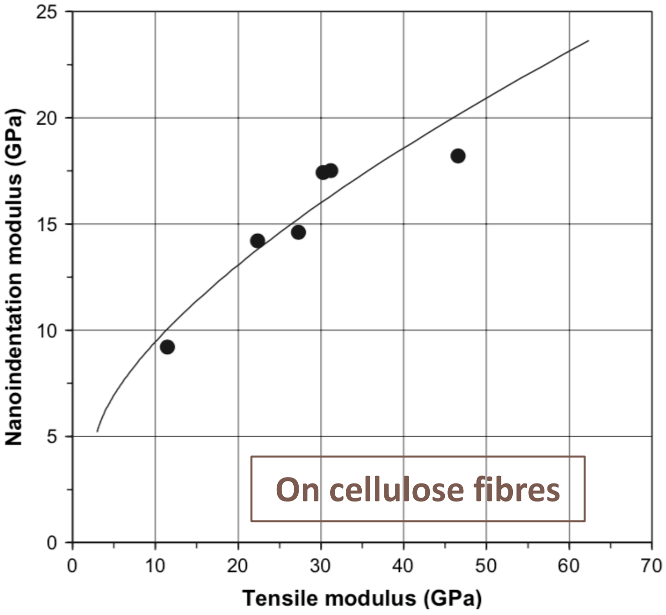
Specific models for anisotropic materials
(Vlassak 2003, Swadener 2001)

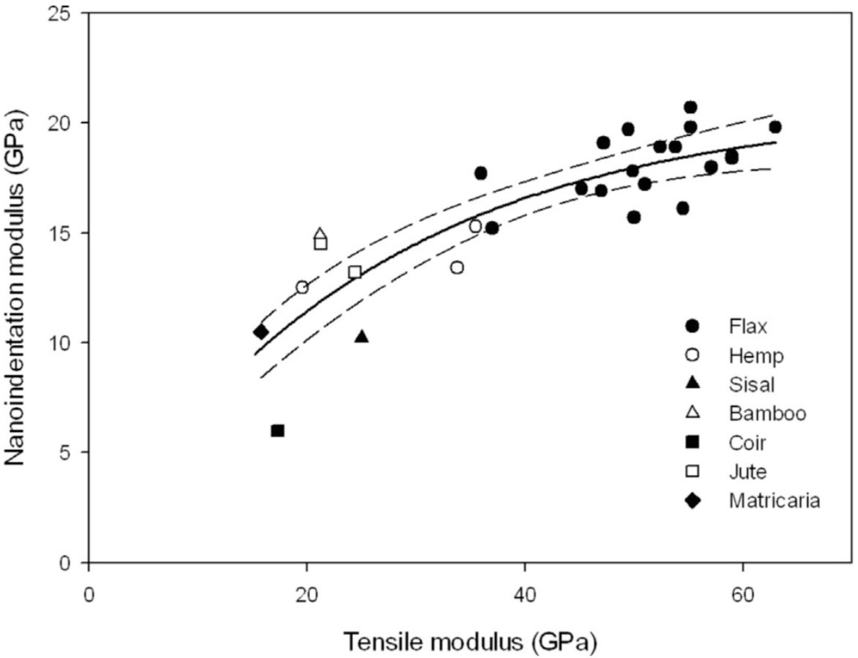
No direct relation between M and E_{Lf}

The indentation modulus is a function of E_{Lf}, E_{Tf}, and, especially, shear modulus of the cell wall material

Jäger, Comp Part A, 2011

NANOINDENTATION Vs TENSILE TEST





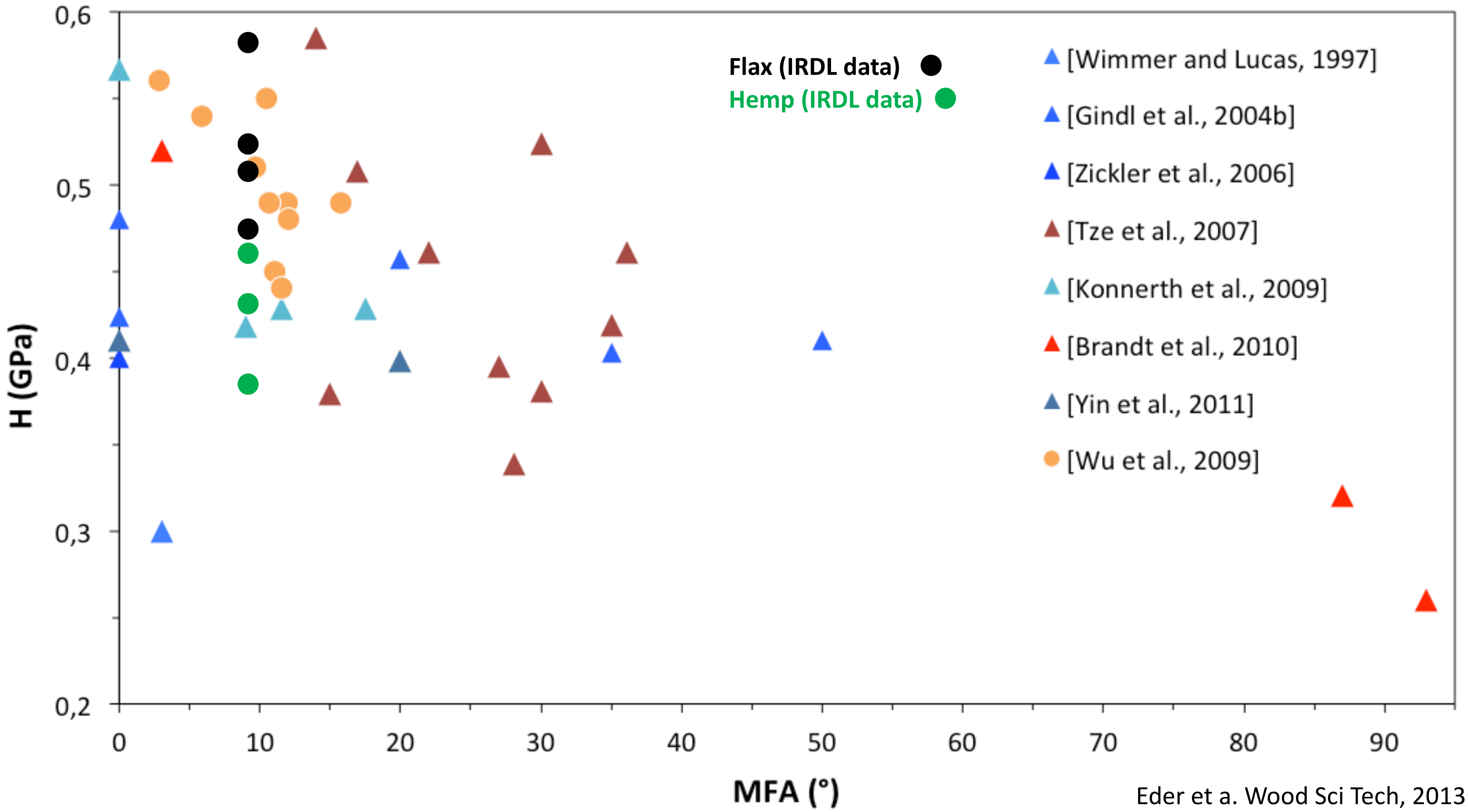
Some correlation attempts in literature between M and El

But very questionable due to the moderate impact of E_{Lf} on M, especially for high MFA and possible discrepancy

Gindl, Polymer 2008

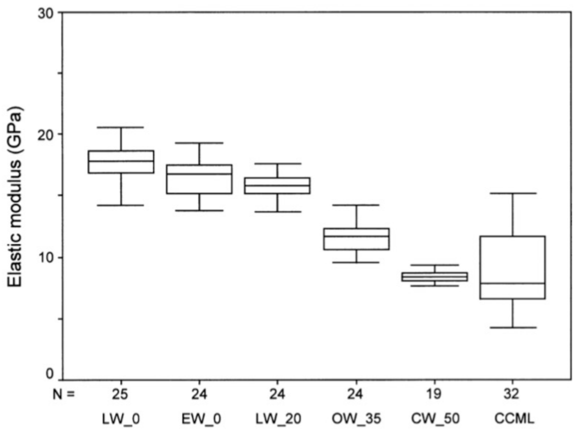
Tanguy, Mat Letters, 2014

HARDNESS MEASUREMENT

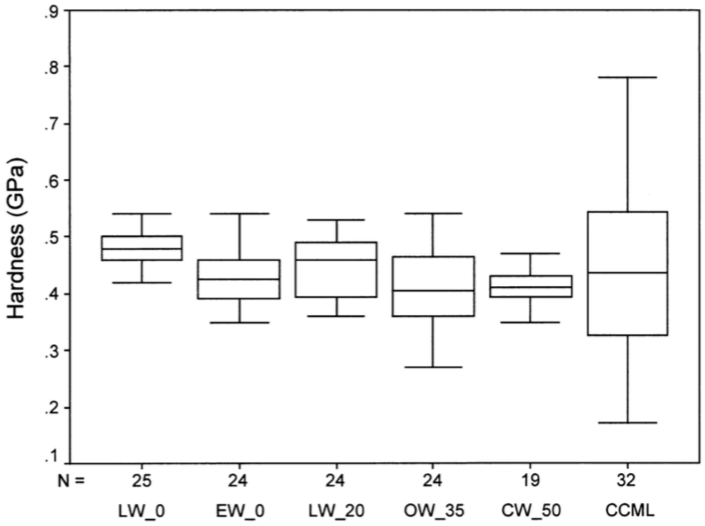


Eder et a. Wood Sci Tech, 2013

IMPACT OF MFA

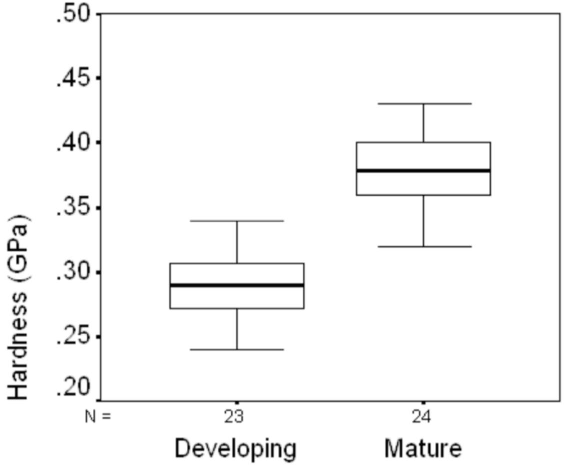


Gindl, Applied Physics, 2004

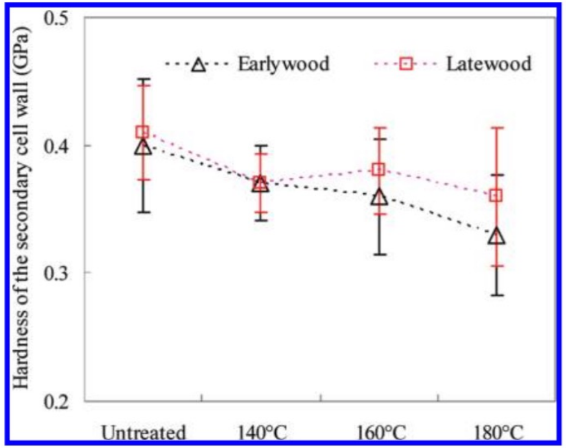


Significant on M, but low on H

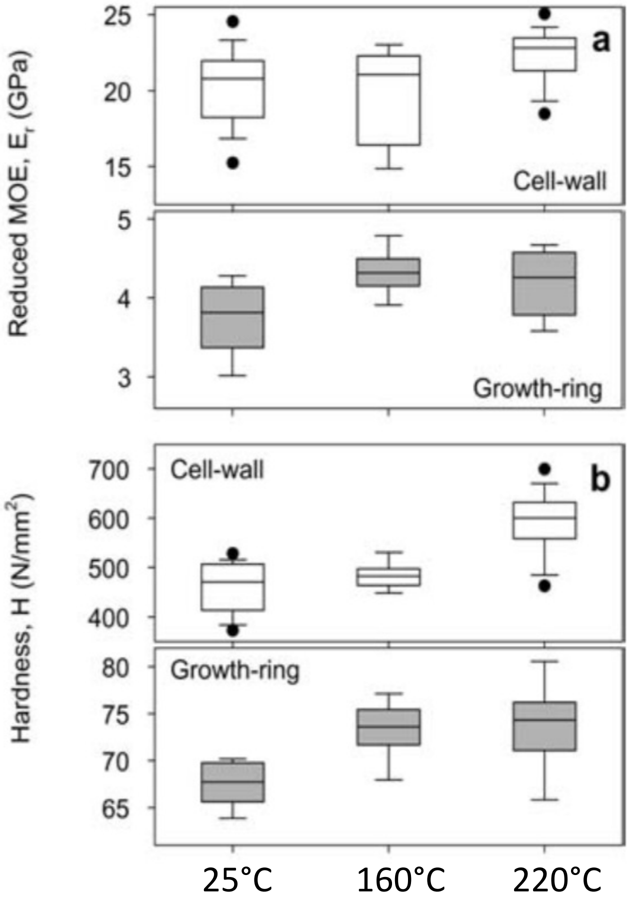
IMPACT OF NON CELLULOSIC POLYMERS



Gindl, Can J Bot, 2002

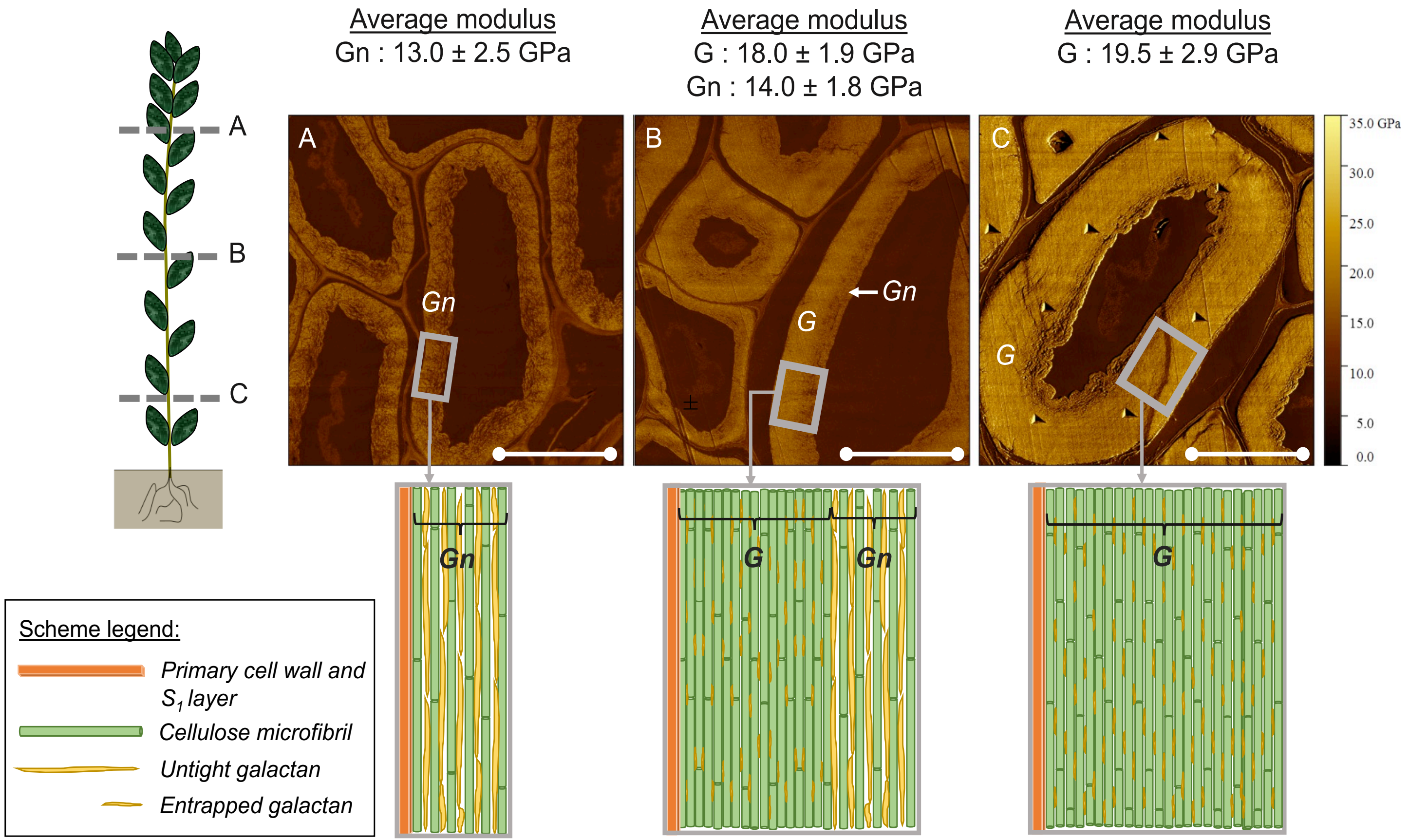


Yin, Biomacrmol, 2011



Stanzl-Tschegg, Holzforschung, 2009

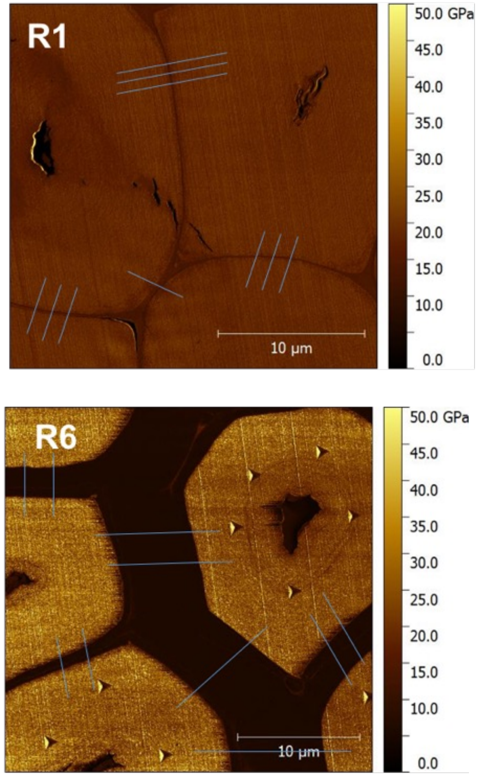
APPLICATION: GROWING FLAX FIBRES (60 DAYS)



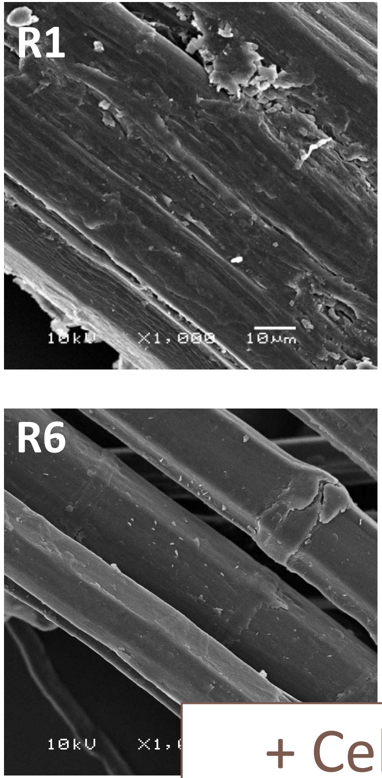
Goudenhooft, Siniscalco, Arnould, Sire, Gorshkova, Bourmaud, Baley, Fibers, 2018

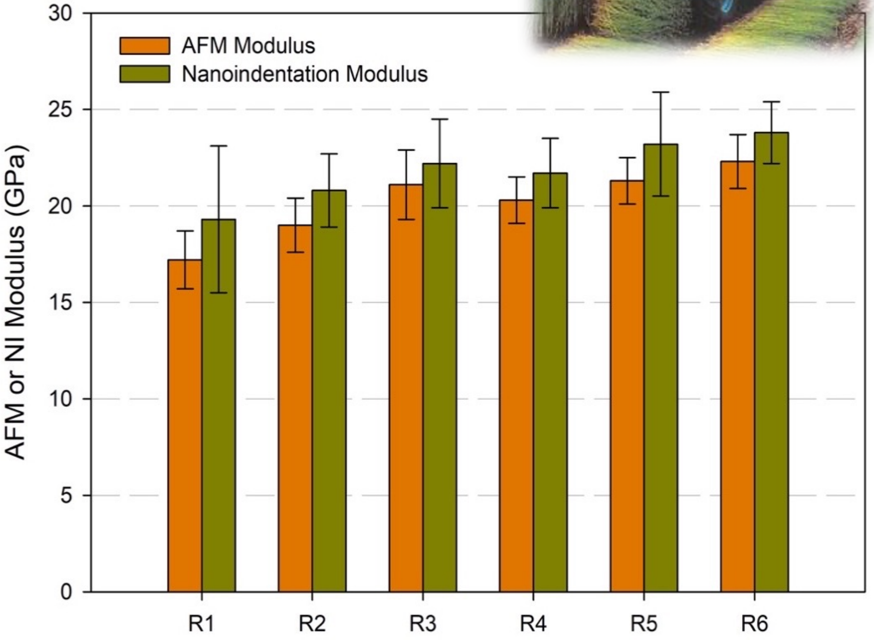
APPLICATION: IMPACT OF RETTING

Contact Modulis (Gpa)



