Optimization Of the Twin-Screw Extrusion (TSE) Process Stability for Cellulose Nanofibril (CNF) Production

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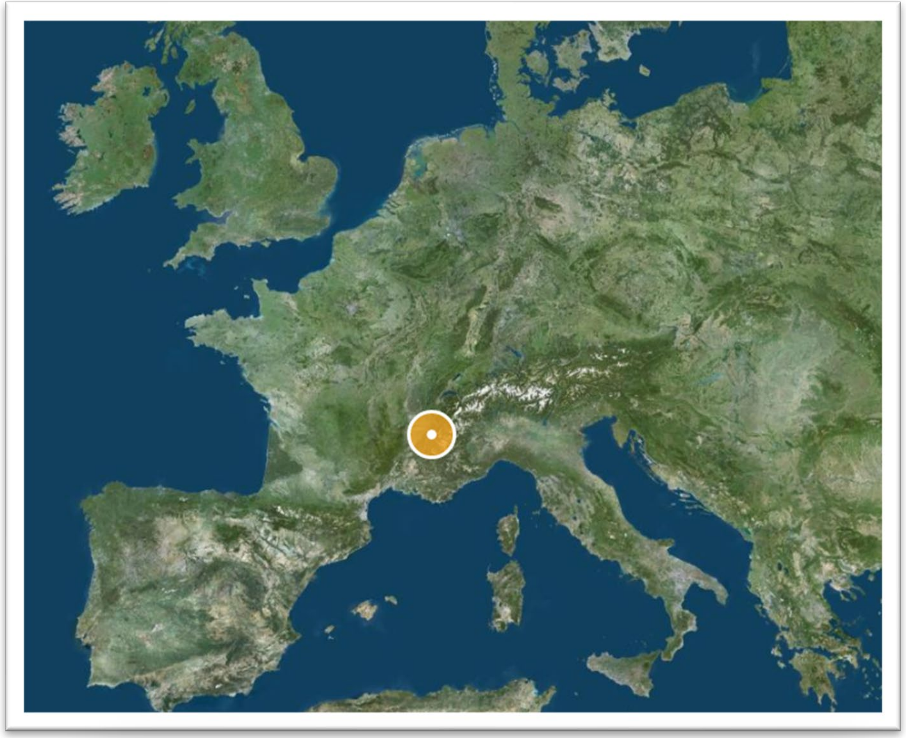
2. Centre Technique du Papier (CTP), F38000 Grenoble, France

