CERISE : CEllulose pRetreatment for In Situ fibrillation by twin screw Extrusion

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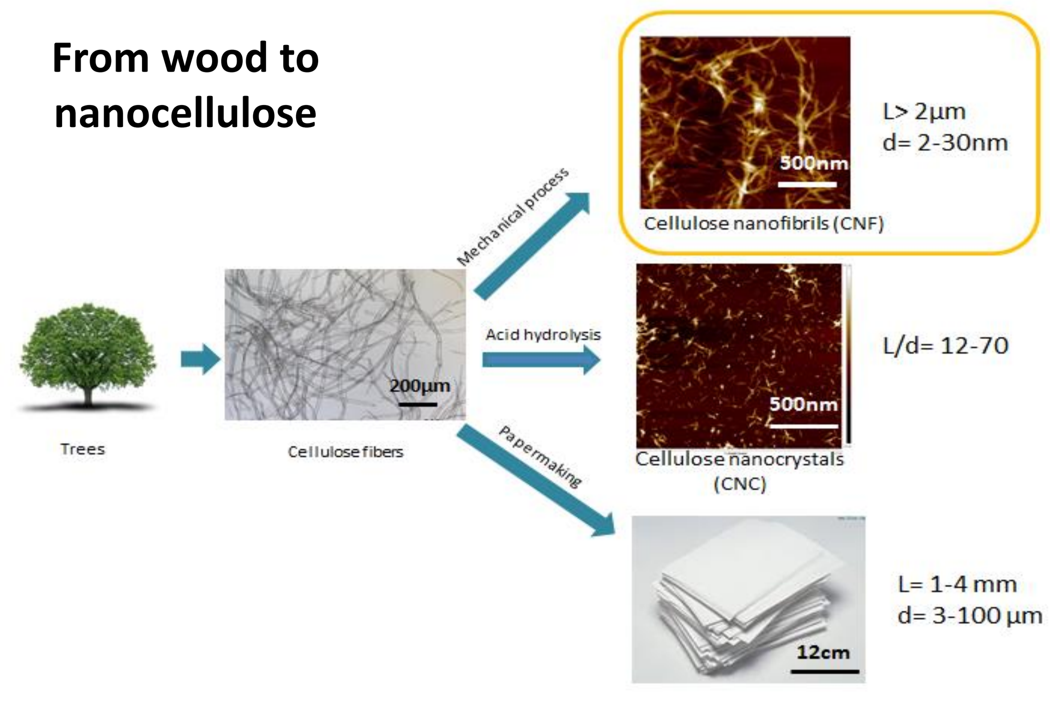
Context

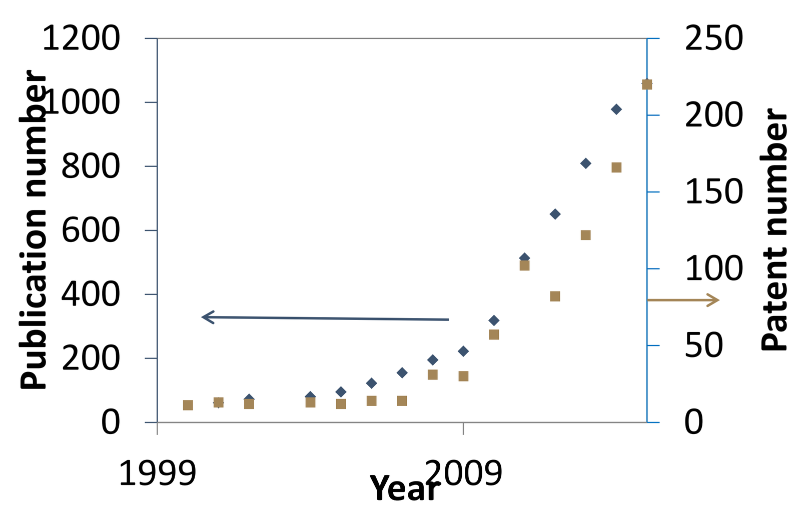
From wood to nanocellulose



Inspired from Emily Cranston, McMaster University

Nanofibrillated cellulose CNF

- Bio-based material
- Renewable & Biodegradable & Biocompatible
- Good mechanical properties
- Barrier properties
- High specific area
- Transparent