SEM Images

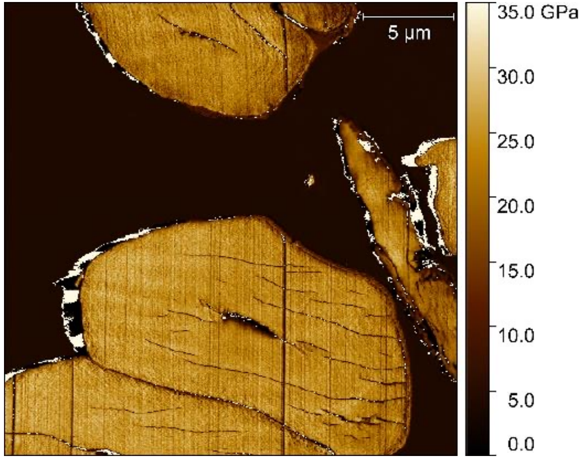




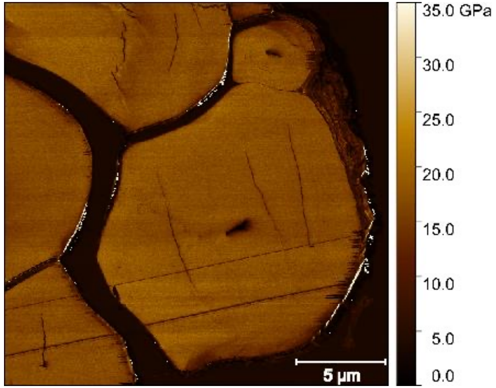
+ Cellulose analysis by XRD and NMR
(↗cristallinité, macrofibrils compaction)

Bourmaud, Beaugrand, Siniscalco, Arnould, Baley, Carbohydrate Polymers, 2019

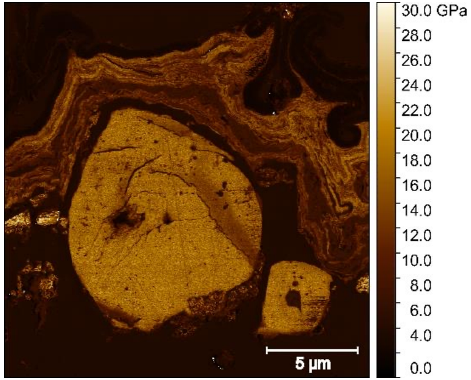
APPLICATION: CULTURAL HERITAGE



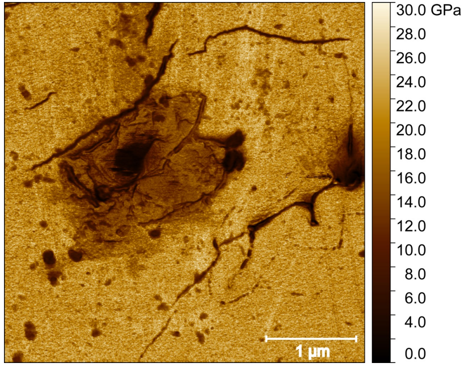
Giulio Benso, San Cristoforo, 1590



Nicola Monti, Madonna col bambino, 1765<<



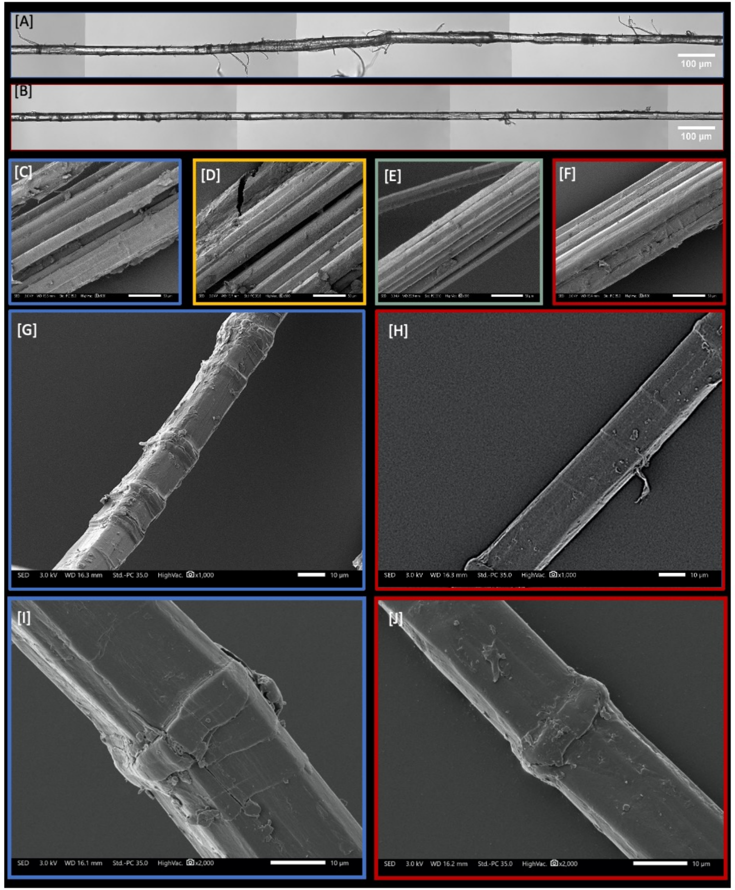
Tommaso Sciacca, Crocifissione, 1765

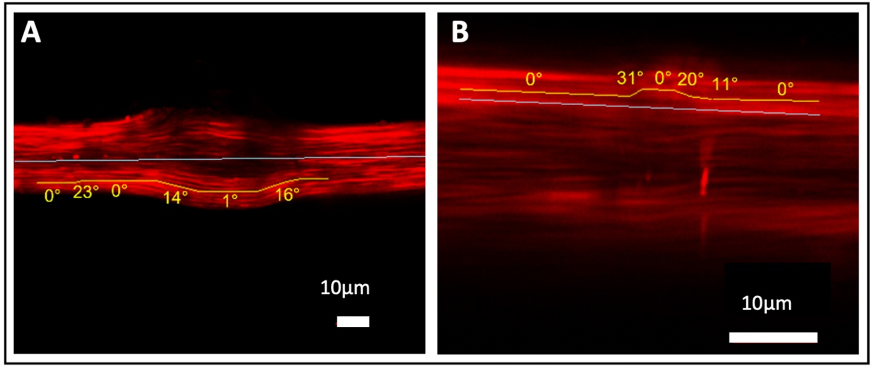


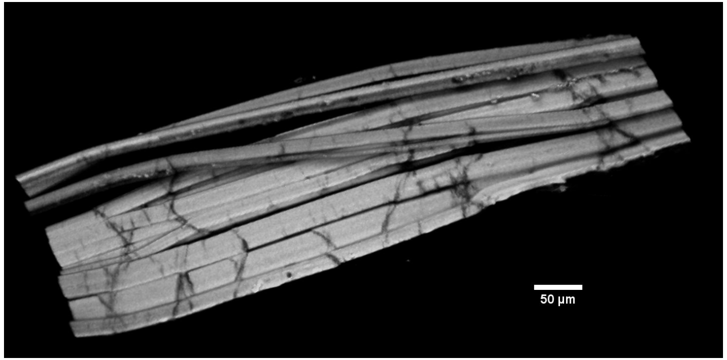
Melelli et al. J of Cult Heritage, 2021

KINK-BAND: A SPECIFIC AND SENSITIVE REGION

KINK BANDS

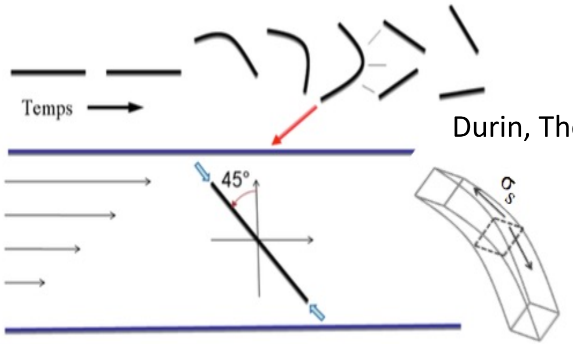




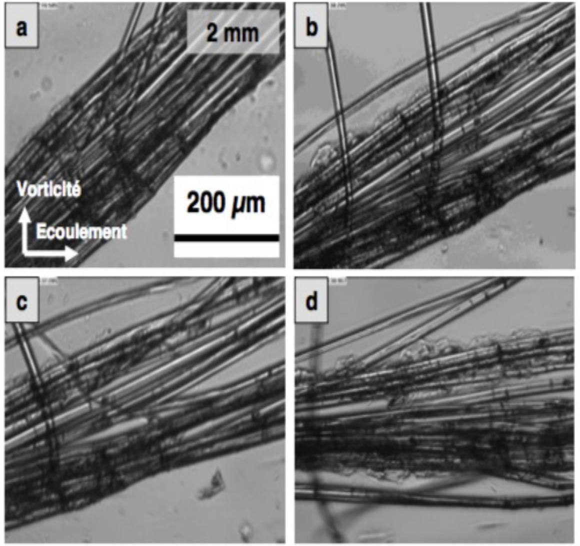


Morgillo et al. Ind Crops & Prod, under writting

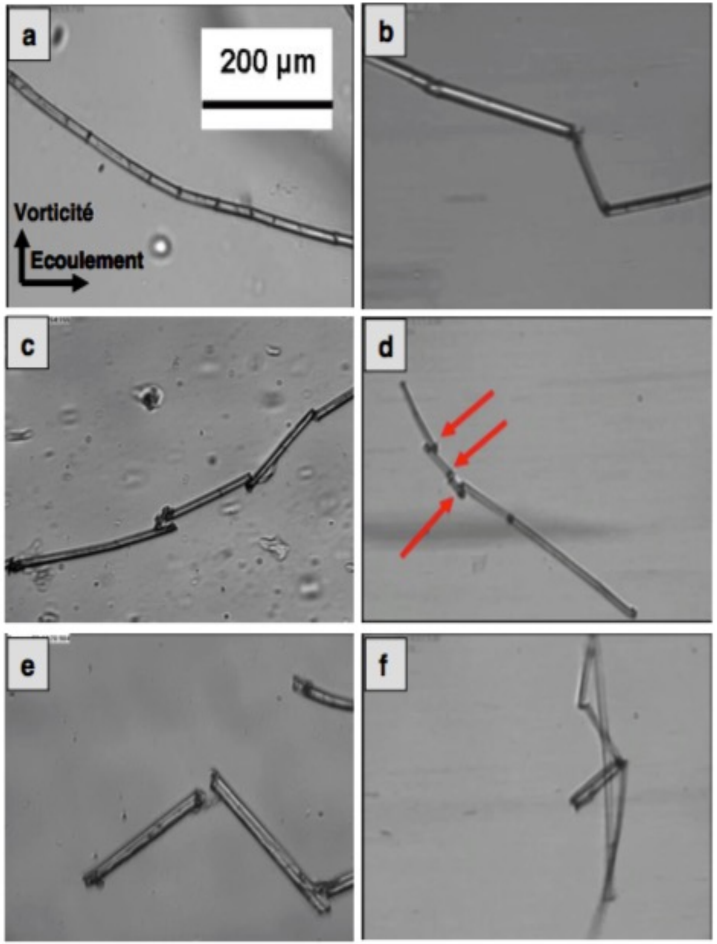
KINK BANDS



Durin, Thèse, 2012

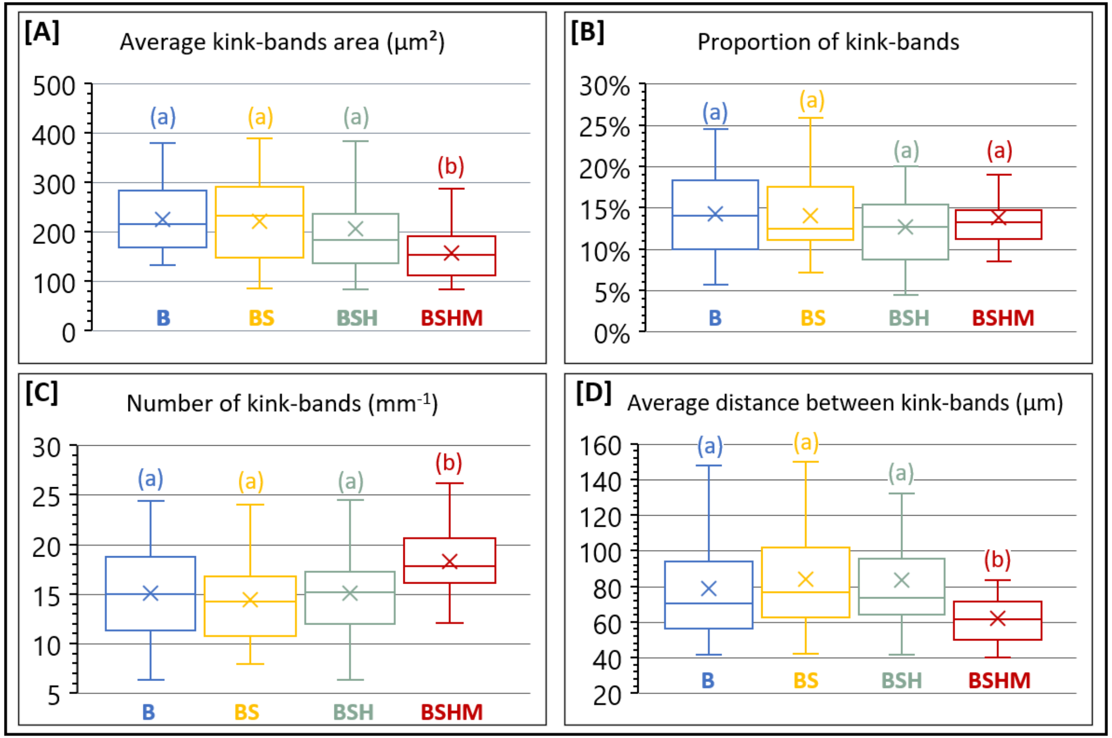


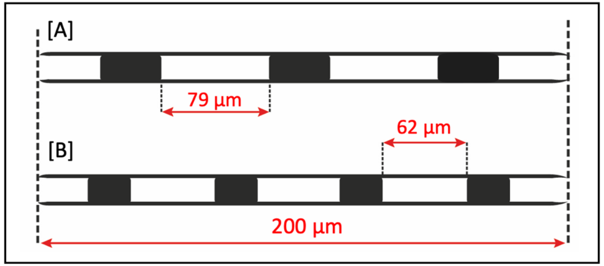
Le Duc, Thèse, 2014



Le Duc et al., Comp Part A, 2011

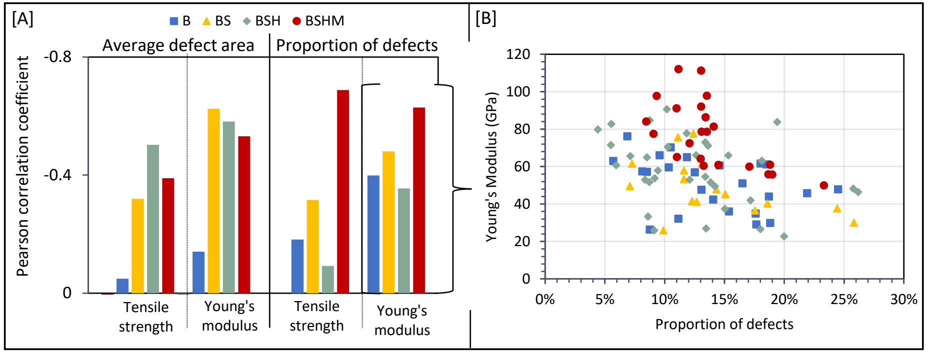
KINK BANDS

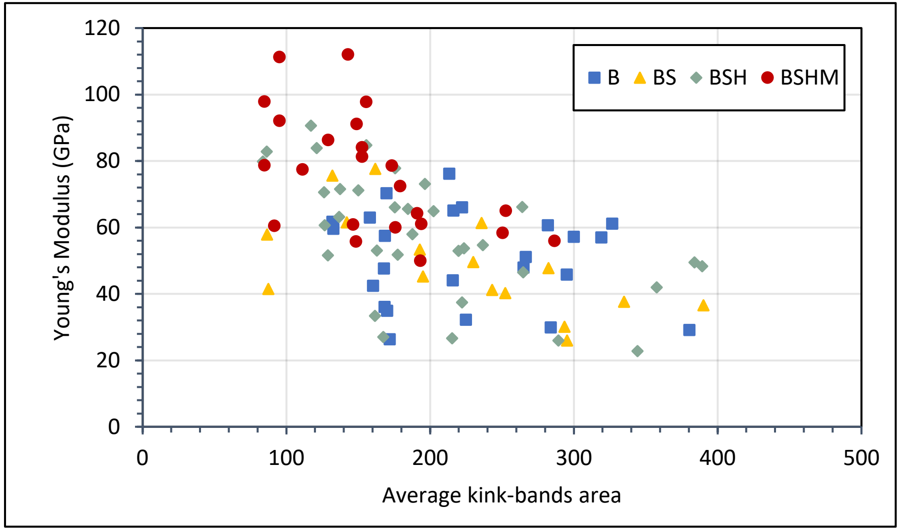




Morgillo et al. Ind Crops & Prod, under writting

KINK BANDS – LINK WITH MECHANICAL PROPERTIES

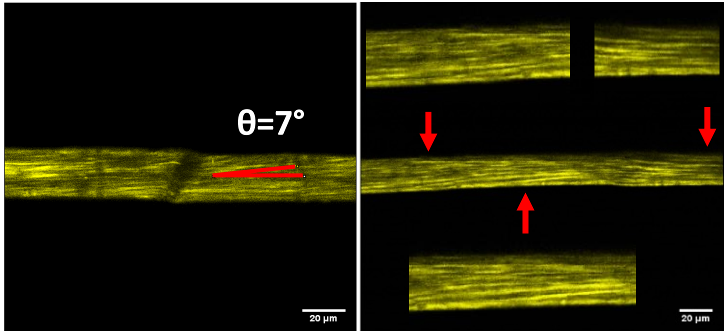


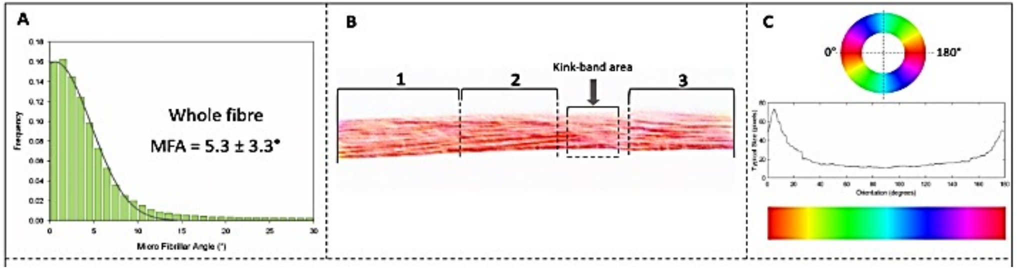


Morgillo et al. Ind Crops & Prod, under writting

KINK BANDS – PROPERTIES AND STRUCTURE

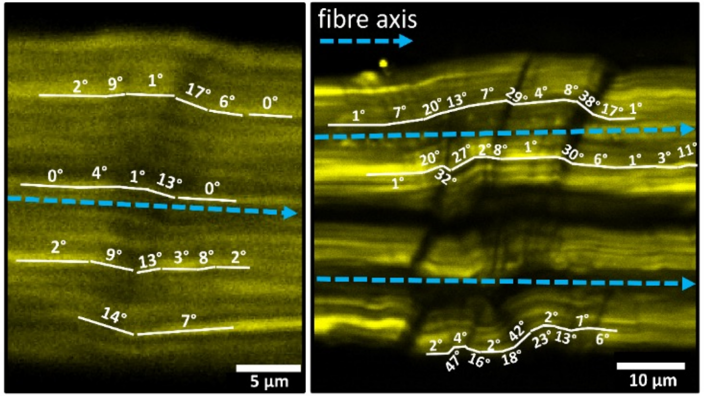
Melelli et al., *Ind Crops Prod*, **2020**



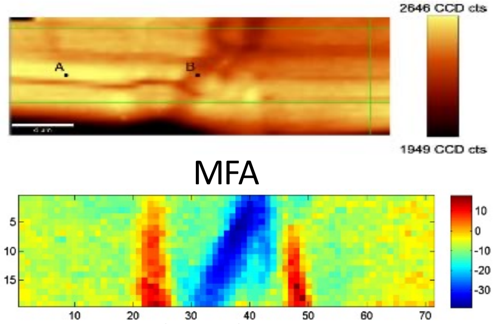


MFA of flax Bolchoï variety: 5.3 ± 3.3°

Melelli et al., *Ind Crops Prod*, **2021**



Polarised Raman microspectroscopy on **hemp**



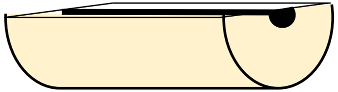
+10° -30°

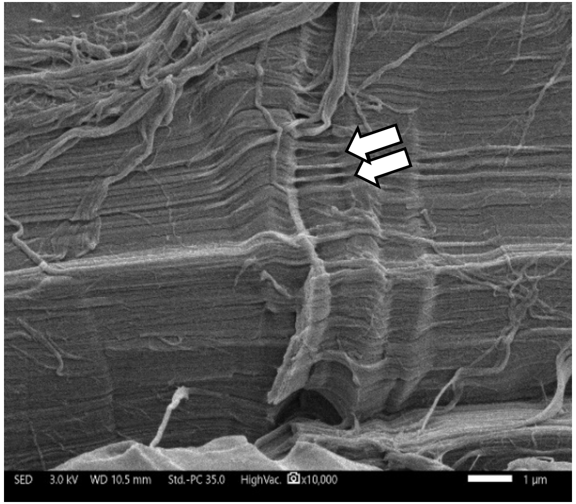
Thygesen et Gierlinger, *J. Struct. Biol.*, **2013**