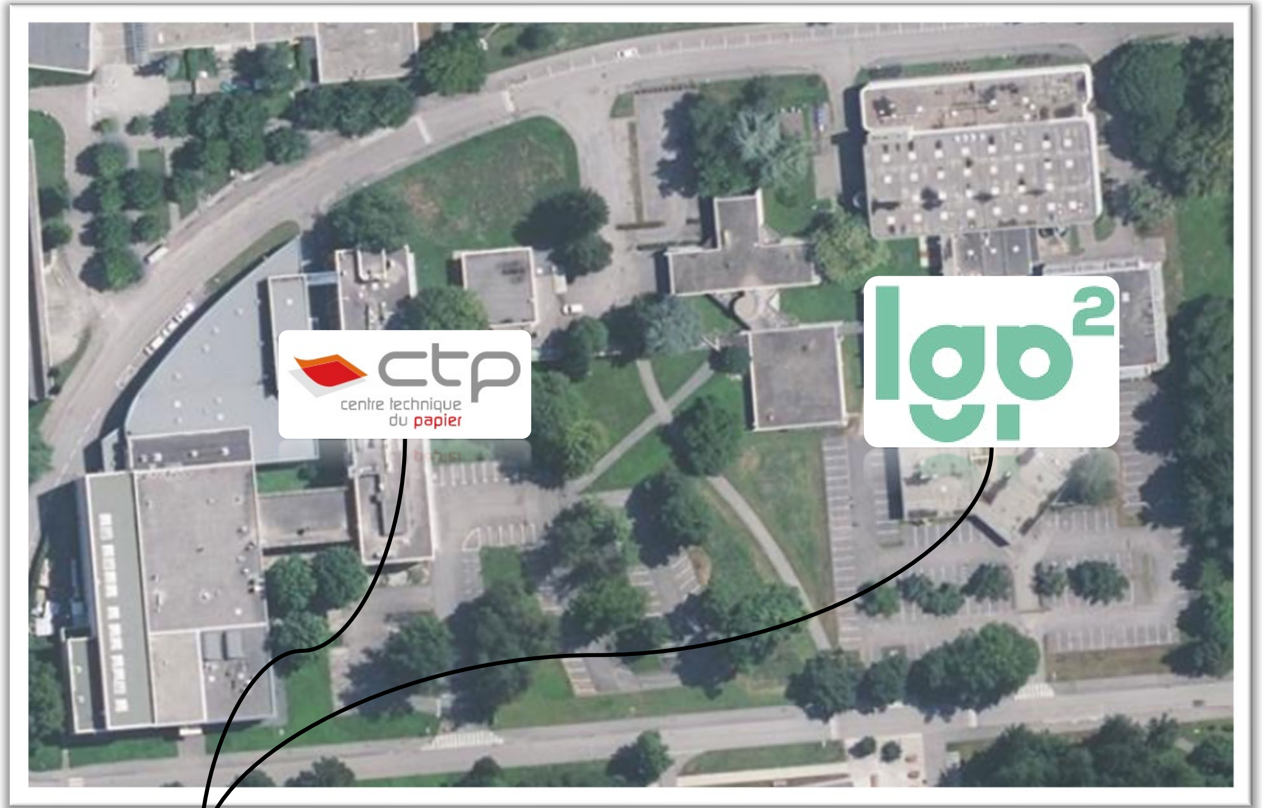
TAPPI NANO DIVISION

Grenoble localization

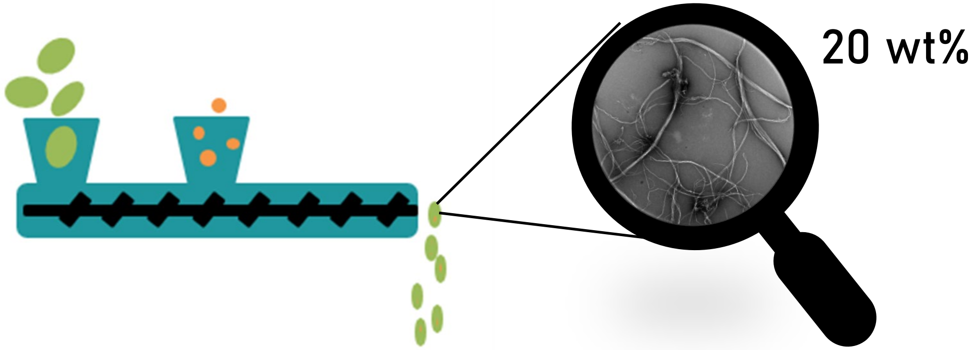
ctp
centre technique
du papier

lgp²



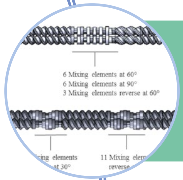


TWILIGHT

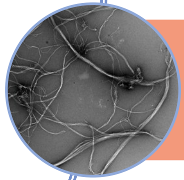


Agenda