- ✓ New fonctionalities
- ✓ Energy decrease

New chemical pretreatment

CERISE Project

- ✓ New chemical pretreatment
- ✓ TSE – common industrial method
- ✓ Energy efficient
- ✓ Highly adaptable procedure
- ✓ Fast procedure
- ✓ High solid content

Novel route for CNF production: Twin Screw Extruder

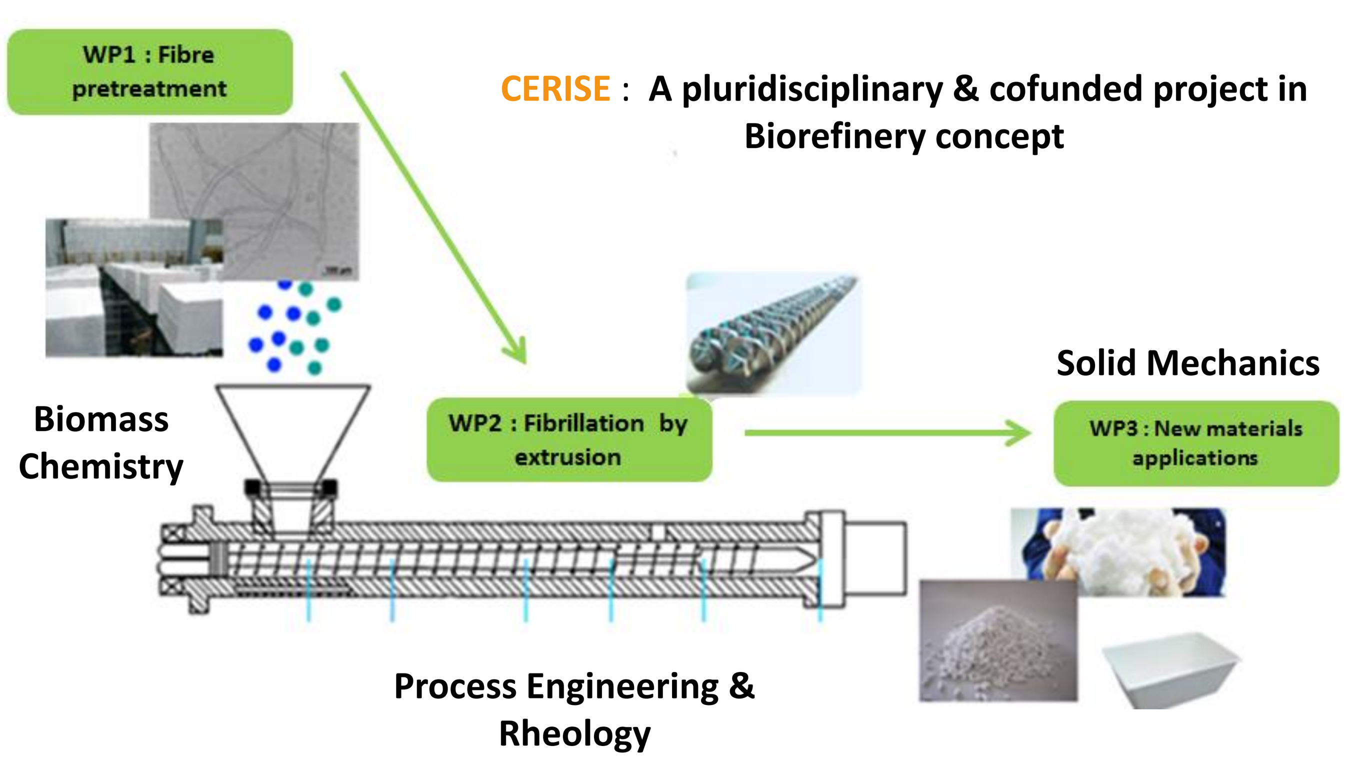
1982 – Discovery; 2008; 2011 – CNF Industrialization; 2014