Melelli et al. Ind Crops & Prod, 2020

KINK BANDS – PROPERTIES AND STRUCTURE

Fibre cut in a half

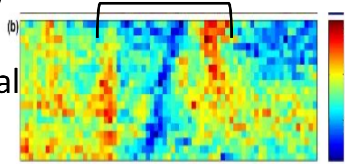




Detachment of macrofibrils because some macrofibrils less deviated than others -> **creation of cavities and pores**

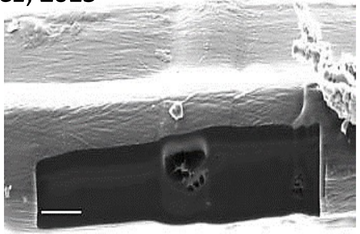
Thygesen et Gierlinger, *J. Struct. Biol.*, **2013**

Crystallinity calculated with Agarwal method I_{380}/I_{1096}

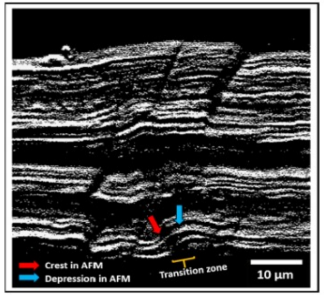


Zhang et al., *WCE*, **2015**

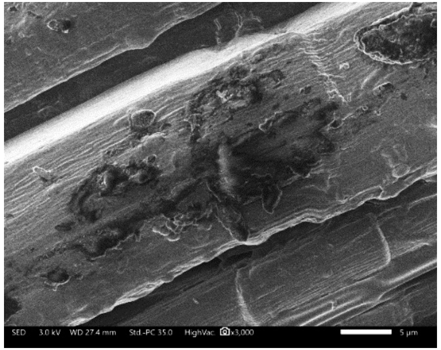
FIB-SEM



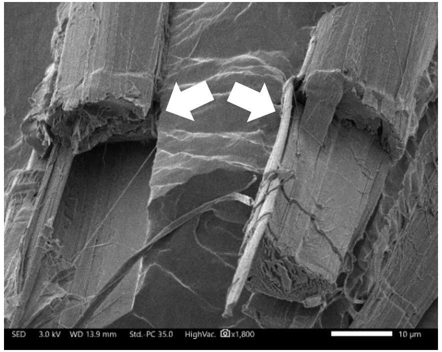
SHG



Biological colonization



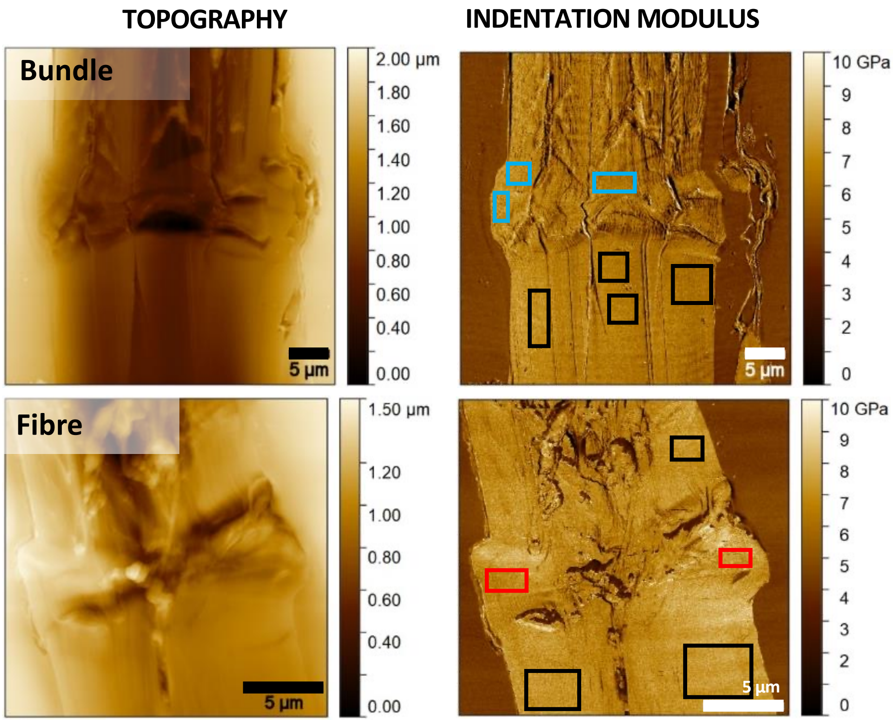
Highly prone to fracture

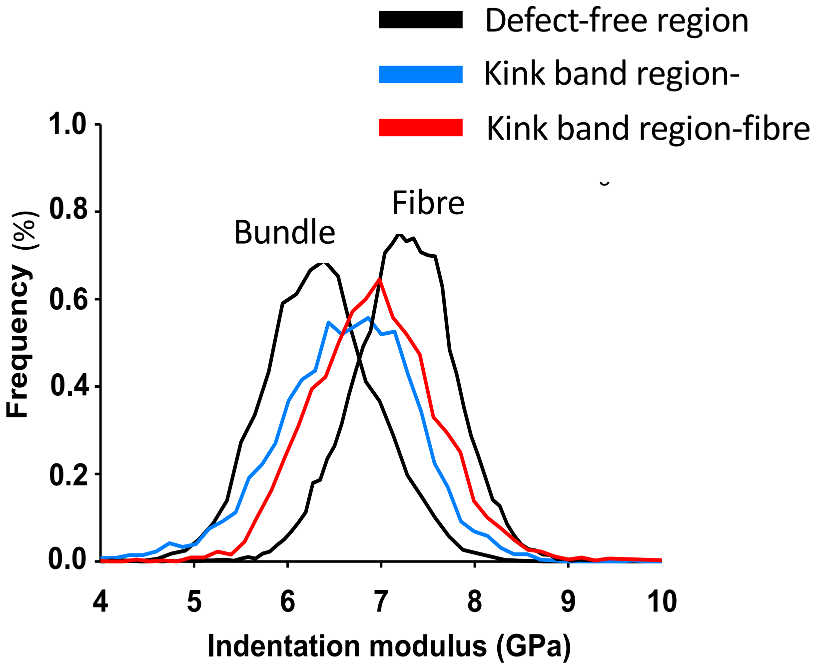


Melelli et al. Ind Crops & Prod, 2020

KINK BANDS – PROPERTIES AND STRUCTURE

Fibres cut in a half



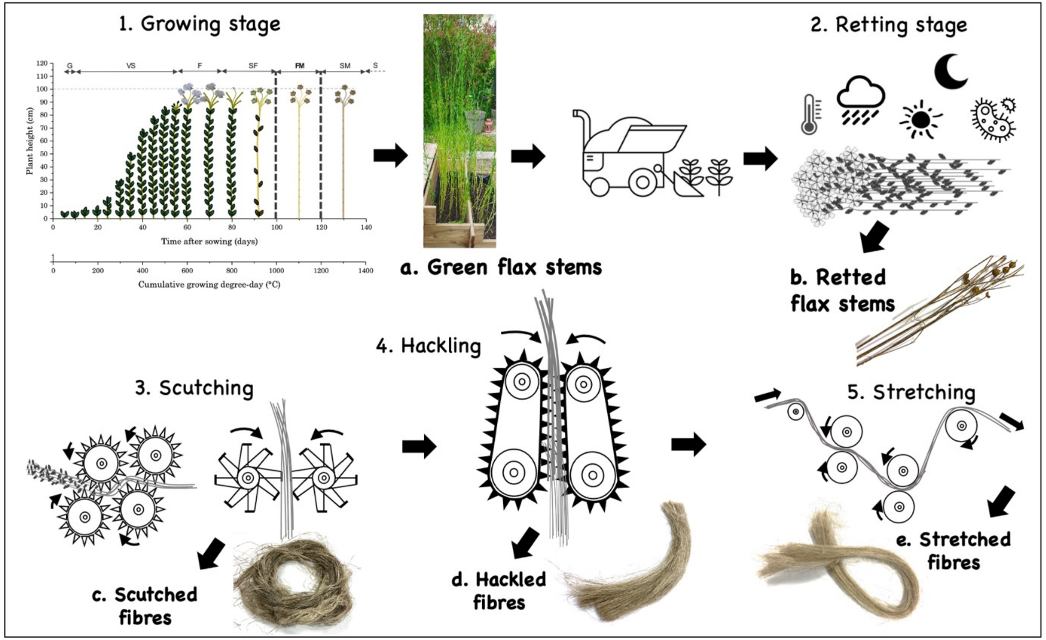


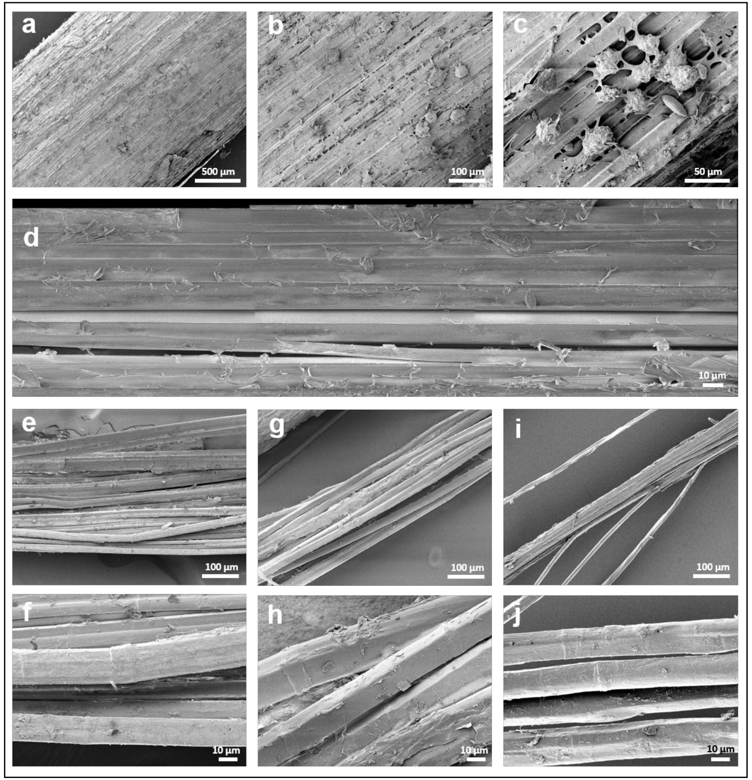
Stiffness:
Kink-band areas **6.9 ±1.2 GPa**
Defect-free regions **6.5 ± 1.5 GPa**

Melelli et al. Ind Crops & Prod, 2020

SYNCHROTRON INVESTIGATIONS

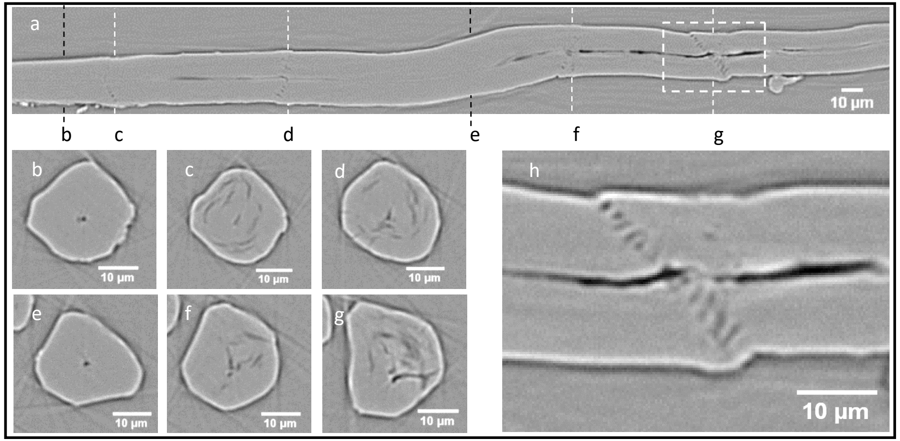
ELUCIDATING THE DEVELOPMENT OF KINK BANDS

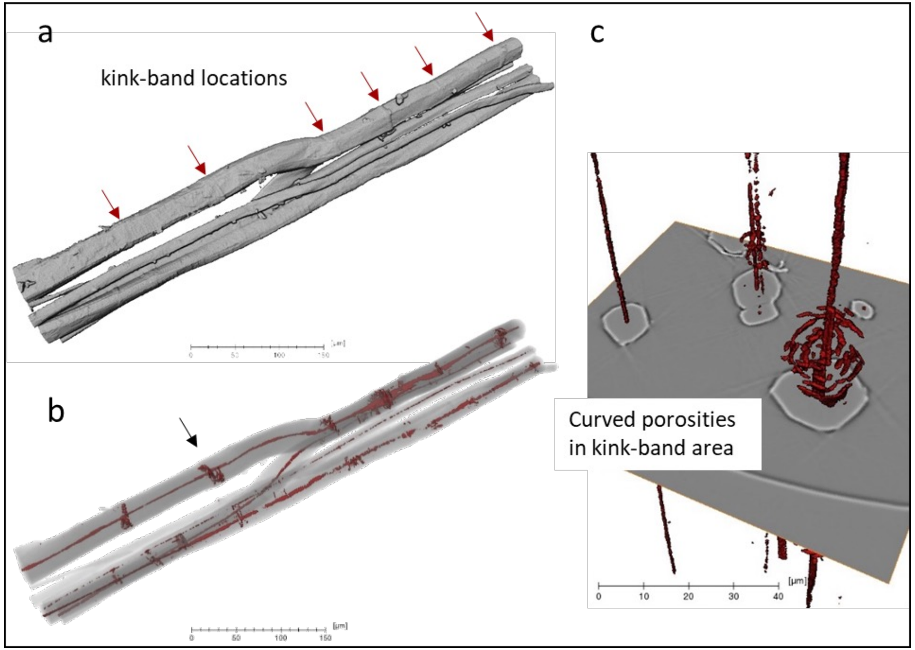




Bourmaud et al, Ind Crops & Prod, 2022

ELUCIDATING THE DEVELOPMENT OF KINK BANDS

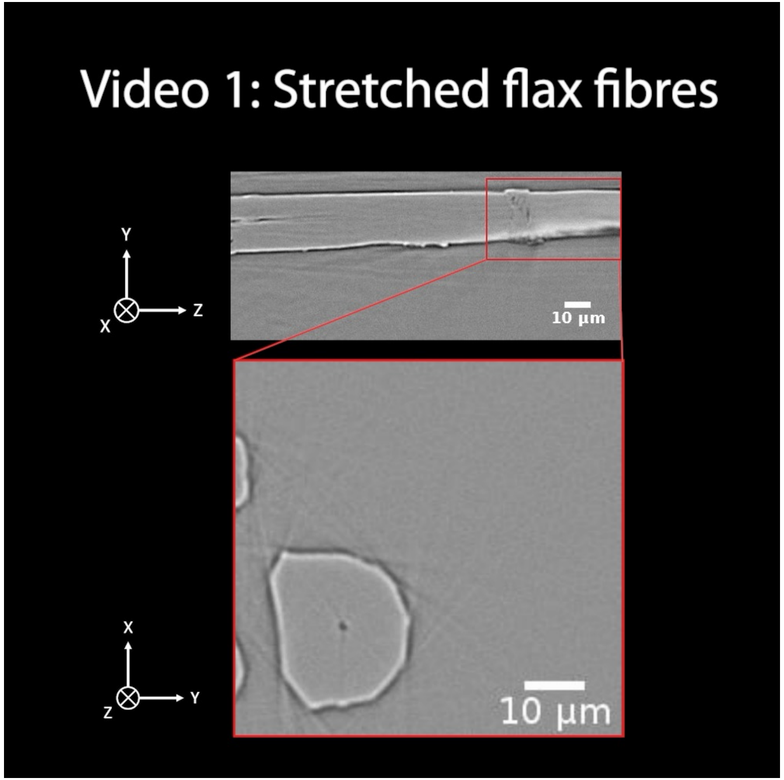


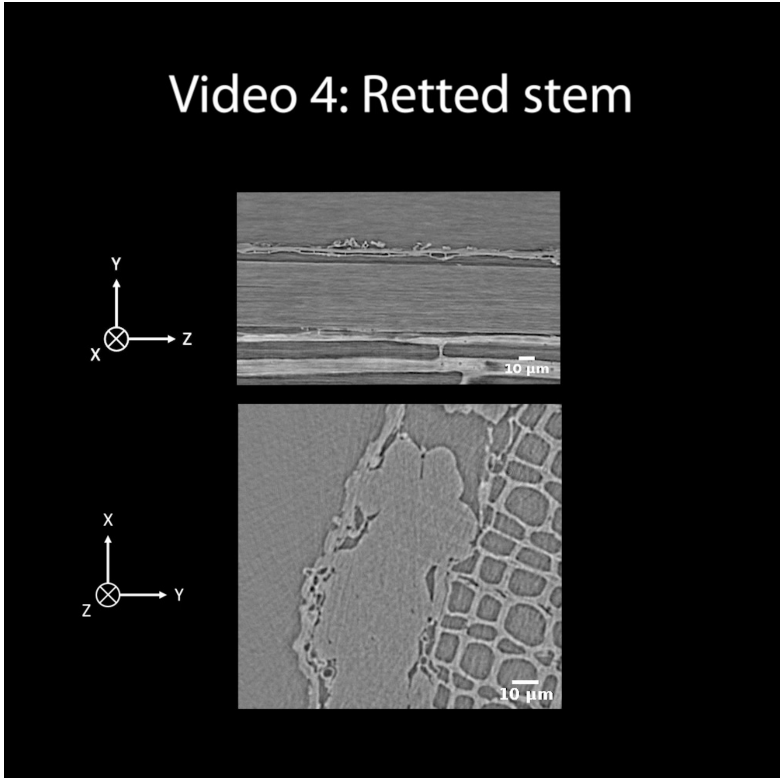


Observations on extracted fibres

Bourmaud et al, Ind Crops & Prod, 2022

ELUCIDATING THE DEVELOPMENT OF KINK BANDS





Bourmaud et al, Ind Crops & Prod, 2022

COMPLEX STRUCTURE OF KINK BANDS

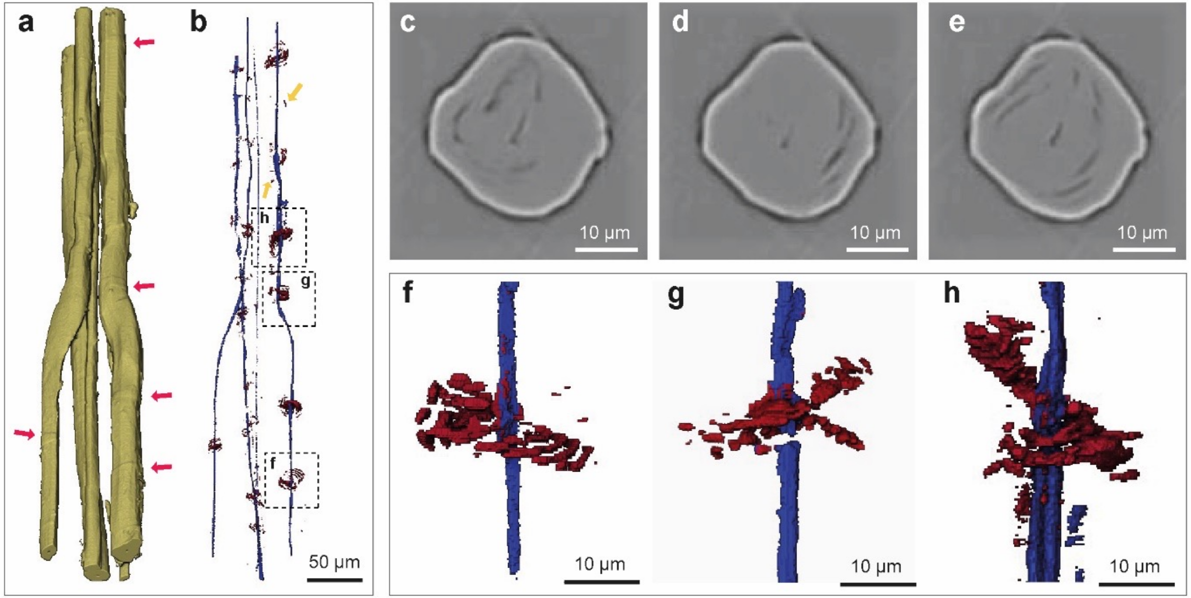


Figure 1. 3D reconstruction of the fibre, before (a) and after (b) segmentation, tomographic slice images of fibres along transverse planes where voids can be seen as dark shapes (c, d, e), 3D reconstruction of porosities in red around the lumen in blue