Introduction & literature



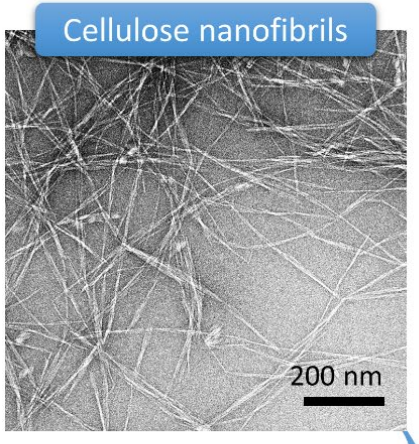
Stability of TSE process

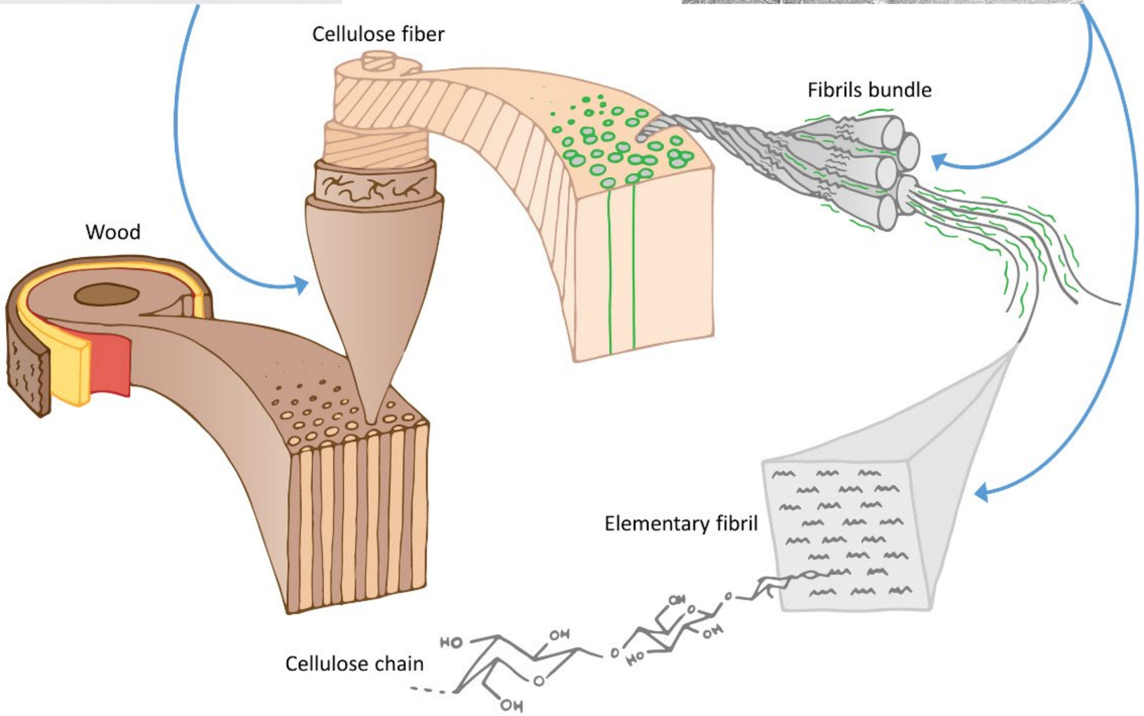


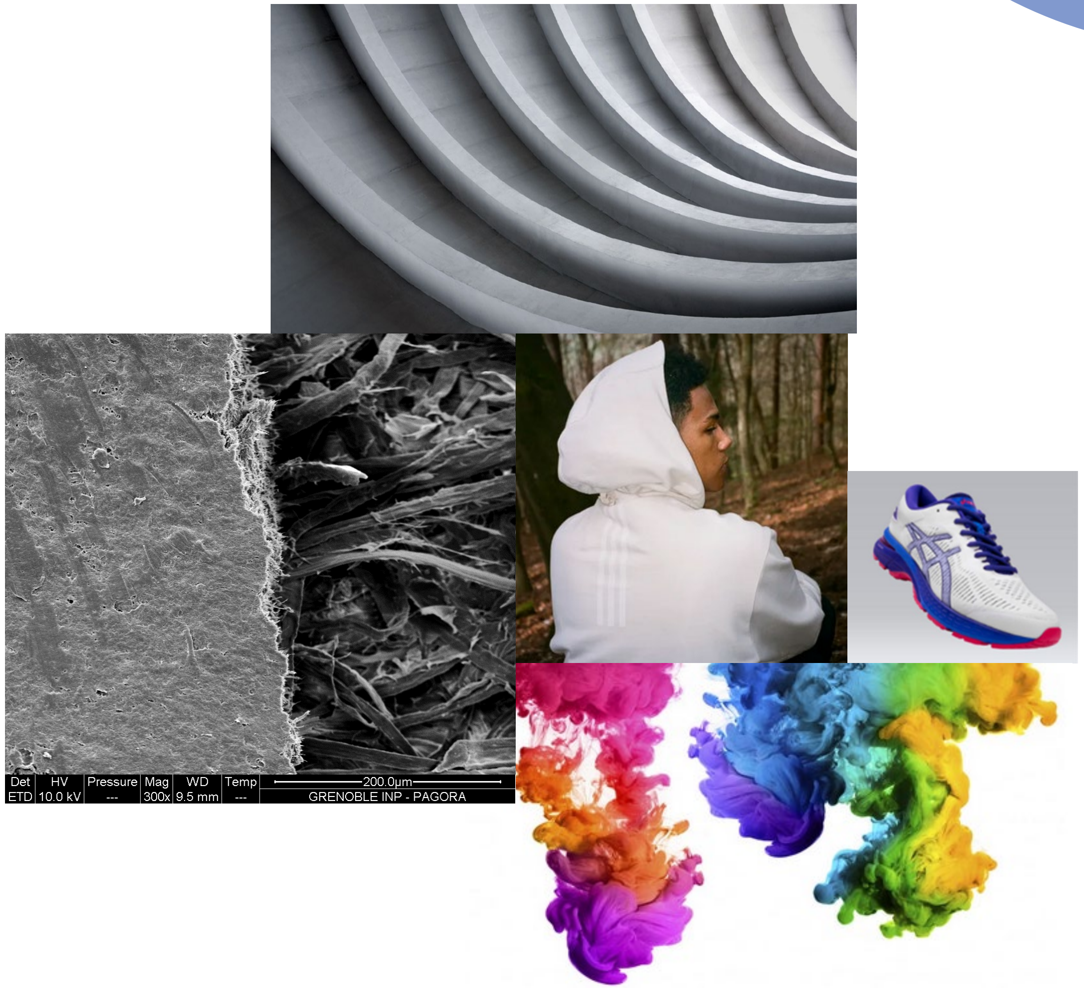
Extruded CNF quality and characterization

Conclusion

Cellulose Nanofibril (CNF)