- Main issues for the industries:
  - ✓ High Costs & Energy consumption
  - ✓ Production at 2 wt% suspension form
  - ✓ Storage ,Transport & Application issues

CERISE Project

- Developing **new « green » pretreatments** to facilitate the liberation of cellulose microfibrils
- Using twin-screw extrusion for a microfibrillation process at high solid content in an energy effective and continuous system
- Preparing new materials made of 100% of CNF or with high concentration formulation

CERISE : **A pluridisciplinary & cofunded project in Biorefinery concept**



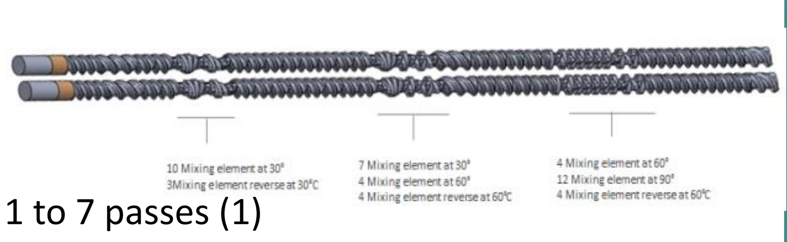
Materials & methods

Materials

- Eucalyptus bleached kraft pulp Fibria
- Enzymatic pretreatment of cellulose fiber

FiberCare R, 50°C, 60L/t, 2h, pH 5

Methods



Comparison of two processes

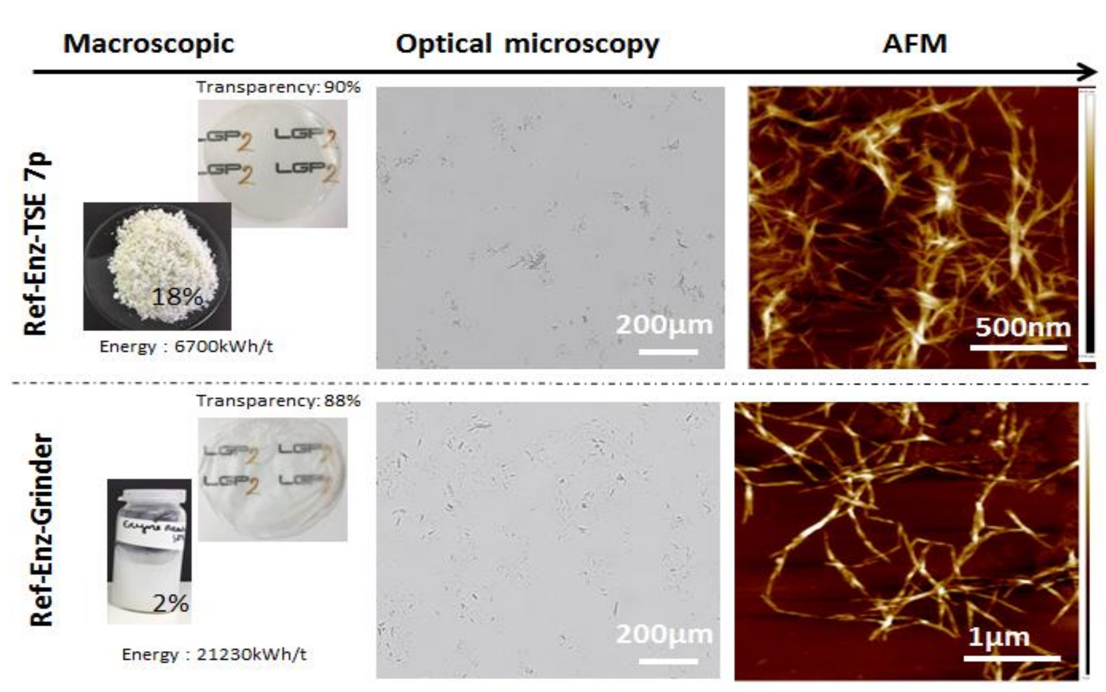
- **Extrusion** : 400rpm, 10°C, from 1 to 7 passes (1)
- Supermasscolloider **grinder** equipped with recirculation, 2h30