Quereilhac et al, Ind Crops & Prod, 2023

COMPLEX STRUCTURE OF KINK BANDS

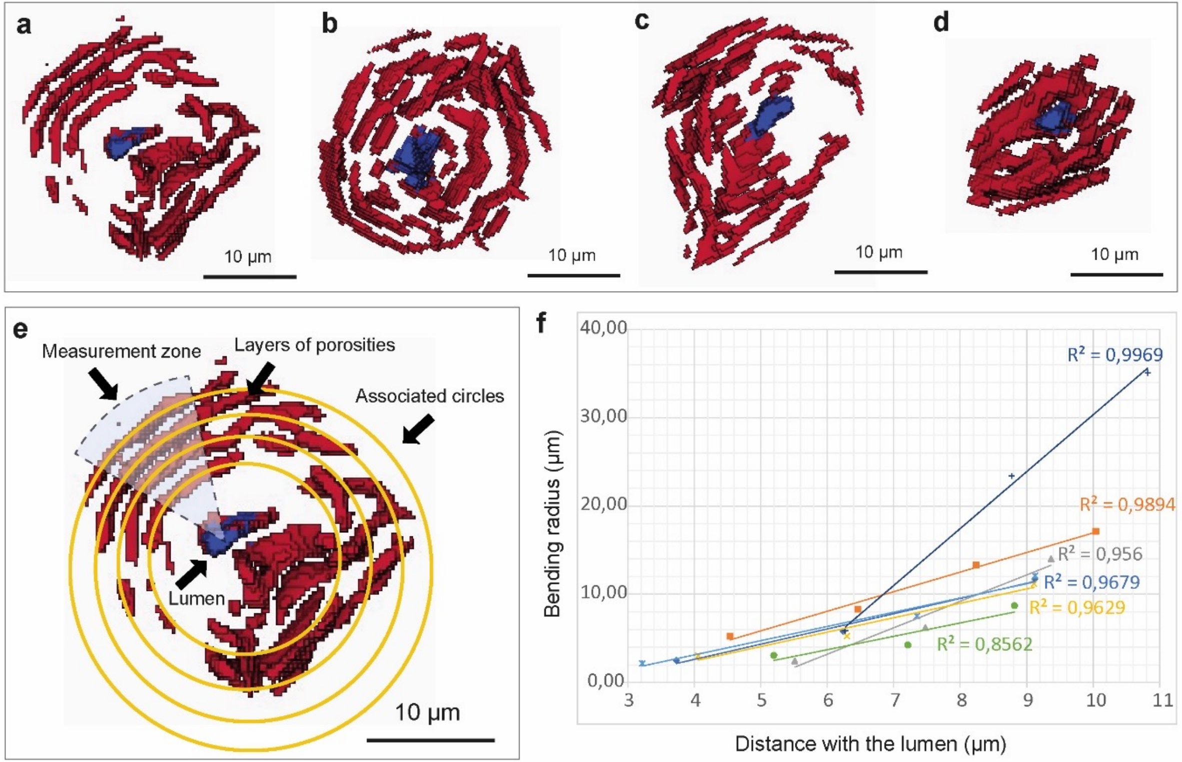


Figure 2. Defects viewed from the Z axe, in blue the lumen can be seen, and the pores layers are in red showing a distinct onion layer organisation (a-d), detail of the measurement method for the curvature radius (e), linear regression of the radius of curvature of the pores layers evolving with the distance with the lumen axis (f)

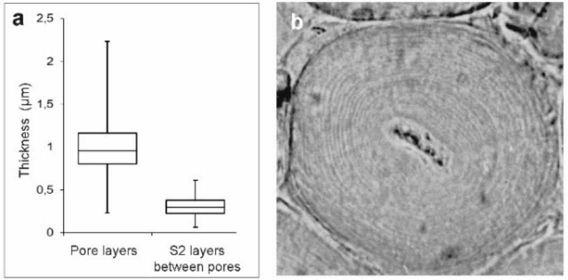
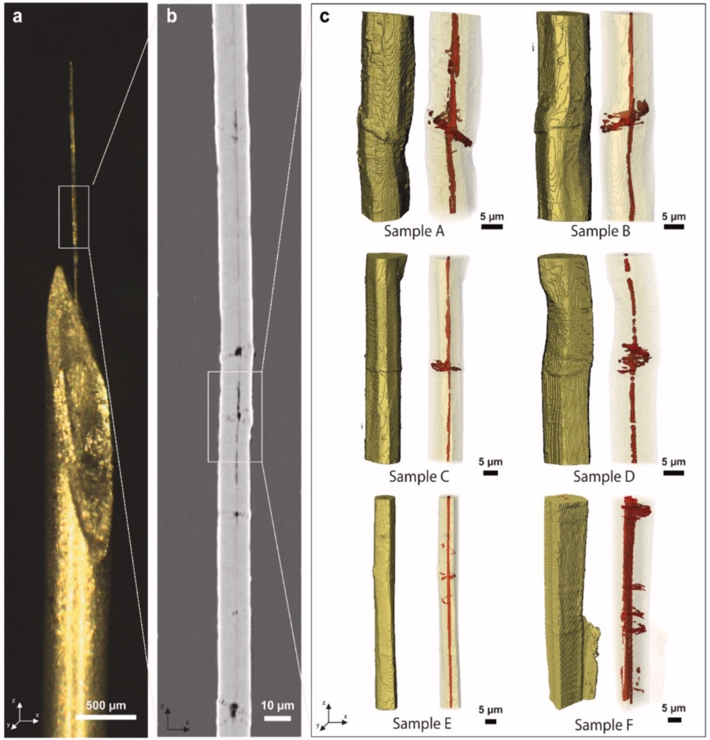
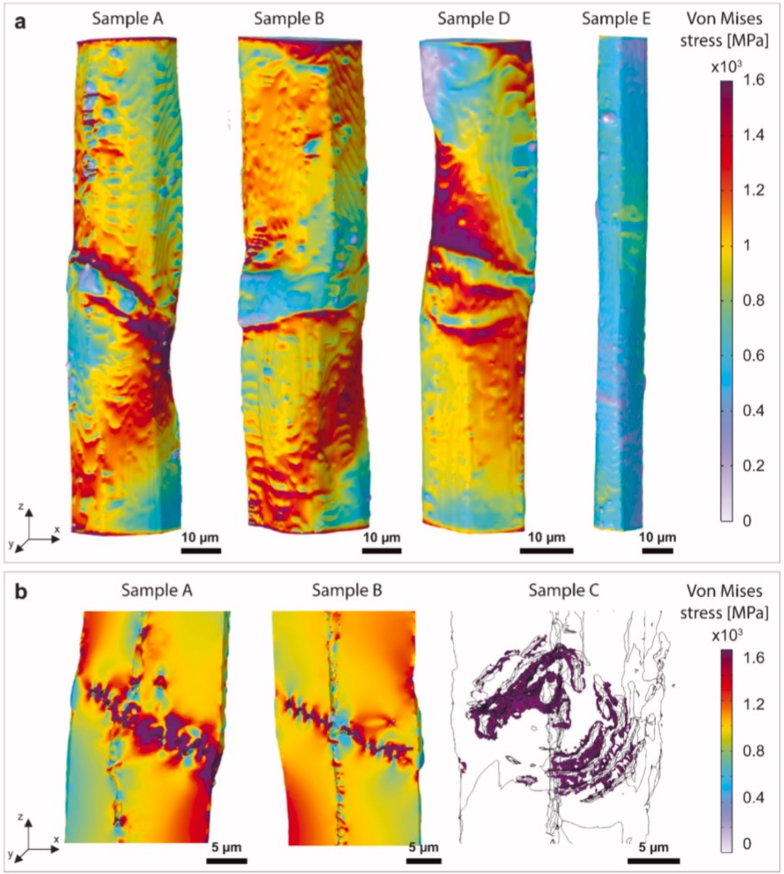


Figure 3. Box-plot presenting the pores thickness and the distance between two pores (a), cross-section of a flax fibre evidencing the successive cellulose layers (b) (Hock, 1942)

Quereilhac et al, Ind Crops & Prod, 2023

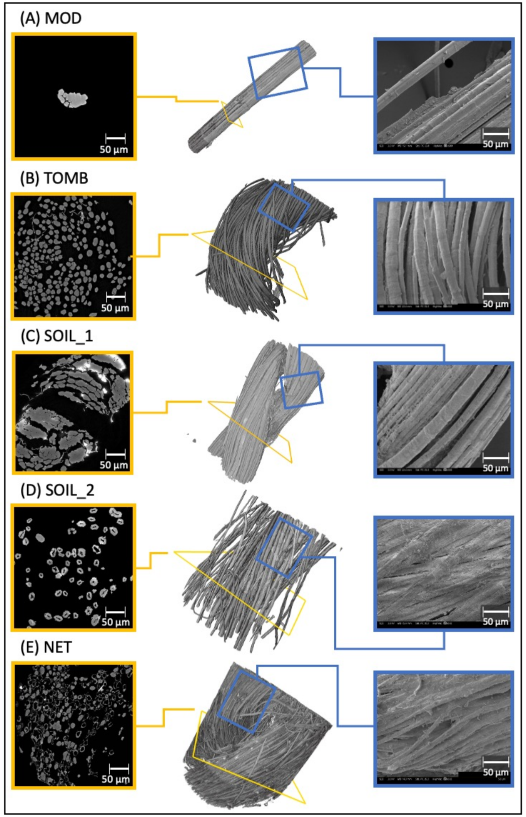
COMPLEX STRUCTURE OF KINK BANDS

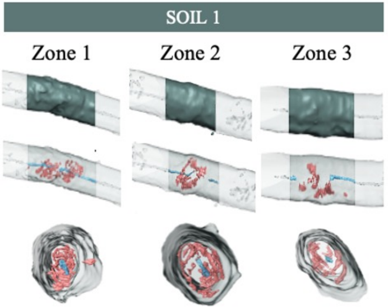


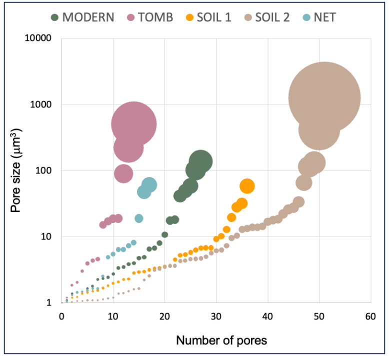


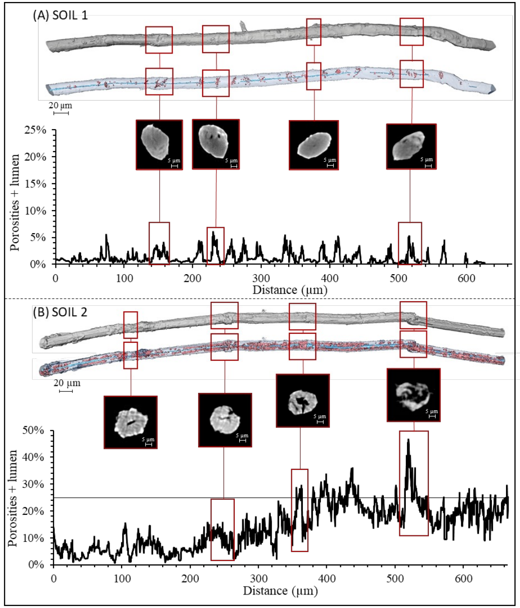
Quereillac et al, Comp Part A, 2024

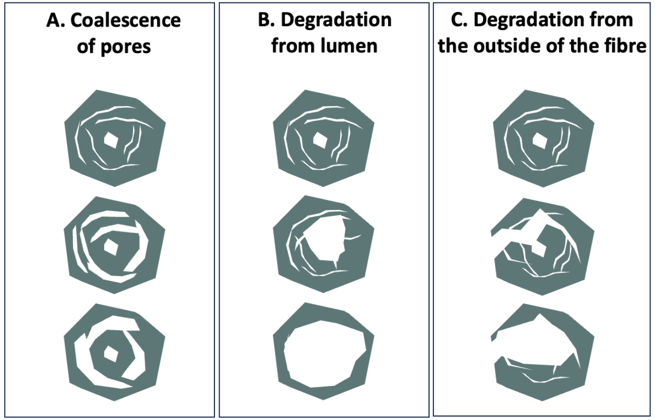
KINK-BANDS AND DURABILITY









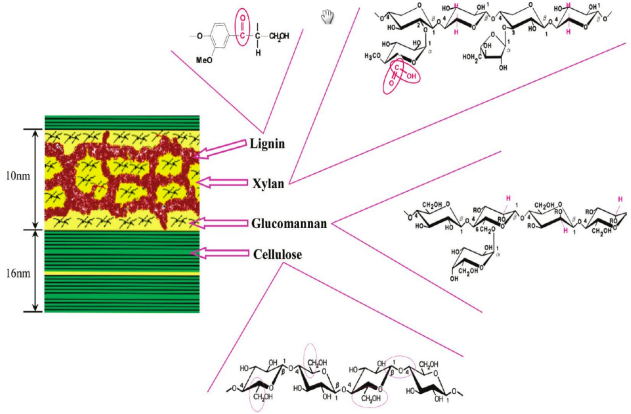


Morgillo, Bourmaud et al, Comp Part B, 2024

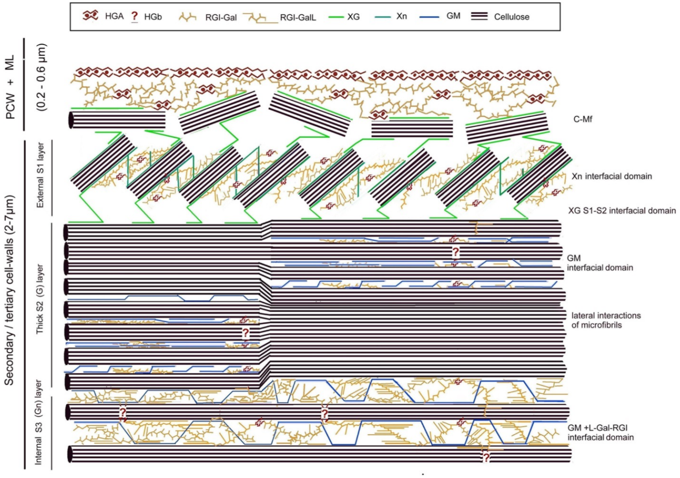
IMPACT OF PROCESS ON PLANT FIBRES AND COMPOSITES PROPERTIES

CASE OF INJECTION AND EXTRUSION

PLANT FIBRES ARE SPECIFIC AND SENSITIVE



Yin et al. Biomacromolecules, 2011



Rihouey et al, Cellulose, 2017

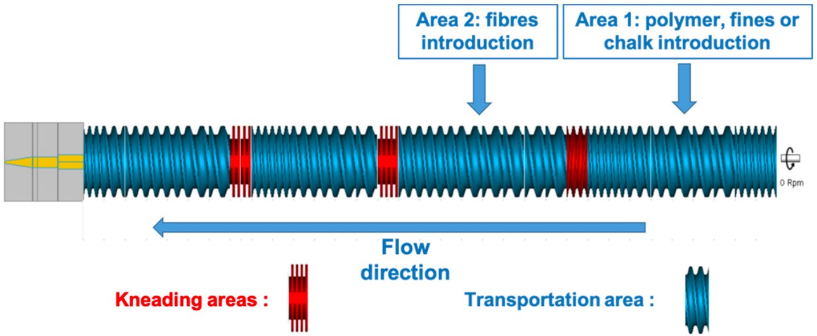
Wood S_2

50% cellulose
25% hemicellulose
25% lignin
+ extractives, ashes
MFA = (0) 8-40°

Flax G

80% cellulose
20% hemicellulose
≈0% lignin
+ protein
MFA = (0) 5-10°

PROCESSING: AN AGRESSIVE STAGE



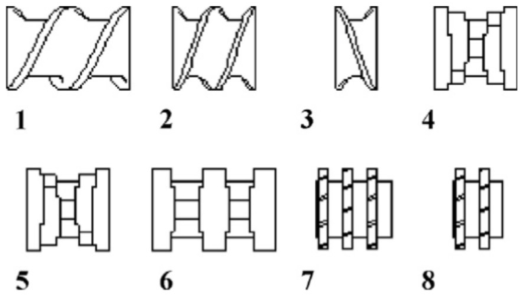
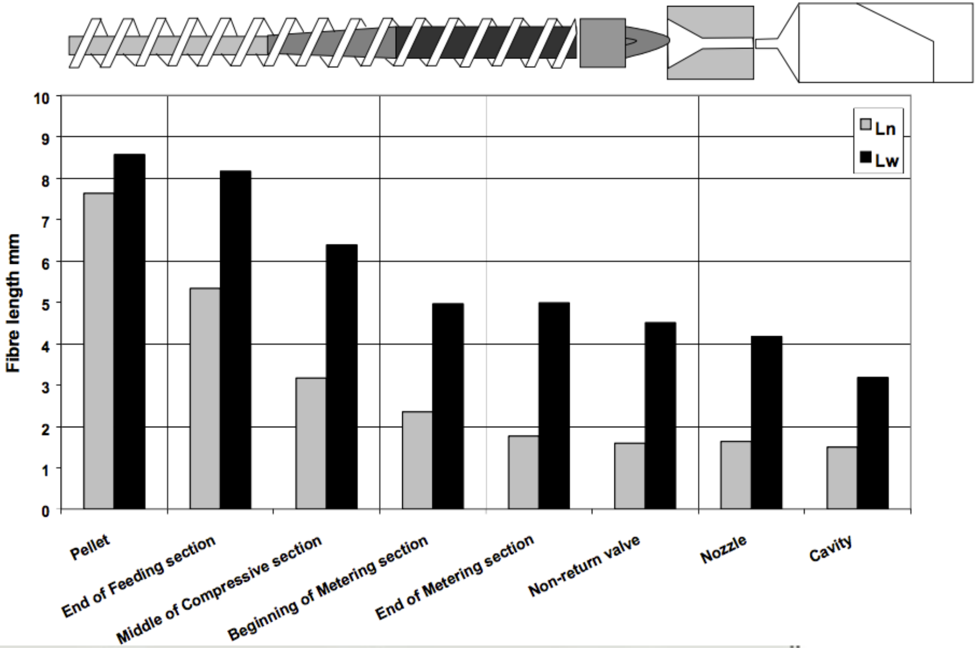


Fig. 2. Screw elements for twin-screw extruder ZE25 from Berstorff; conveying elements (1 and 2), back-conveying element (3), kneading elements (4–6), mixing elements (7 and 8).



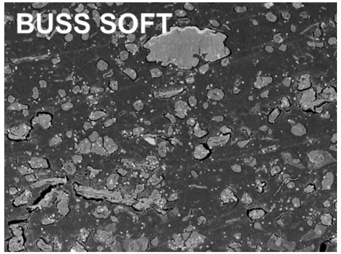
Extr
Rang
1000 10000 100000
SHEAR RATE, 1/s

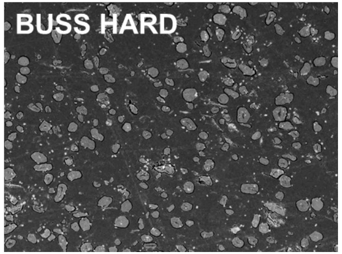
COMPOUNDING

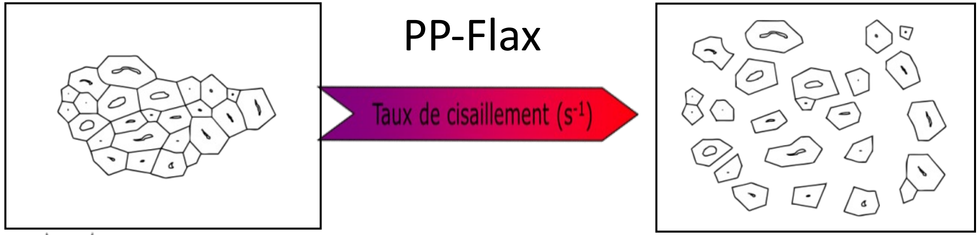
INJECTION

Bourmaud et al., Ind Crops & Prod, 2019 Lafranche et al., Adv in Pol Techno, 2005 Villmow et al., Comp Sc & Tech, 2010 Polyone, doc com, 2015

IMPACT OF PROCESSING TOOL





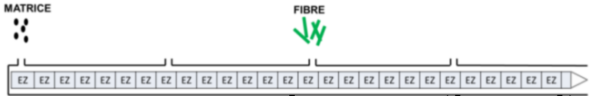


R = 0,79

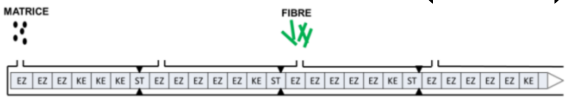
R = 1,34

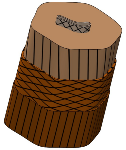
Compounding: 2 ways

Buss Comalaxor (soft)



Buss Comalaxor (hard)





Injection

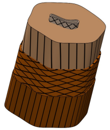
	Buss Soft	Buss Hard
Dispersion factor	0,79	1,34

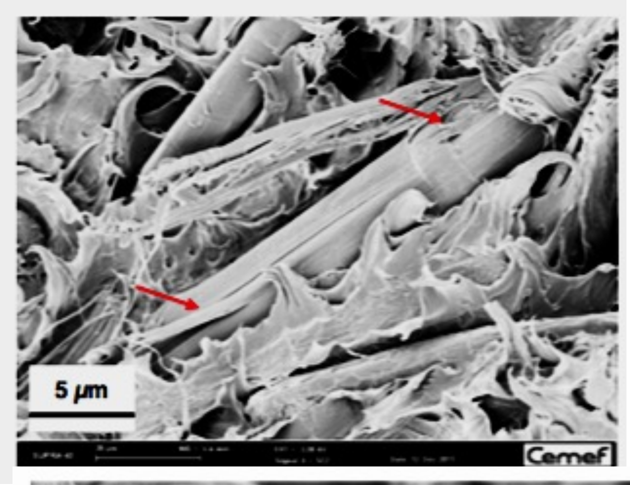
Increase of dispersion, reduction of bundles => Improvement of mechanical properties