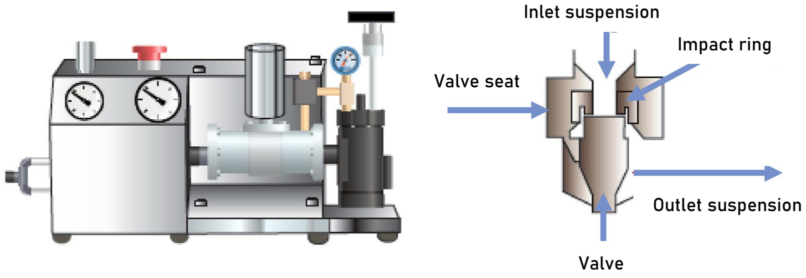




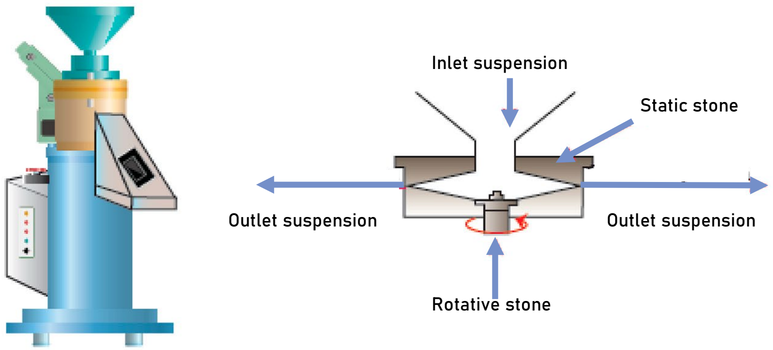
Gabriel Banvillet, PhD thesis, (2020)

Classical ways to produce MFC

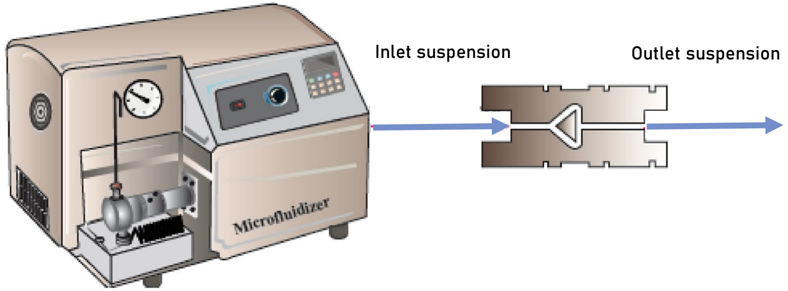
High pressure homogenizer [1, 4]



Grinding [1,4]



Microfluidizer [1, 4]