Characterizations

Characterizations	Methods
AFM	10^{-2}%, mica disk, Scan assist mode
Optical microscopy	0,5%, Carl Zeiss Axio Imager M1 optical microscope
Mechanical properties	Instron, 5kN, 50mm/min, 15mm*50mm
Transparency	Haze meter, NF: T 54-111,1971
DP	ISO 5351:2010
Energy	Torque*velocity/flow

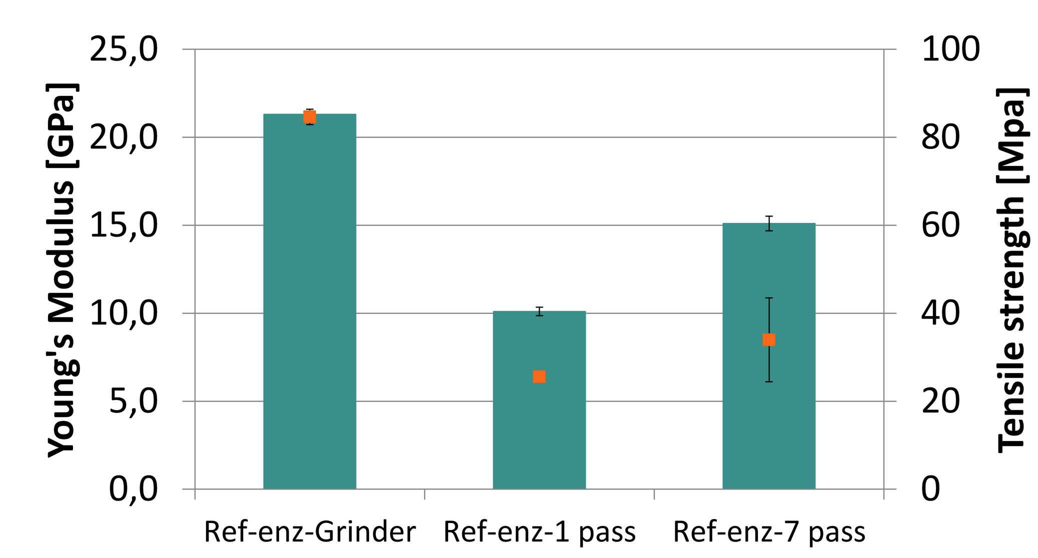
First Results

Nanofiber morphology



- Twin screw extruder leads to nanofiber with the same morphological properties
- After 7 passes through the TSE, cellulose nanofibers are transparent