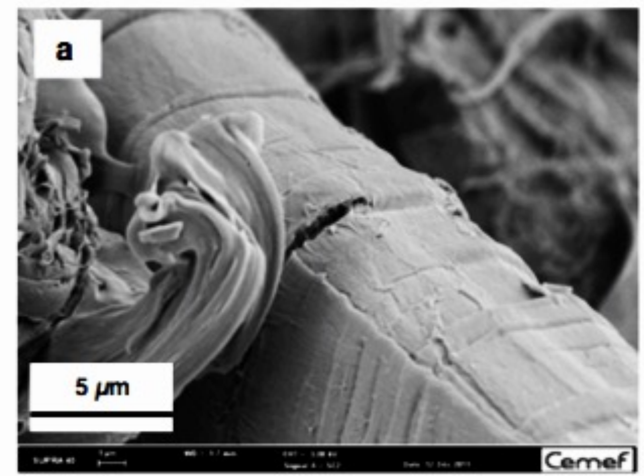
	Buss Soft	Buss Hard
Stress (MPa)	23,8	28,1

Coroller, Phd Thesis, UBS-LIMATB Lorient, 2013

PROCESSING: AN AGRESSIVE STAGE

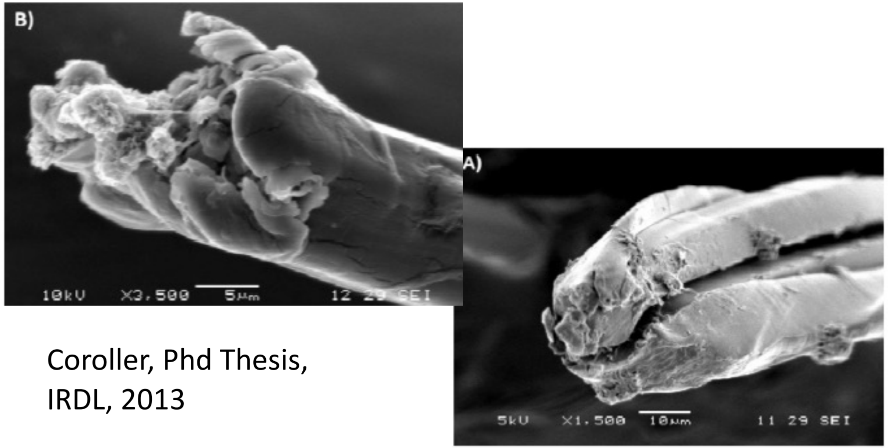






Le Duc, PhD Thesis, 2014

Baley, Macromol Symp, 2005

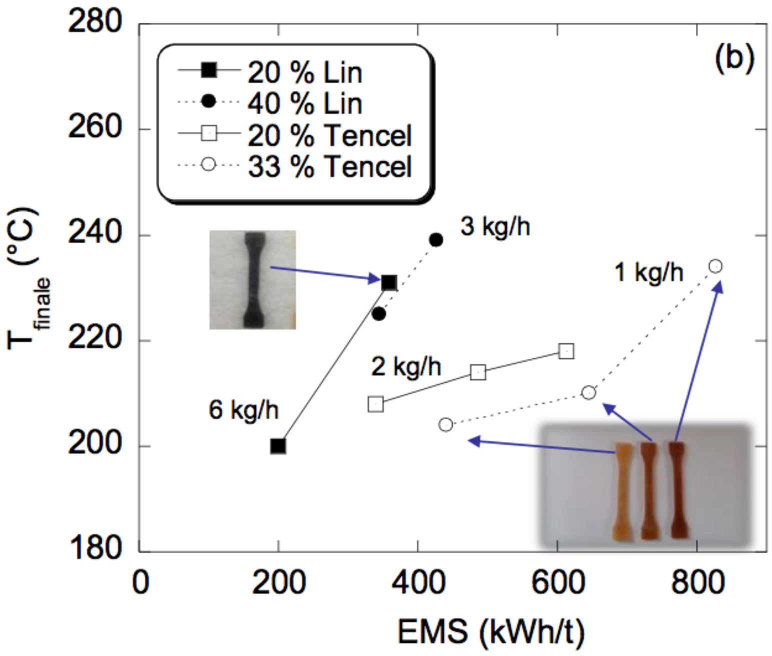


Coroller, Phd Thesis, IRDL, 2013

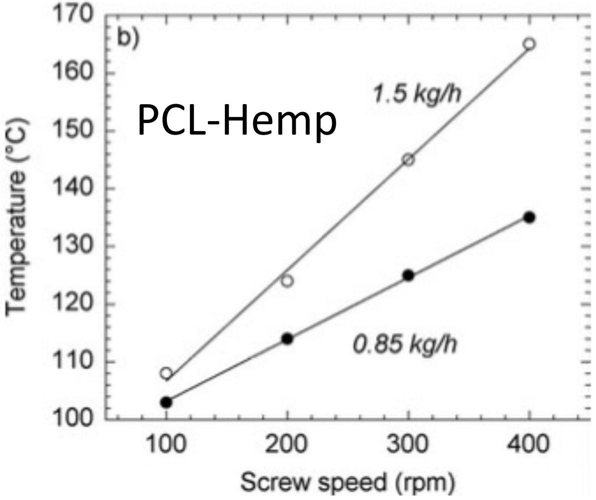
PROCESSING: AN AGRESSIVE STAGE

Temperature is related to the polymer properties but only defining a temperature is too simplistic

Self-heating



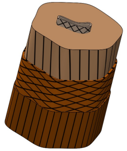
Le Duc, Thèse, 2014



Beaugrand & Berzin, JAPS, 2012

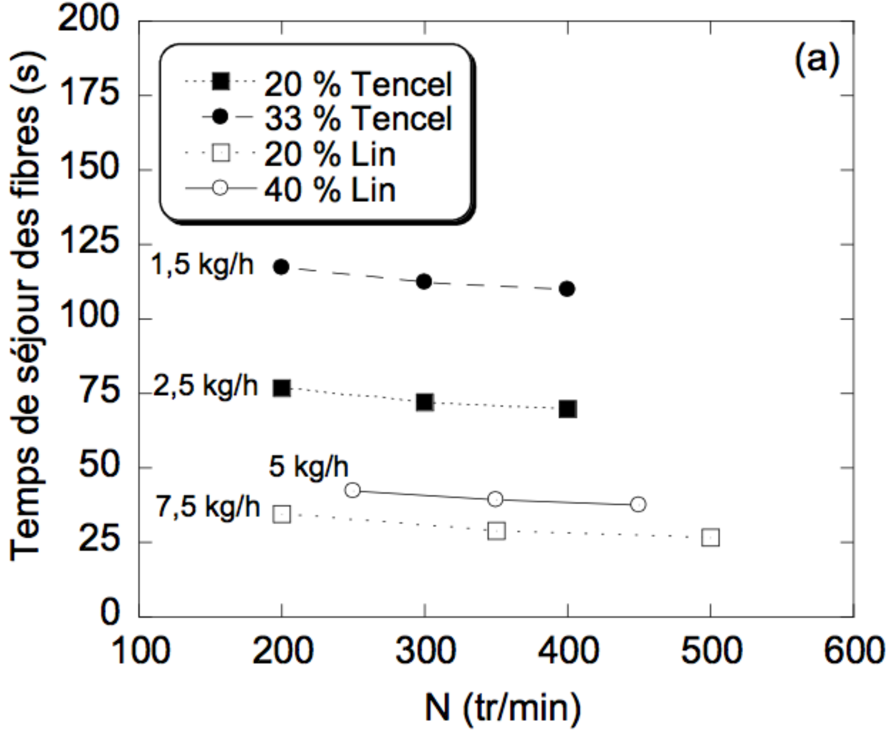
Importance of the fibre content on the *in-tool* temperature

PROCESSING: AN AGRESSIVE STAGE

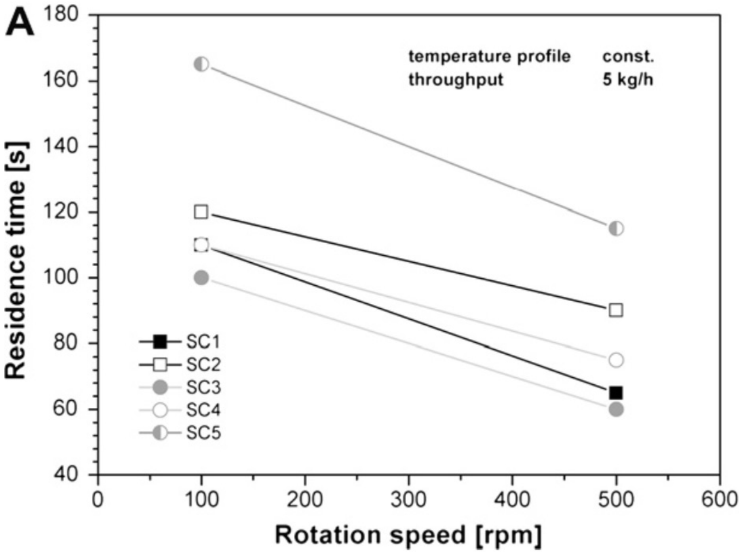


Very different residence times for different tools

Villmow et al., Comp Sc & Tech, 2010



Le Duc, Thèse, 2014

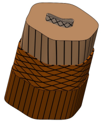


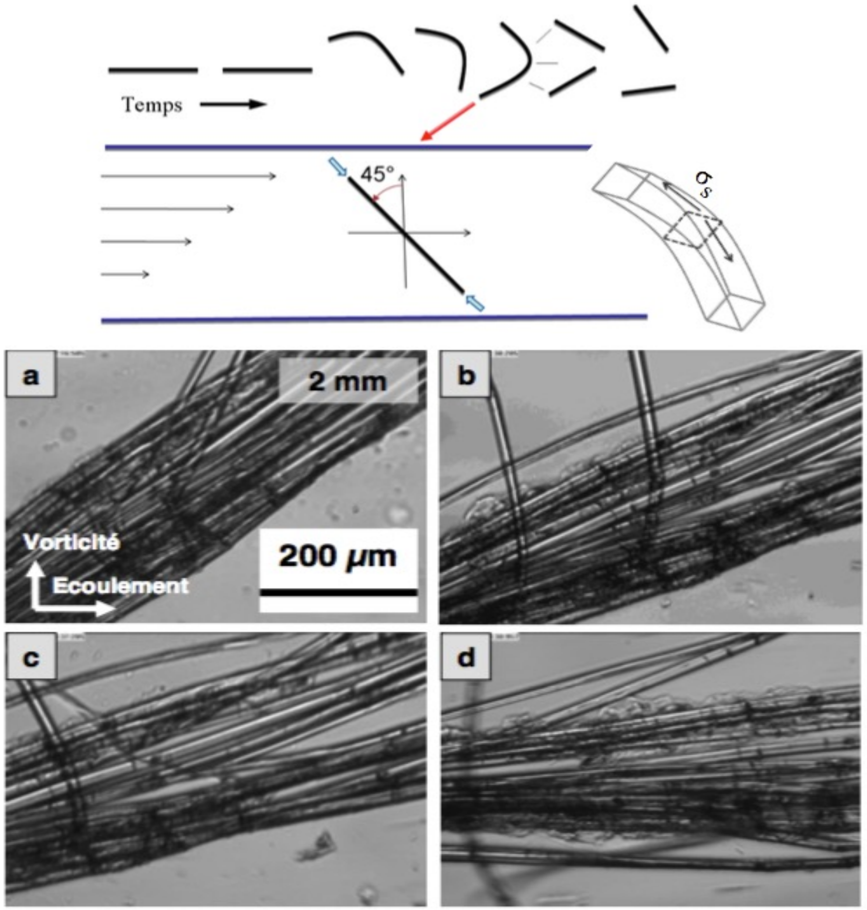
BUSS: 100 to 200 s

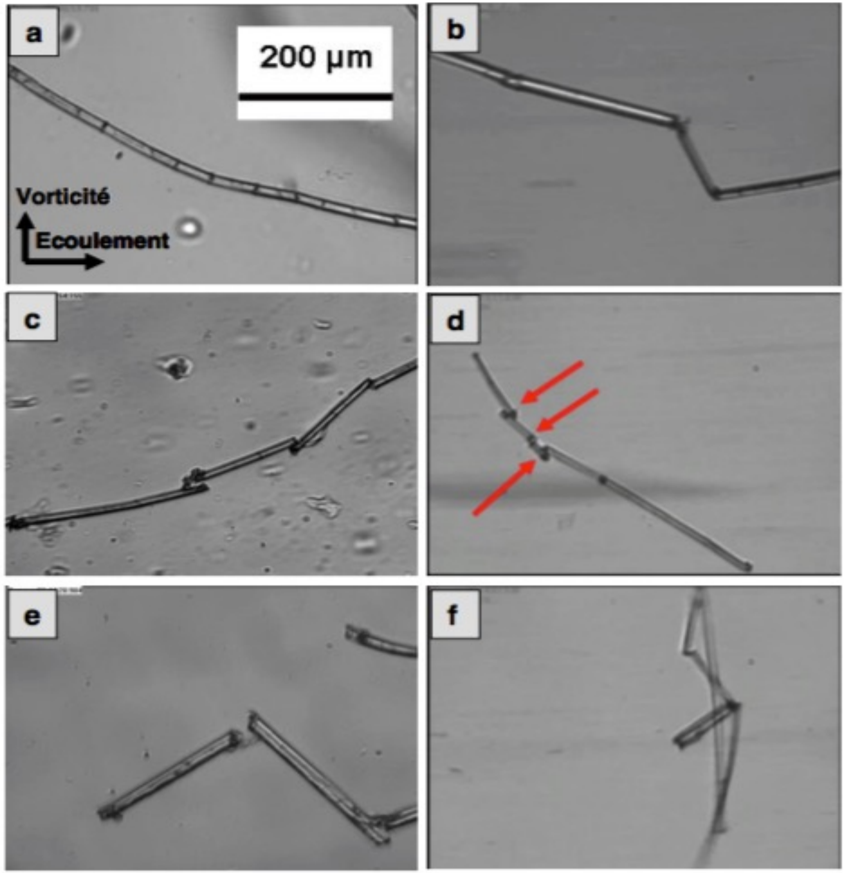
Twin screw: 10 to 180 s

Injection: 30 to 300 s

PROCESSING: MORPHOLOGICAL DAMAGES





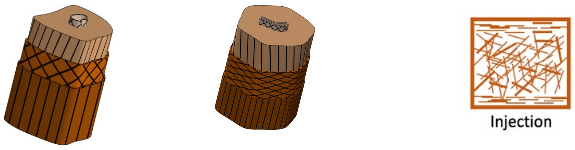


Durin, PhD Thesis, 2012

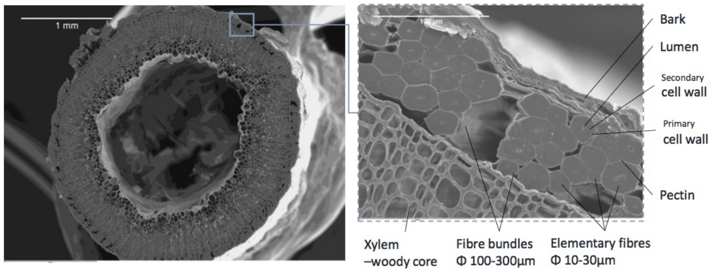
Le Duc, PhD Thesis, 2014

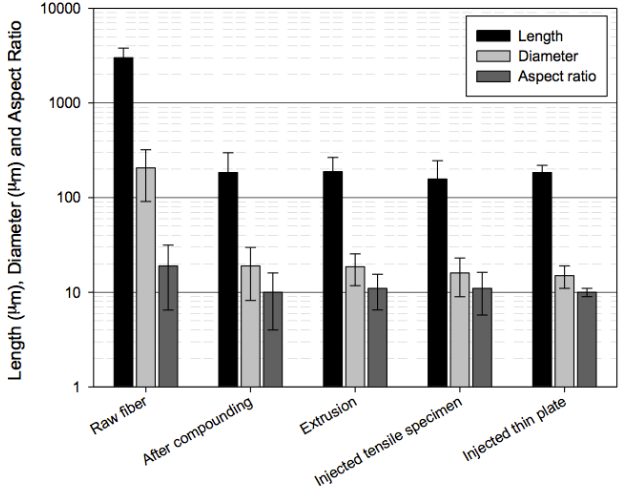
Le Duc et al., Comp Part A, 201

PROCESSING: MORPHOLOGICAL DAMAGES

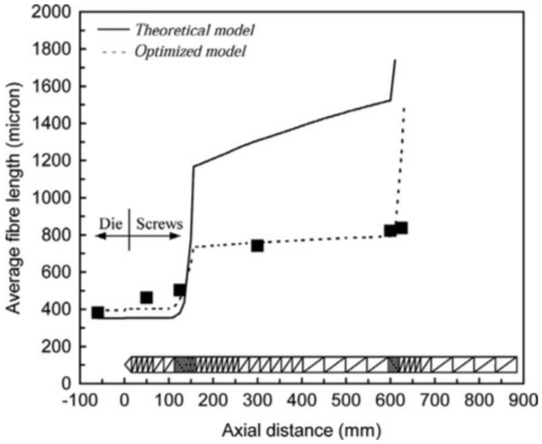


Zheng et al., Comp Part A, 2014



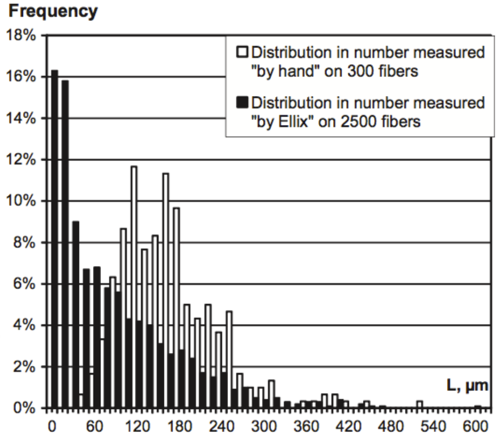


Berzin & Beaugrand, Comp Part A , 2014



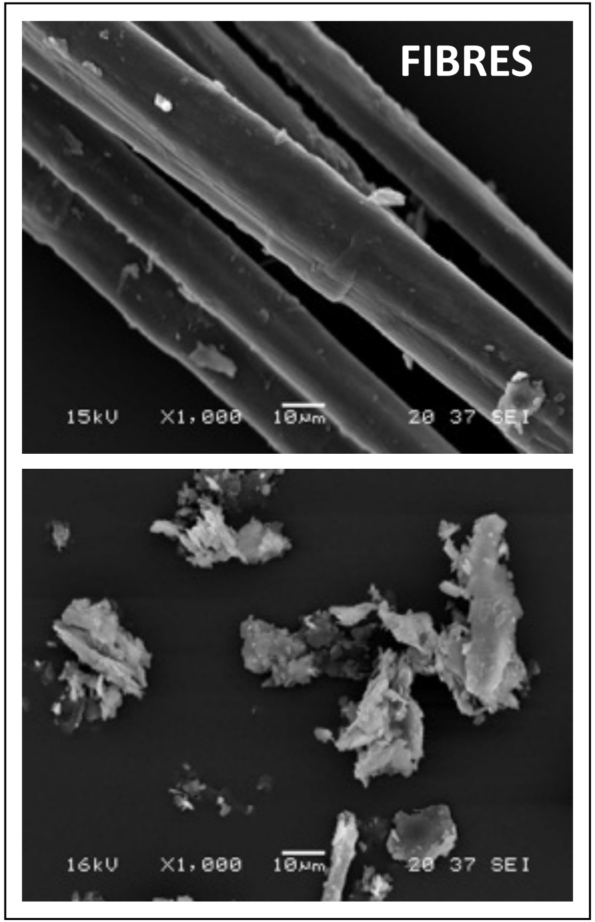
Specificity of plant fibres: fibrillation and appearance of fine particles

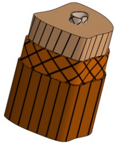
Le Moigne et al., Comp Part A , 2011

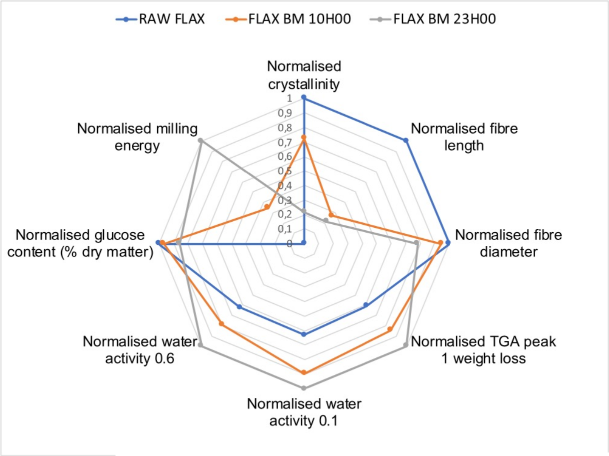


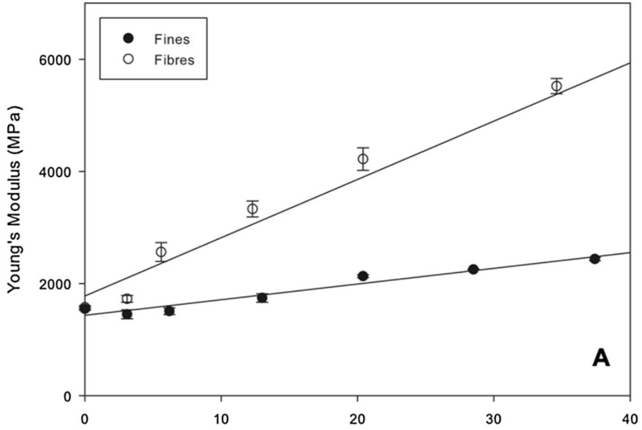
Coroller, Phd Thesis, UBS-LIMATB Lorient, 2013