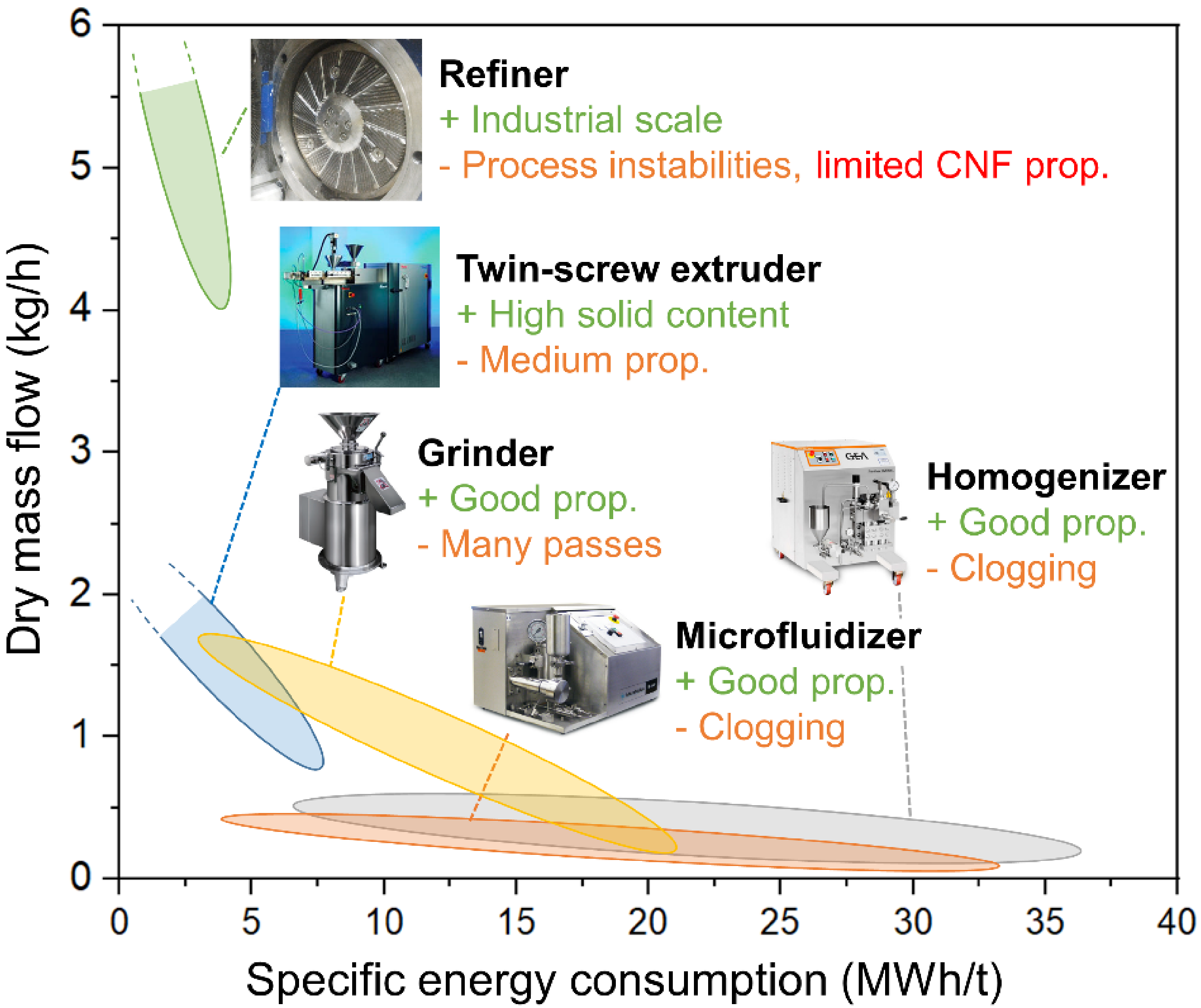


[2, 3, 4]

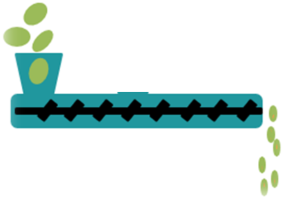
Low solid content : 0,5-4 wt% → High transportation & drying costs
High energy consumption : 8-70 MWh/t

1. Rashki et al. Cellulose nanofibrils manufactured by various methods with application as paper strength additives, *Frontiers in Bioengineering and Biotechnology* (2021).
2. Turbak *et al.*, Microfibrillated cellulose, a new cellulose product: properties, uses, and commercial potential, *J. Appl. Polym. Sci.: Appl. Polym. Symp.* **37** (1983).
3. Herrick *et al.*, Microfibrillated cellulose: morphology and accessibility, *J. Appl. Polym. Sci.: Appl. Polym. Symp.* **37** (1983).
4. Nechyporchuk *et al.*, Current Progress in Rheology of Cellulose Nanofibril Suspensions, *Industrial Crops and Products* **93**, 2–25 (2016).

Classical ways to produce MFC



NFC production by twin screw extrusion



"Process for the production of microfibrillated cellulose in an extruder and microfibrillated cellulose produced according to the process"[1]

2011

2014

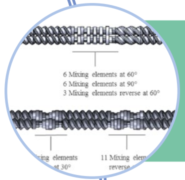
2016
2019

2017
2020

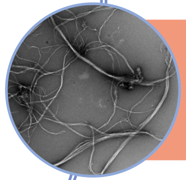
1. Stora Enso, *WO2011051882A1*
2. Ho *et al.,Cellulose* (2014)
3. Fleur ROL, PhD thesis (2019)
4. Banvillet *et al., BioResource Technology,* (2021)
5. Banvillet *et al., Cellulose*, (2023)

Agenda

Introduction & literature



Stability of TSE process



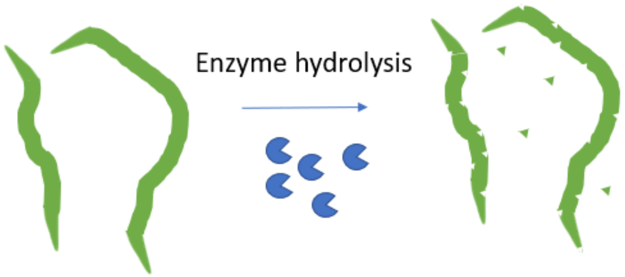
Extruded CNF quality and characterization

Conclusion

Material and methods

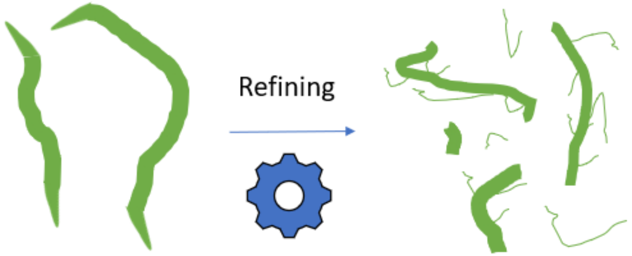
Birch fibers

1) Enz



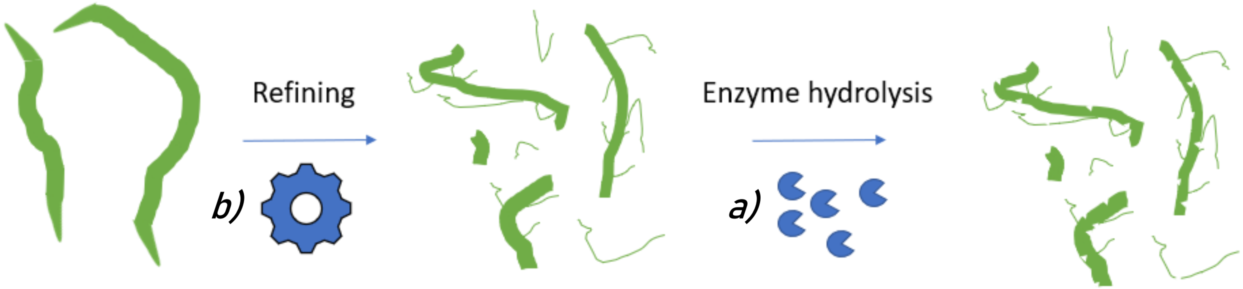
a) FiberCare R 300 ECU/g
50°C, pH = 5, 2h