Mechanical properties



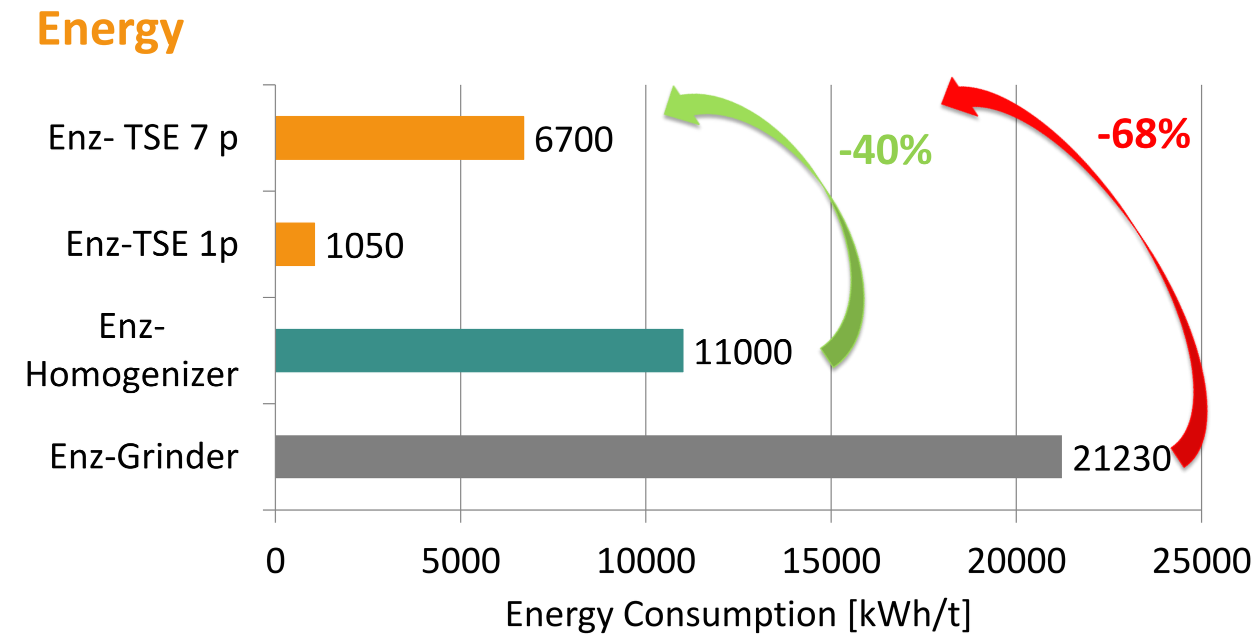
- Mechanical properties are very closed
- Young's modulus are in accordance with the literature

Nanofiber properties

Reference	Refined [Y/N]	Chemical pretreatment	Mechanical pretreatment	Number of pass	Solid content [%]	Young's modulus [GPa]	Tensile Strength [MPa]	DP	Transparency [%]
Commercial	Y	Enzyme	Homogenizer	5	**2**	11.9 +/-3.9	67.9 +/-3.5	194 +/- 2	**90.6** +/-0.6
Ref-Enz-TSE1p	Y	Enzyme	Extruder	1	**18.2**	10.1 +/-0.2	25.6 +/-1.1	323+/- 2	**89.4** +/-0.3
Ref-Enz-TSE 7p	Y	Enzyme	Extruder	7	**18.7**	15.1 +/-0.4	33.9 +/-9.5	219+/- 1	**89.6** +/-0.1
Ref-Enz-Grinder	Y	Enzyme	Grinder	2h30	**2**	21.3 +/-0.1	84.6 +/-1.8	215 +/- 1	**88.0** +/-0.7

➡ Similar properties whatever the process
But higher solid content

Energy



Mechanical method	Total energy consumption for the production [kWh/t]	Reference
Homogeneizer	70 000	(Eriksen 2008)
Homogeneizer Gaulin (20 passes, 55MPa)	22 000	(Spence et al. 2011)
Homogeneizer	12 000 -25 000	(Klemm et al. 2011)
Microfluidizer (20 pass, 69MPa)	3200	(Spence et al. 2011)
Grinder (1 to 10 hours, 1500 rpm)	5000-30 000 kWh/t	(Wang et al. 2012)

- Twin screw extruder allows to reduce considerably the energy consumed compare to other processes

Conclusion & Perspectives

Nanofibrillation of pulp with same quality as other mechanical treatments but...

- 5 to 10 times less water
- Strong energy savings (40 to 68%)
- Production time is reduced by 2 to 4
- Production costs are reduced
- Transport cost are reduced

- Try different pretreatments : TEMPO oxidation, cationic,…
- Optimise extrusion to obtain MFC after 1 pass
- Understand what happens during extrusion
- Develop in situ extrusion
- Try combination of different processes

(1) Ho, T. T.; Abe, K.; Zimmermann, T.; Yano, H. Nanofibrillation of pulp fibers by twin-screw extrusion. *Cellulose* 2014, *22* (1), 421–433

Acknowledgments

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