PROCESSING: MORPHOLOGICAL DAMAGES





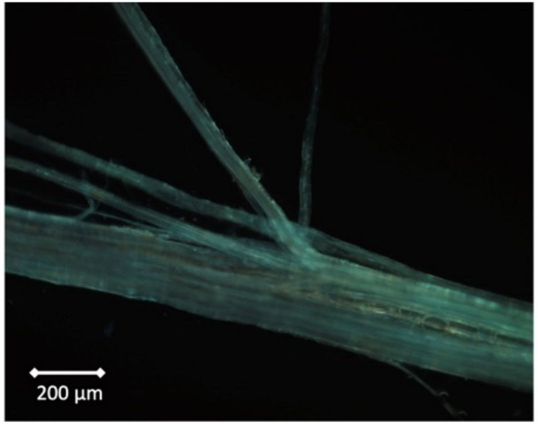


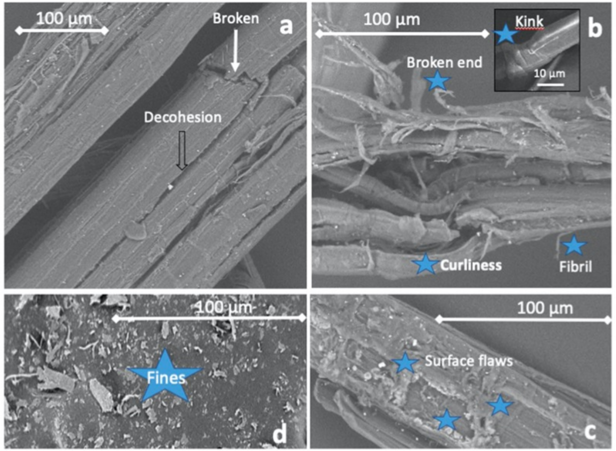


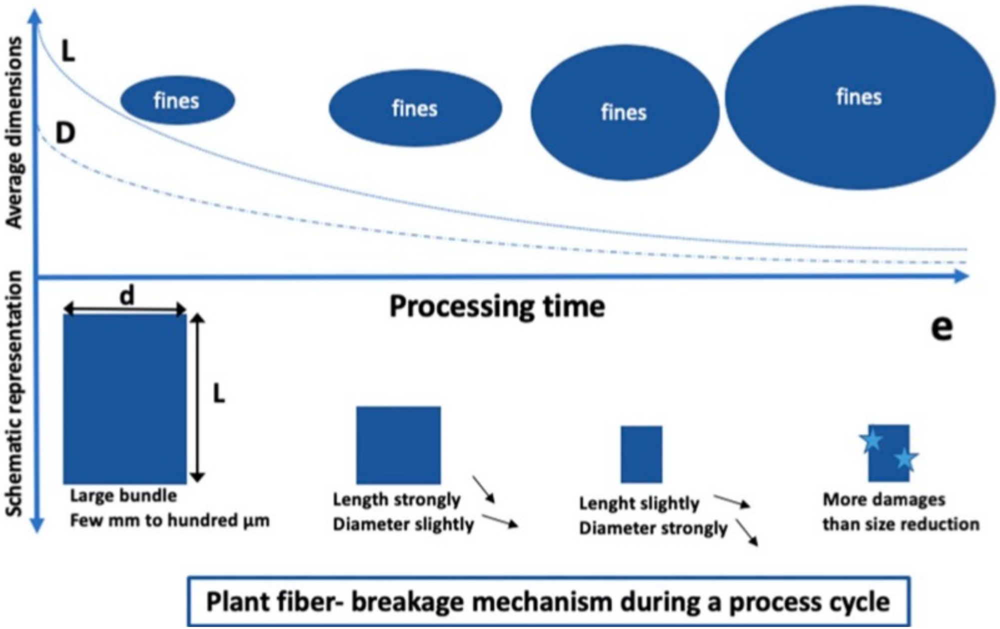
Bourmaud et al., Ind Crops & Prod, 2019

Mayer et al., Powder Tech, 2020

PROCESSING: MORPHOLOGICAL DAMAGES



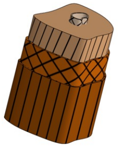




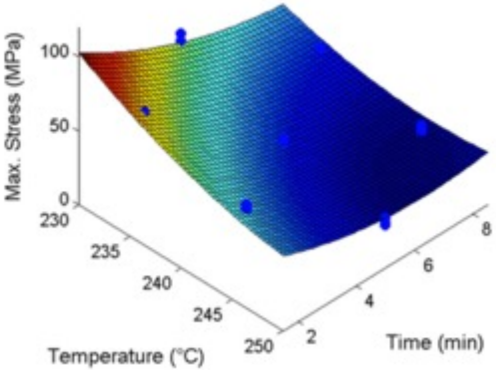
Similar behaviour after several process or recycling stages

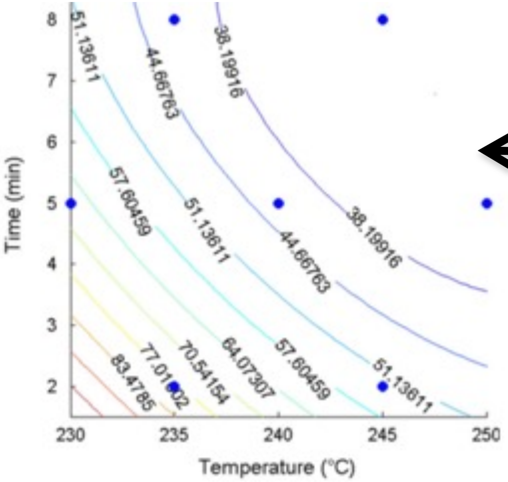
Bourmaud, Shah, Beaugrand and Dhakal., Ind Crops & Prod, 2020

IMPACT AT COMPOSITE SCALE



PA6-Flax



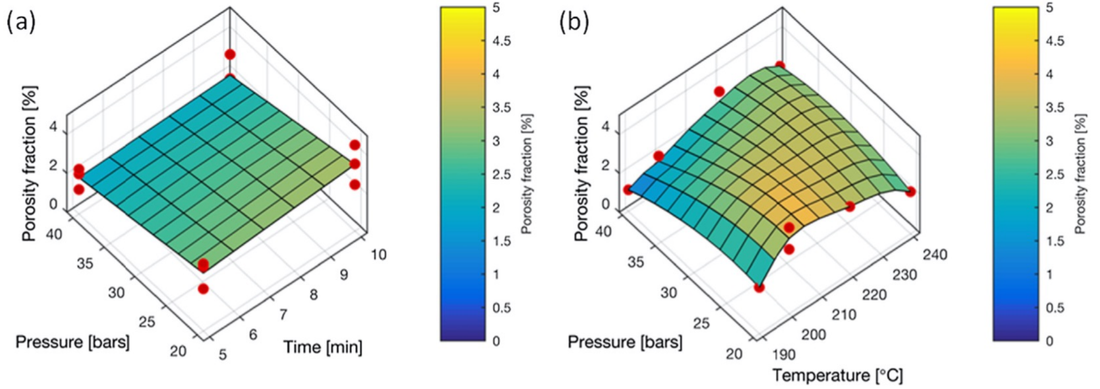


Liang et al.,J Mat Sc, 2015

Time and temperature are impacting on composite properties: exemple of stress (Mpa)

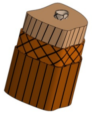
PP-Flax

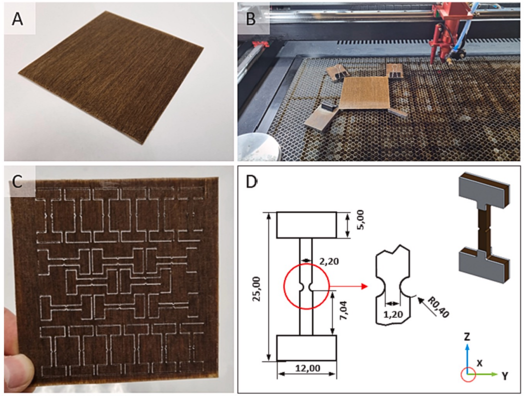
Pressure has also a great impact on porosity fraction

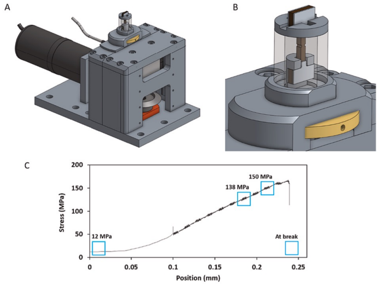


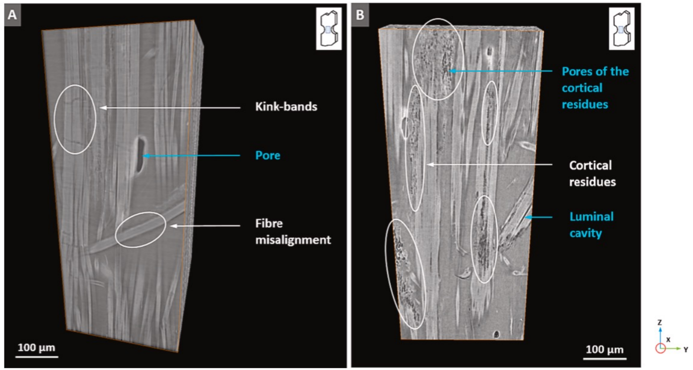
Ramakrishnan, Le Moigne et al., Comp Part A, 2019

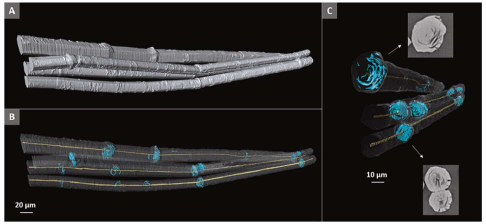
IMPACT AT COMPOSITE SCALE

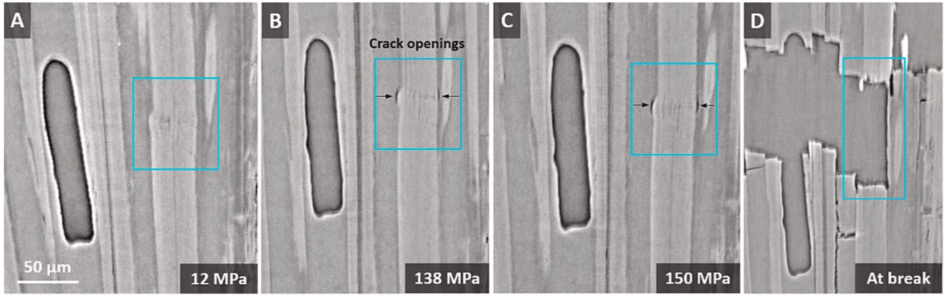


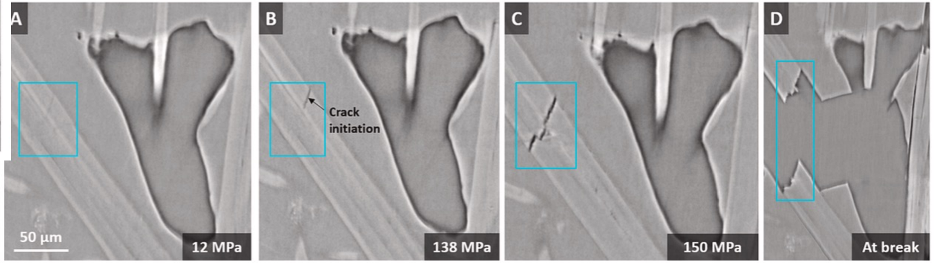






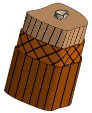




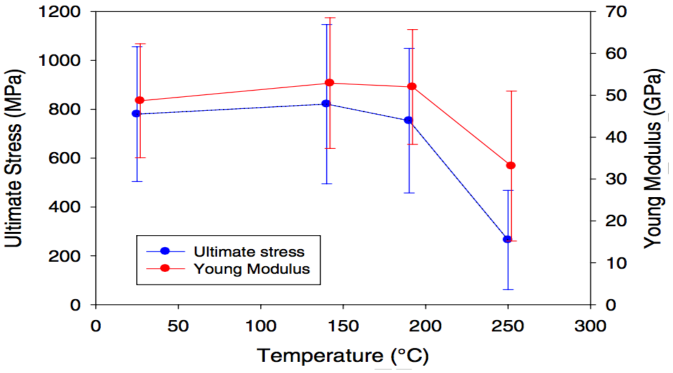


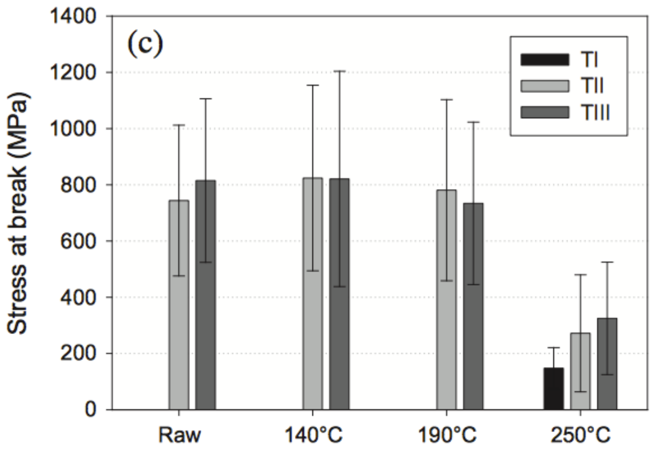
Guillou, Ouagne, Bourmaud et al., Comp Part B, 2024

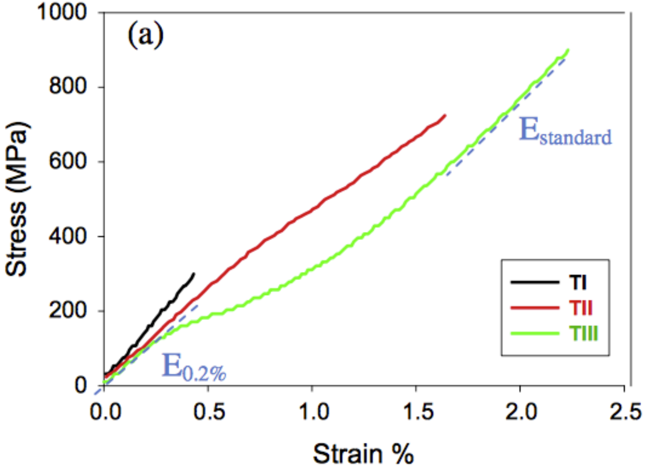
IMPACT AT FIBRE SCALE

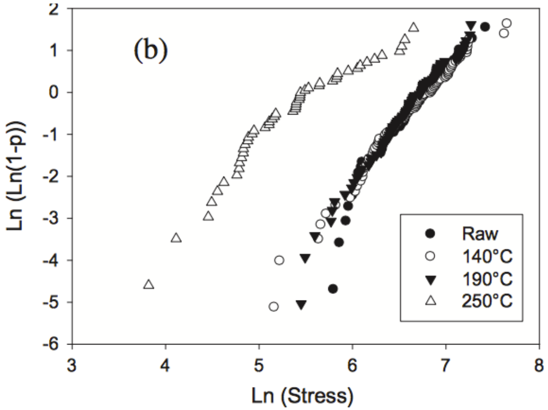


Irreversible damage to the fibre



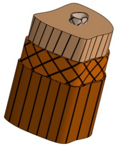


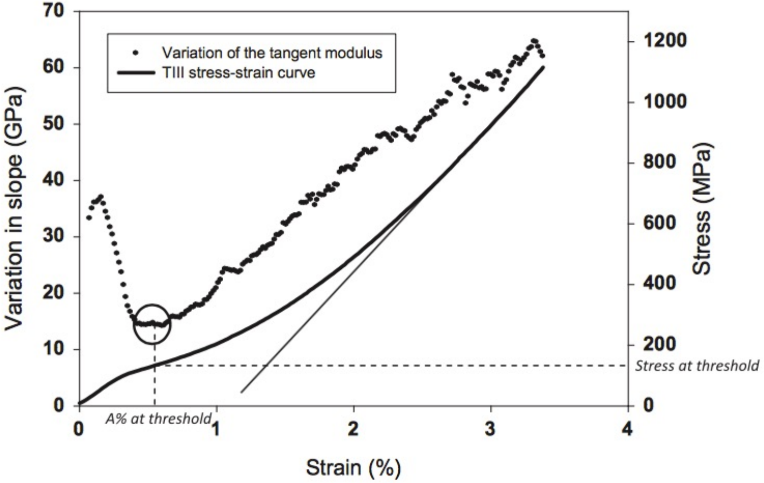


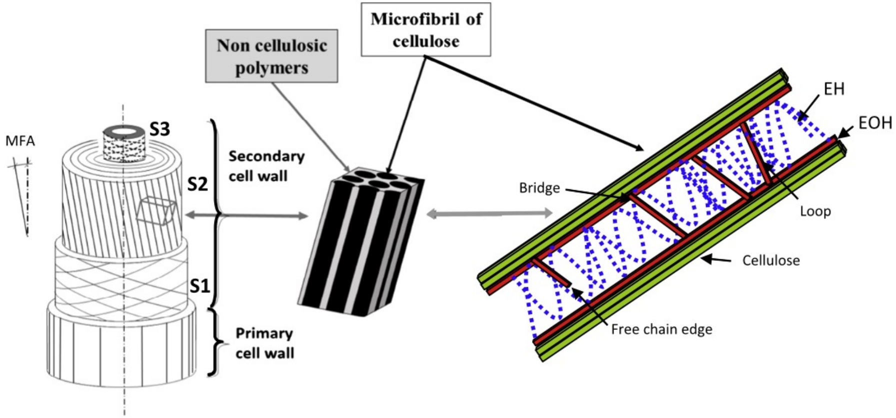


Gourier et al., Comp Part A, 2014

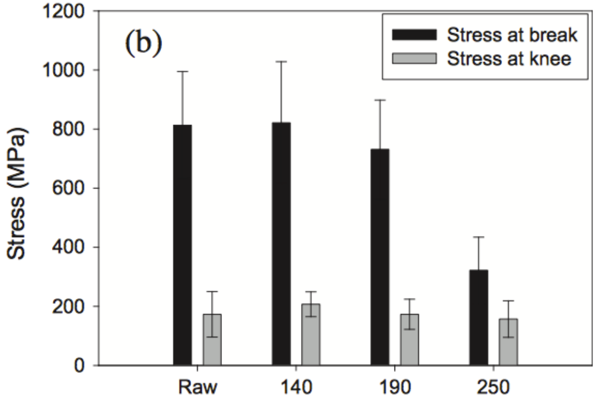
IMPACT AT FIBRE SCALE

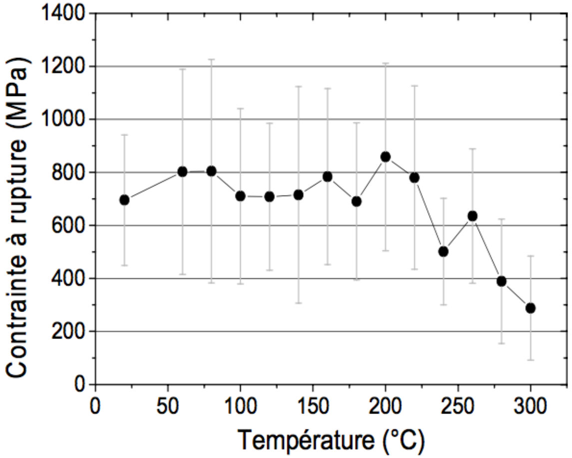






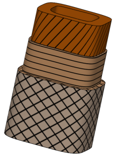
Gourier et al., Comp Part A, 2014

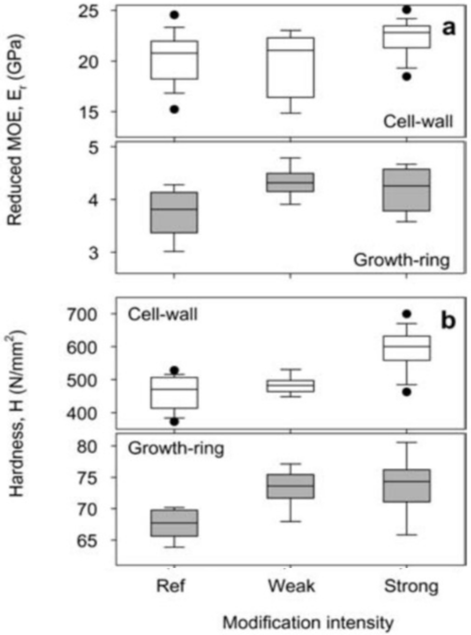




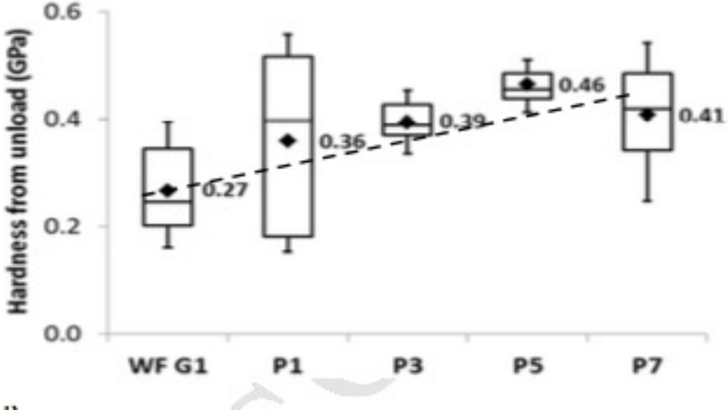
Destaing, PhD Thesis, 2012

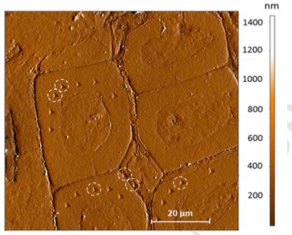
IMPACT AT CELL WALL SCALE - WOOD



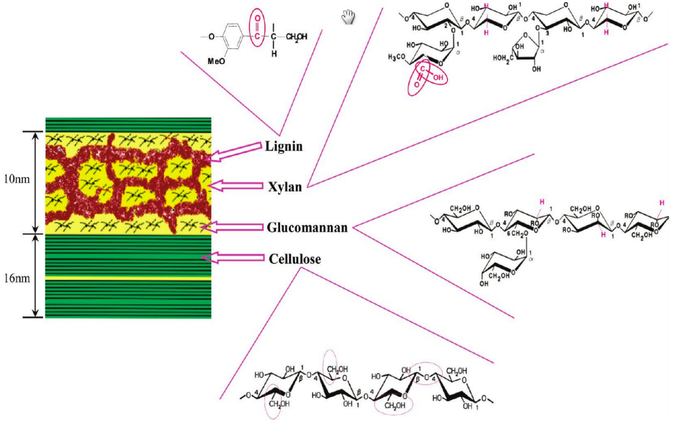


Stanzl-Tschegg et al. Holzforschung, 2009





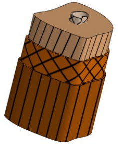
Soccalingame et al. Pol Deg & Stab, 2015