2) Mech

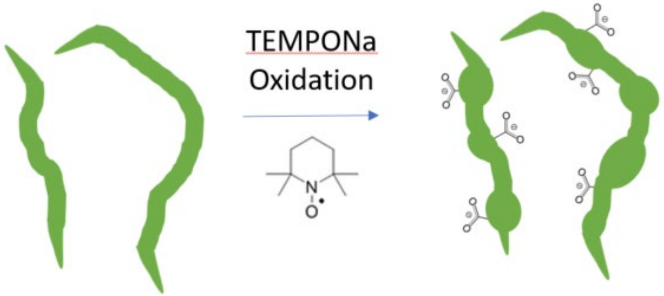


b) Valley Beater, 80°SR

3) Mech + Enz

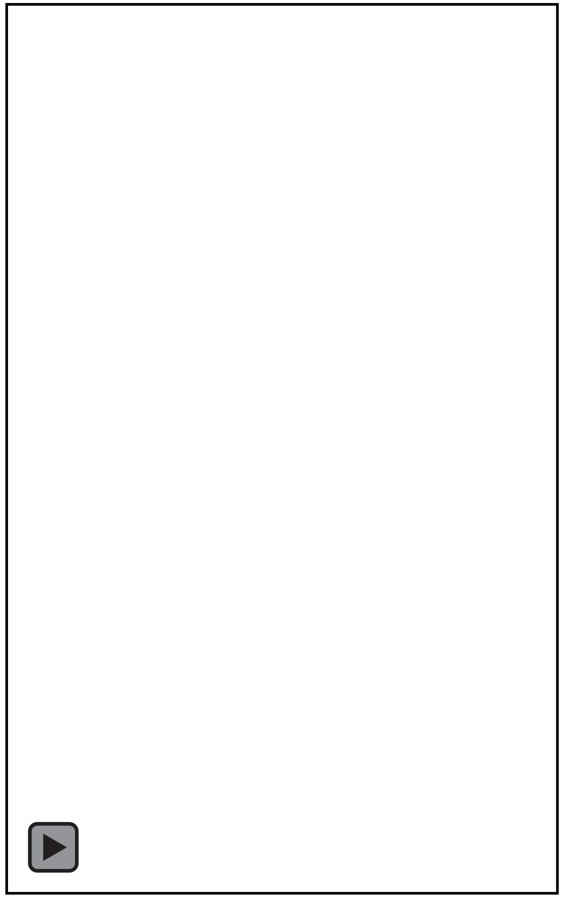


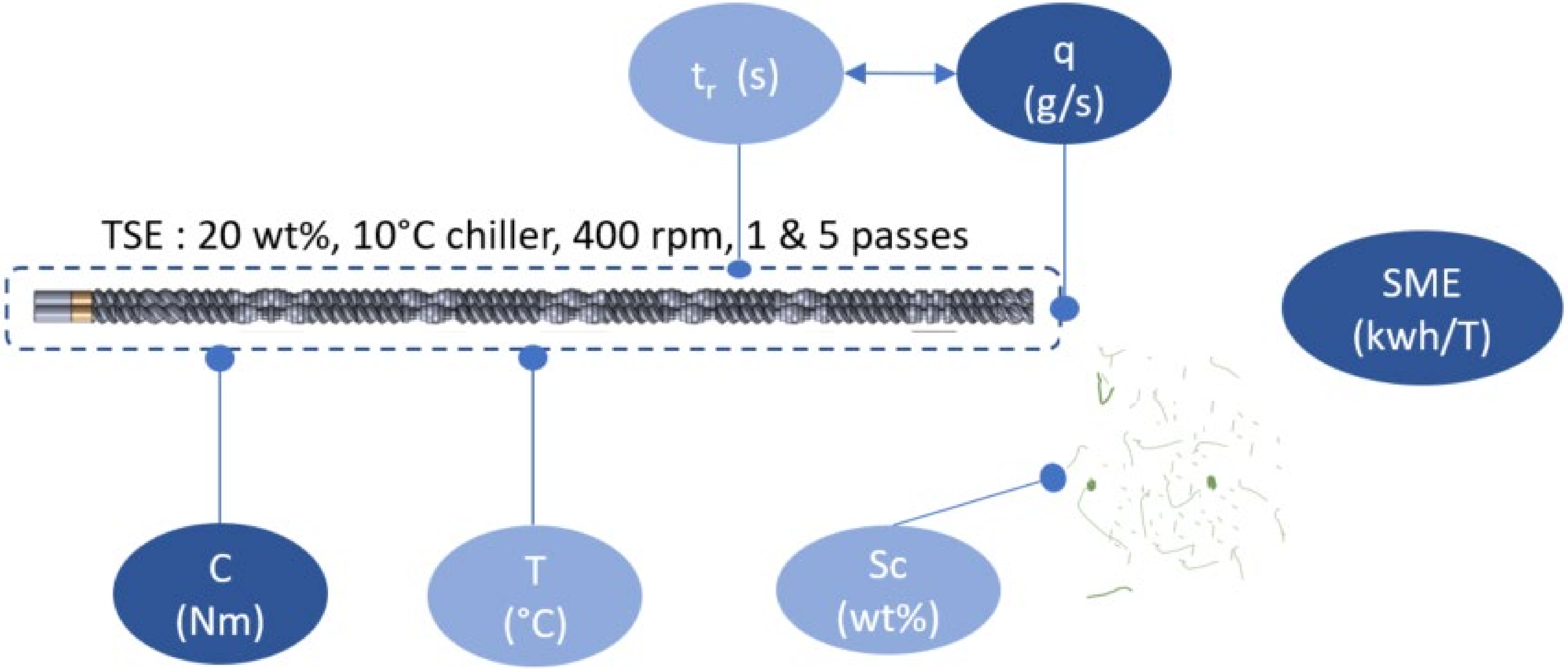
4) TEMPO



COO = 1,5 mmol/g

TSE parameters