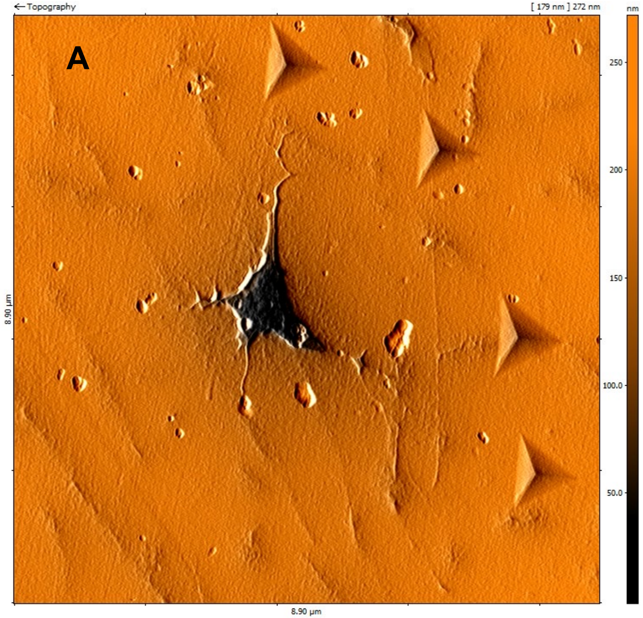
Yin et al. Biomacromolecules, 2011

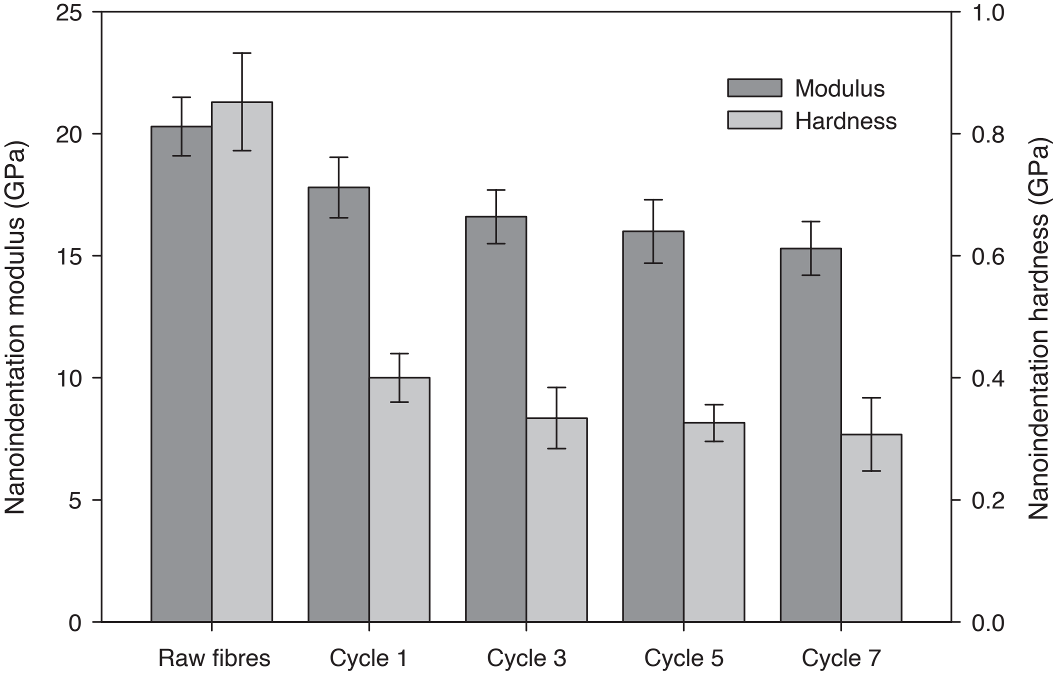
Hypothesis: cross-linking of matrix components (cellulose-xylan-lignin bonds)

IMPACT AT CELL WALL SCALE - FLAX



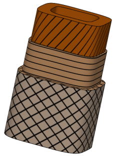
Flax plant walls after several injection cycles

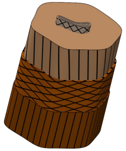




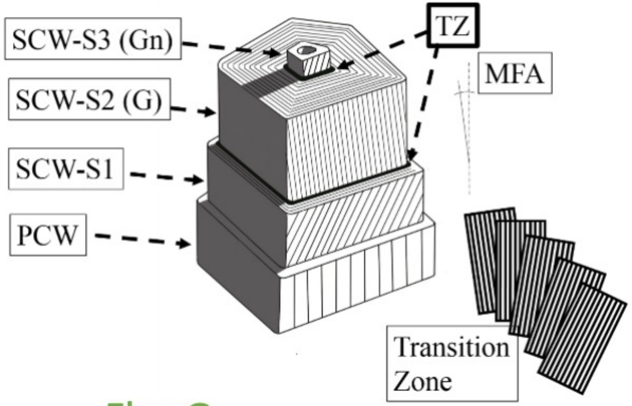
Bourmaud et al., Pol Deg & Stab, 2016

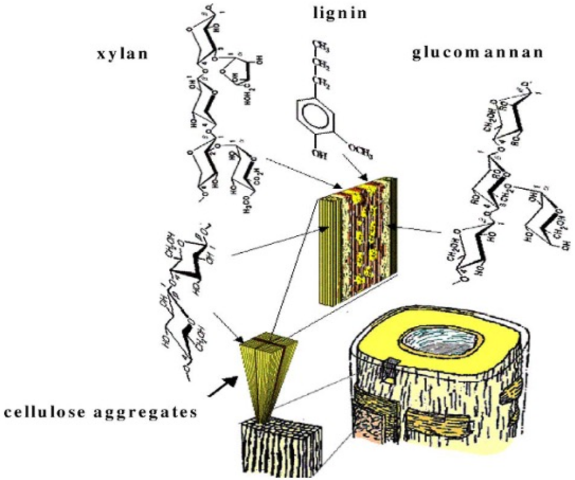
...BUT BIOCHEMICAL STRUCTURE IS DIFFERENT





Wood cell wall layer ultrastructure
[Salmén, *C.R. Biologies*, 2004]





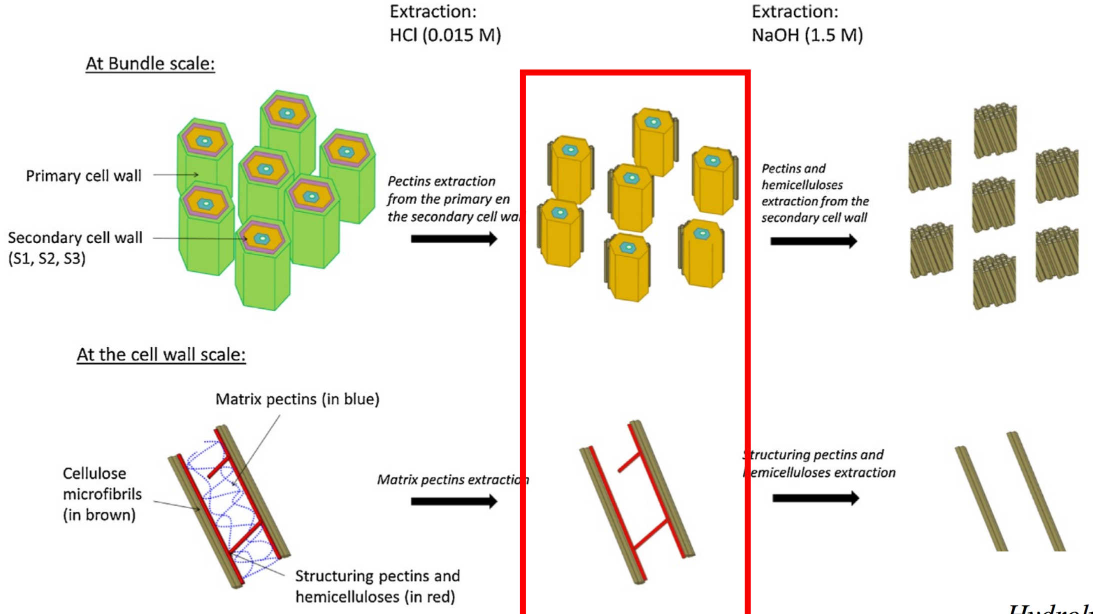
Wood S_2

50% cellulose
25% hemicelluloses - pectines
25% lignin
+ extractives, ashes
MFA = (0) 8-40°

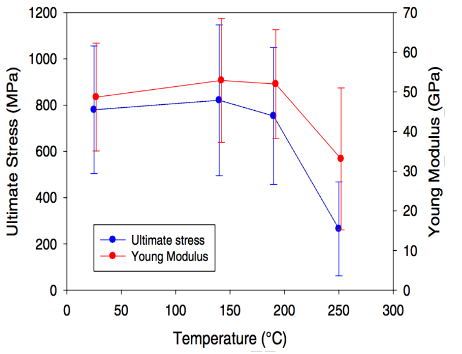
Flax G

80% cellulose
20% hemicelluloses - pectines
≈0% lignin
+ protein
MFA = (0) 5-10°

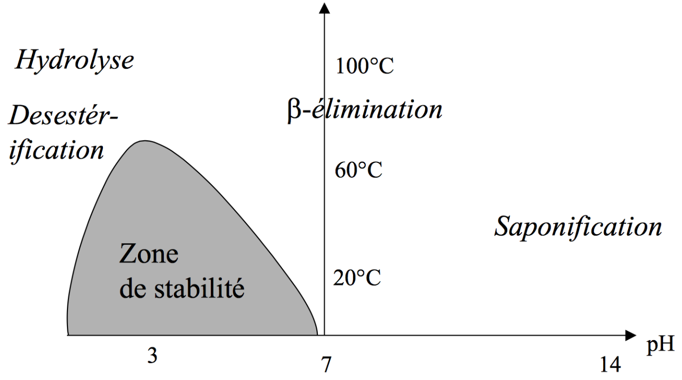
IMPACT OF NON CELLULOSIC POLYMERS ON PERFORMANCES



E=-38%
Sigma=-55%



Gourier et al., Comp Part A, 2014

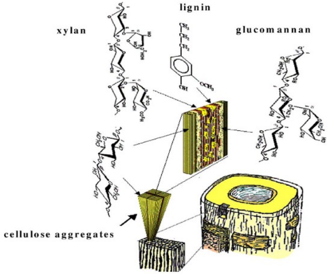


What about the effect of temperature on these polymers?

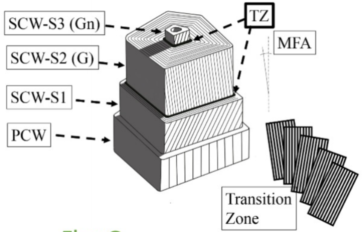
Lefeuvre et al., Ind Crops & Prod, 2015

Renard, seminar INRA, 2010

IMPACT ON COMPOSITE STRUCTURE



Wood cell wall layer ultrastructure
[Salmén, *C.R. Biologies*, 2004]

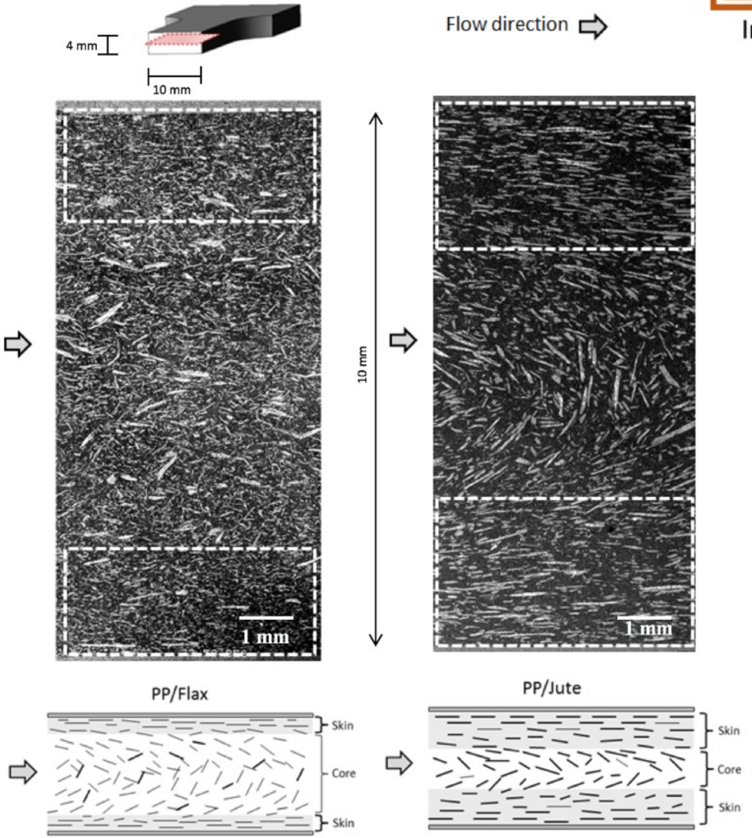


Wood S_2

50% cellulose
25% hemicelluloses - pectines
25% lignin
+ extractives, ashes
MFA = (0) 8-40°

Flax G

80% cellulose
20% hemicelluloses - pectines
≈0% lignin
+ protein
MFA = (0) 5-10°

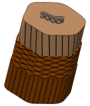


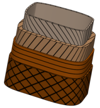
A strong influence of biochemical composition on orientation behaviour

Mikshina et al., INTECH, 2013

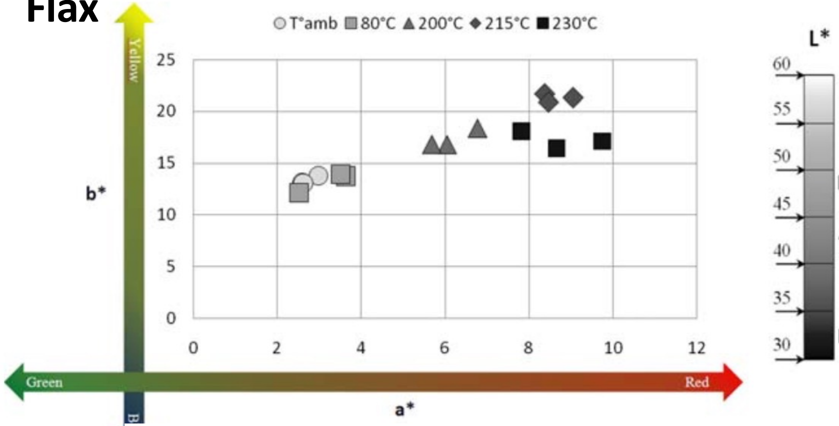
Tanguy, Beaugrand, Gaudry, Bourmaud & Baley, Comp Part B, 2017

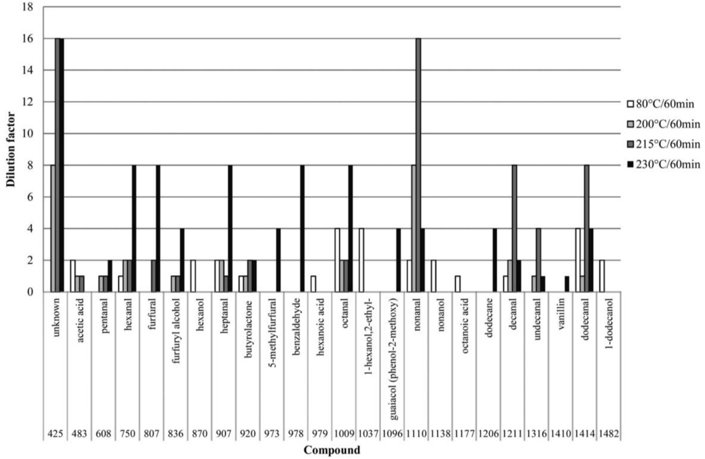
IMPACT ON VOC EMISSIONS



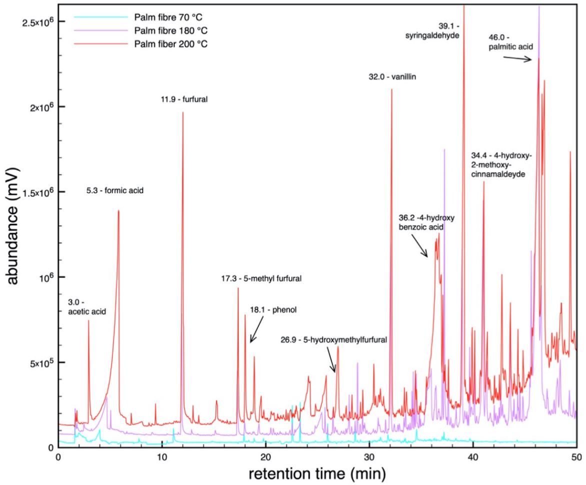


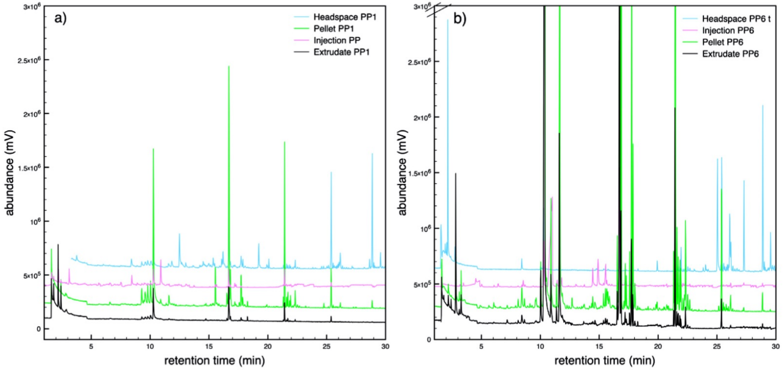
Flax





Palm

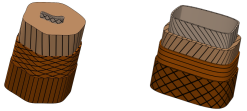




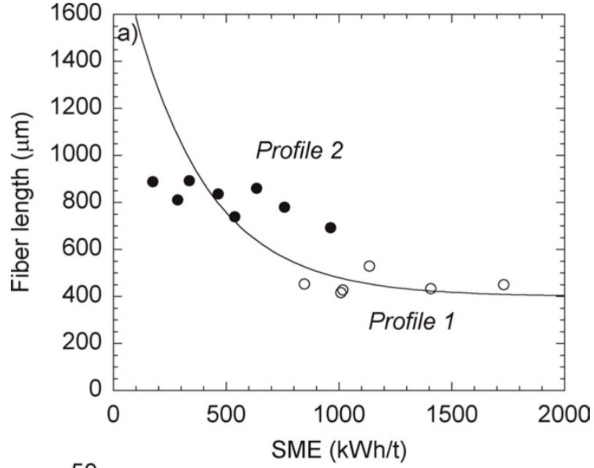
Savary et al., JAPS, 2016

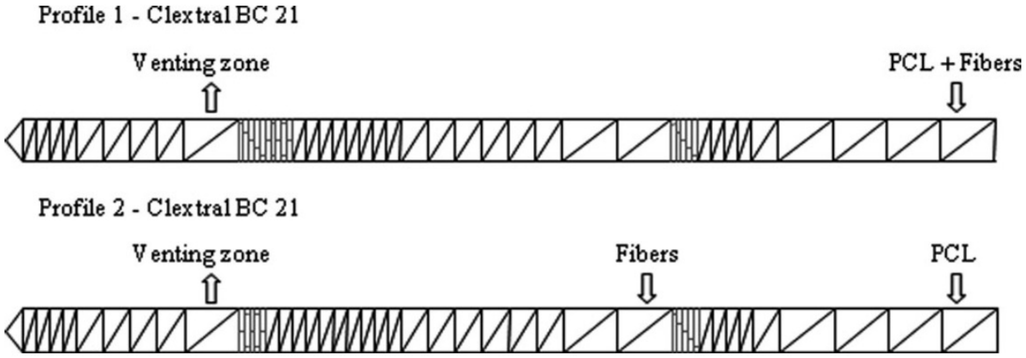
Domenek, Beaugrand et al. PDST, 2021

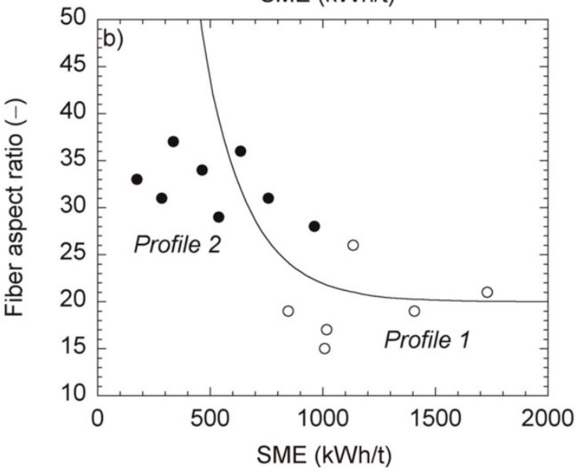
ADJUSTING THE PROCESS

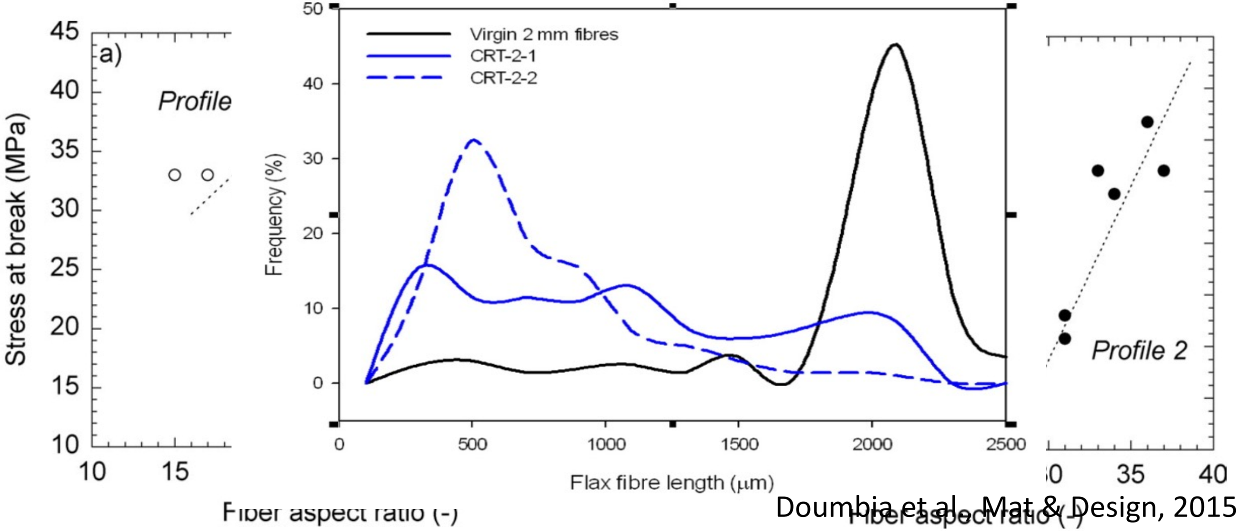


Advantage: preserve fibre lengths





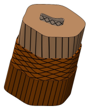




Beaugrand et al., JAPS, 2012

Doumbia et al., Mat & Design, 2015

ADJUSTING THE PROCESS

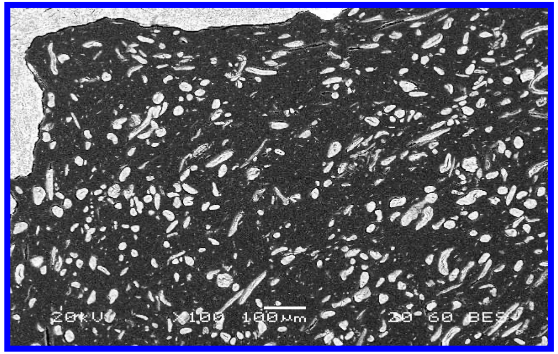


TOWARDS OPTIMAL INDIVIDUALISATION : [BUSS]

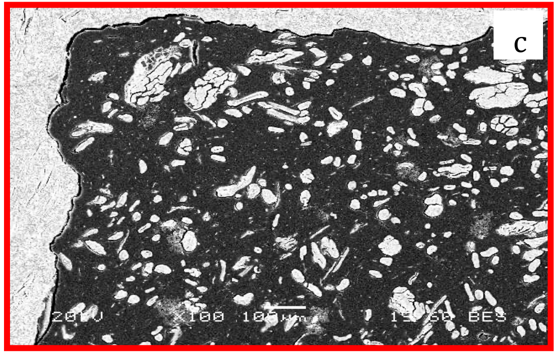
Compounding tool	Diameter (mm)	Dispersion factor R	Composite modulus (Mpa)	Composite strength (Mpa)
Single screw	28.6	0.94	3702±121	33.6±0.1
Twin screw	15.1	1.21	4405±133	37.4±0.1
Buss Hard	**18.2**	**1.26**	**4419±157**	**38.5±0.5**
Buss Soft	24.9	1.01	3924±196	35.6±0.3

Coroller, PhD Thesis, 2012

BUSS



TWIN-SCREW



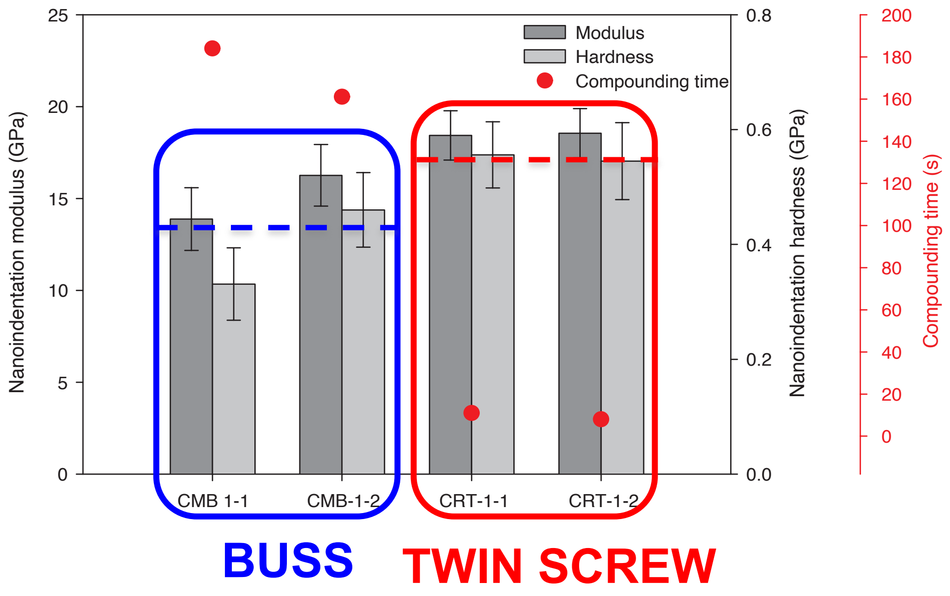
Higher composite stress for BUSS, similar stiffness

Doumbia et al., Mat & Design, 2015

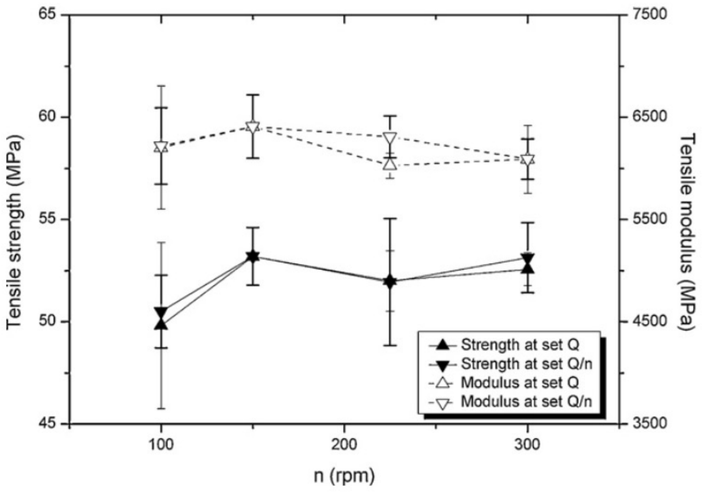
ADJUSTING THE PROCESS

Injection

SHOULD RESIDENCE TIMES OR TEMPERATURE BE LIMITED?



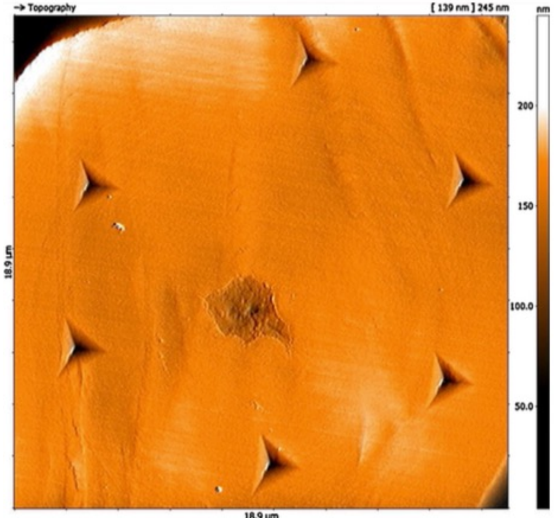
Doumbia et al., Mat & Design, 2015

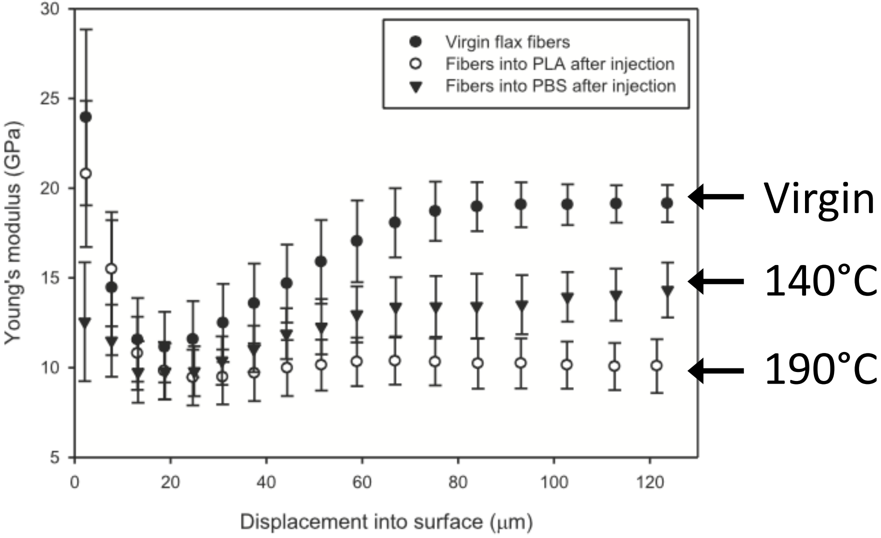


Gamon et al. Ind Crop & Prod, 2013

Injection

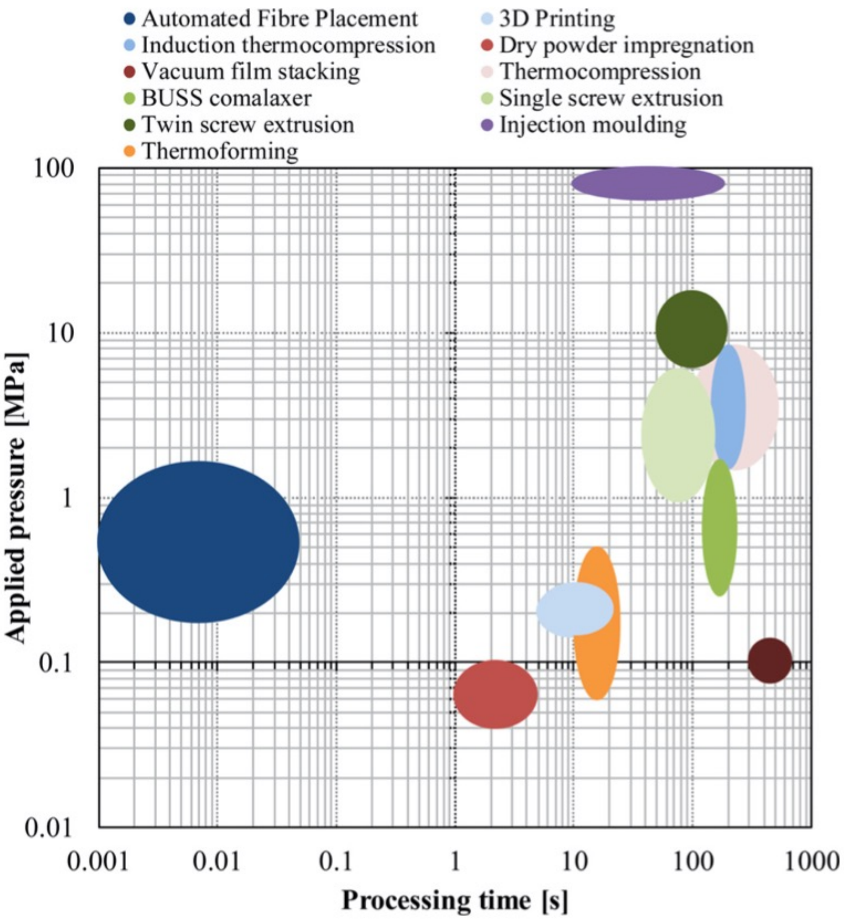
SHOULD RESIDENCE TIMES OR TEMPERATURE BE LIMITED?





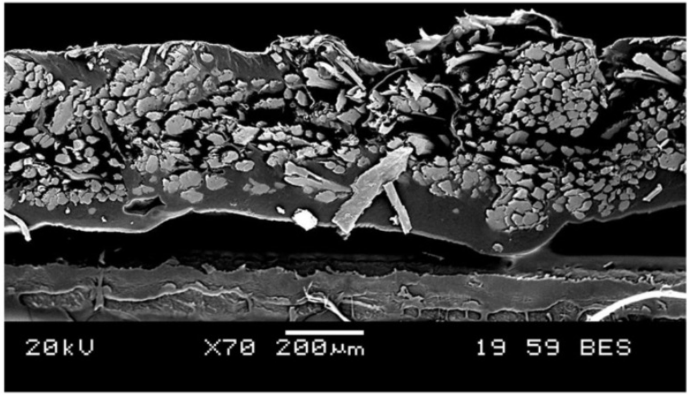
Bourmaud et al. Ind Crop & Prod, 2014

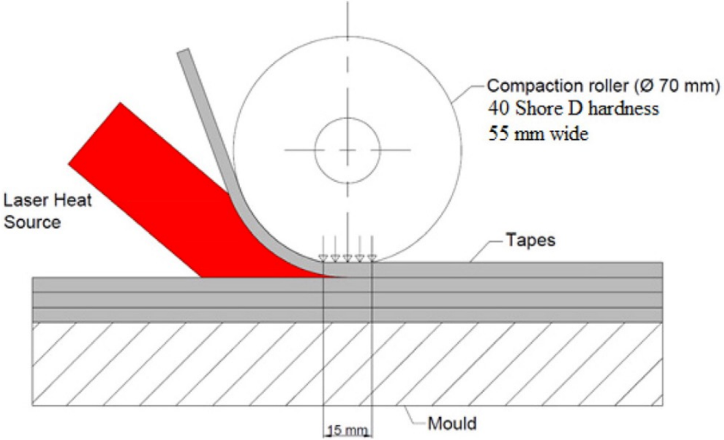
ADJUSTING THE PROCESS

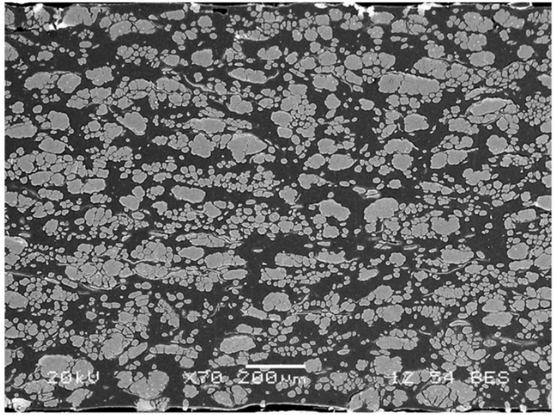


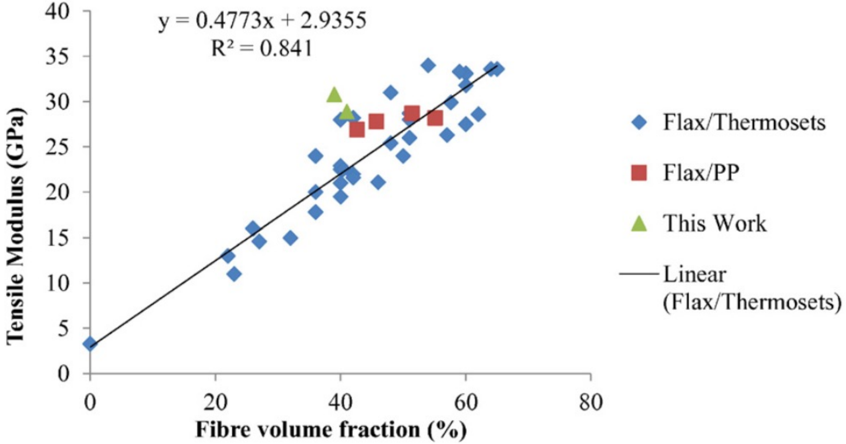
Bourmaud, Shah, Beaugrand & Dhakal. Ind Crop & Prod, 2020

ADJUSTING THE PROCESS – EXAMPLE OF AFP





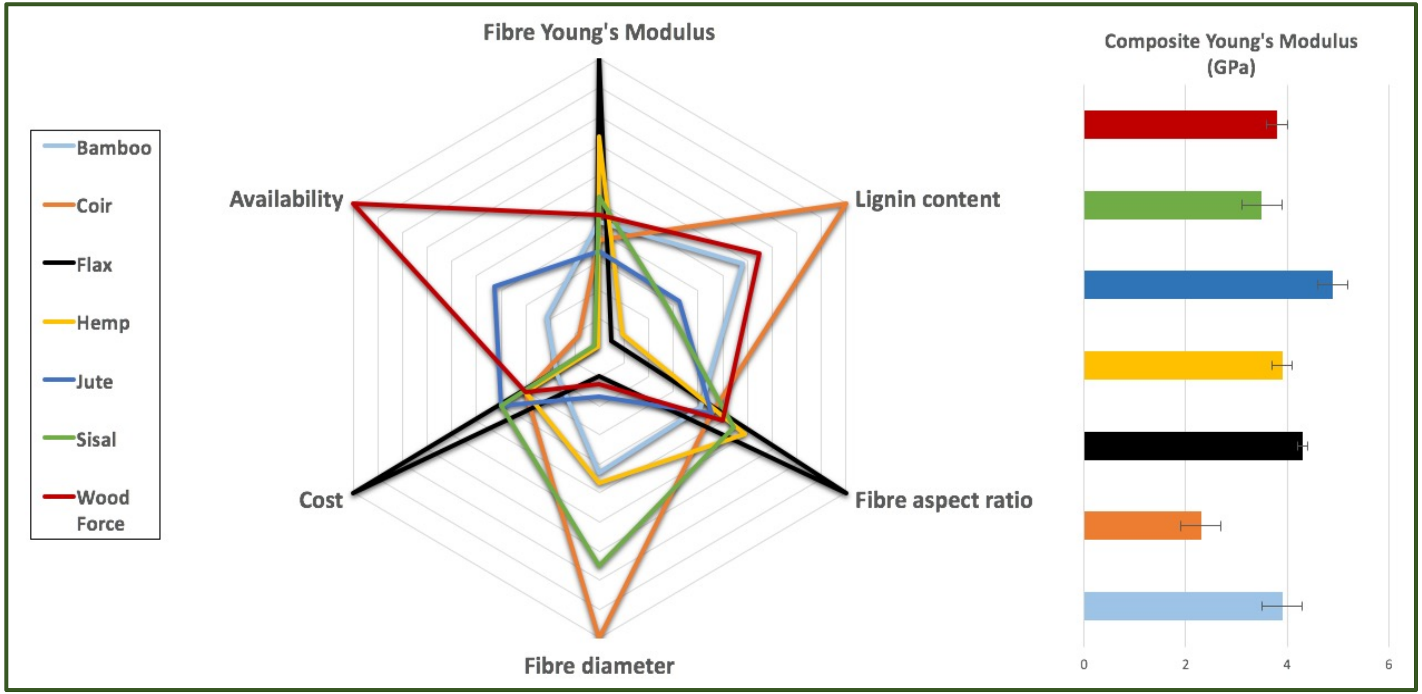




Baley, Bourmaud, Lan et al., Mat & Des, 2016

OR AN APPROPRIATE CHOICE OF PLANT FIBRES ?

Assesment / Injection



- Same matrix (PP)
- Same compounding way
- Same testing

It is necessary to take other parameters than fibre modulus into consideration

Bourmaud, Beaugrand, Shah, Placet, Baley, PMS, 2018