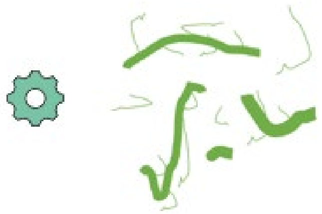
TSE parameters

Solid content after pretreatments 20wt%

1st PASS Feeding rate

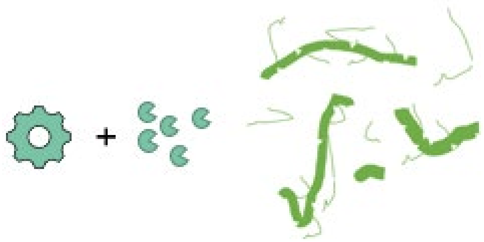
Enz

Mech



2%

Mech + Enz



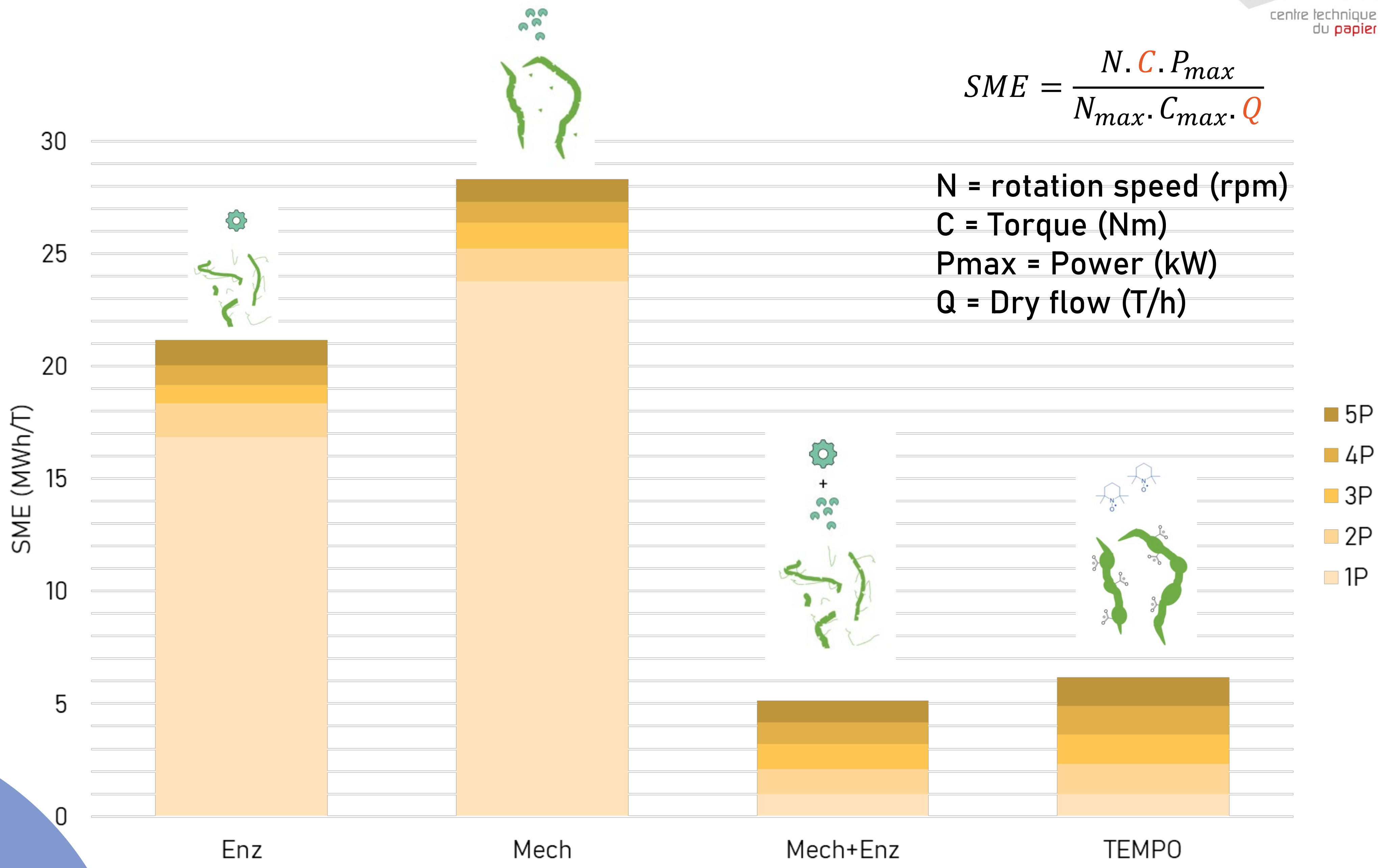
TEMPO

20%

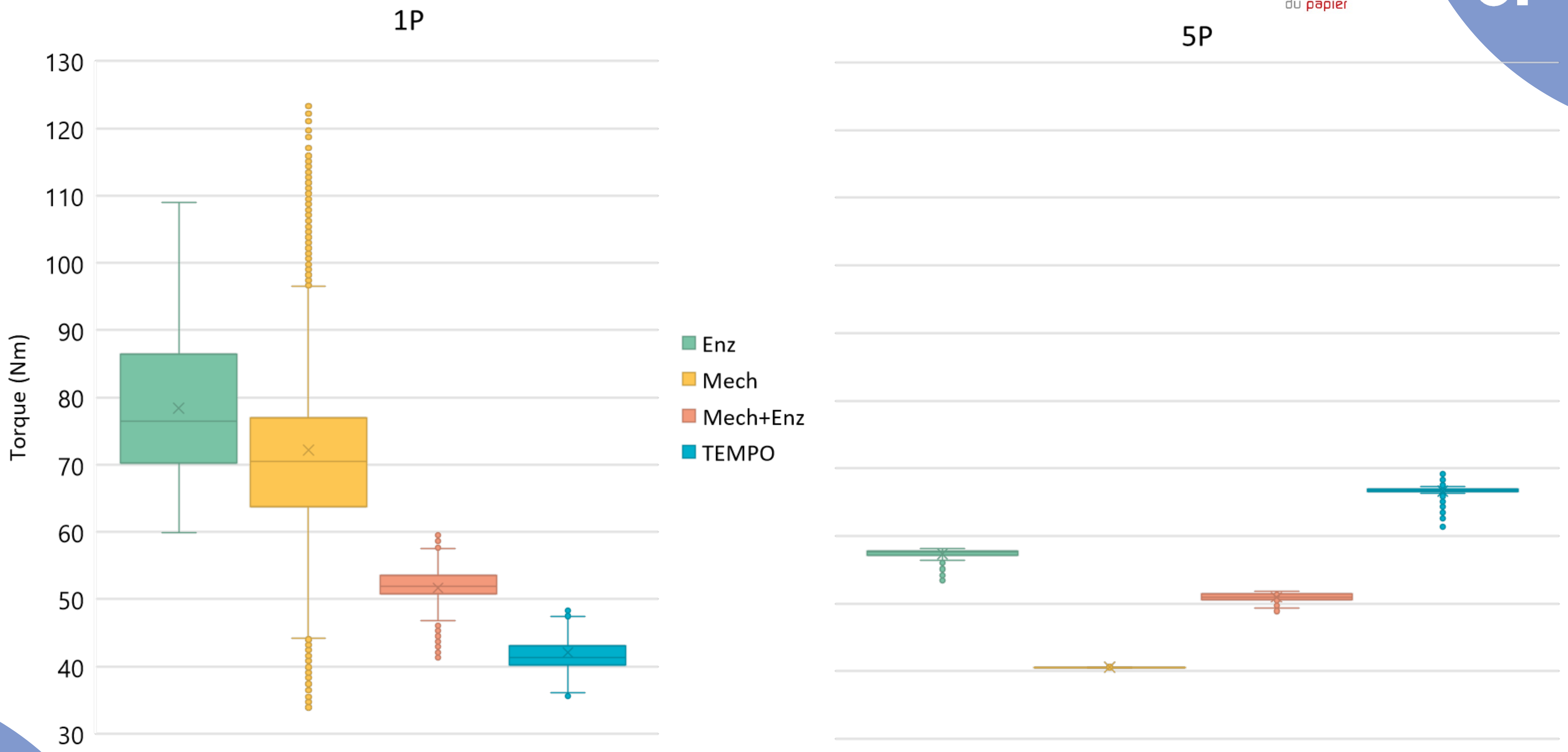
Energy consumption

$$SME = \frac{N.C.P_{max}}{N_{max}.C_{max}.Q}$$

N = rotation speed (rpm)
C = Torque (Nm)
Pmax = Power (kW)
Q = Dry flow (T/h)

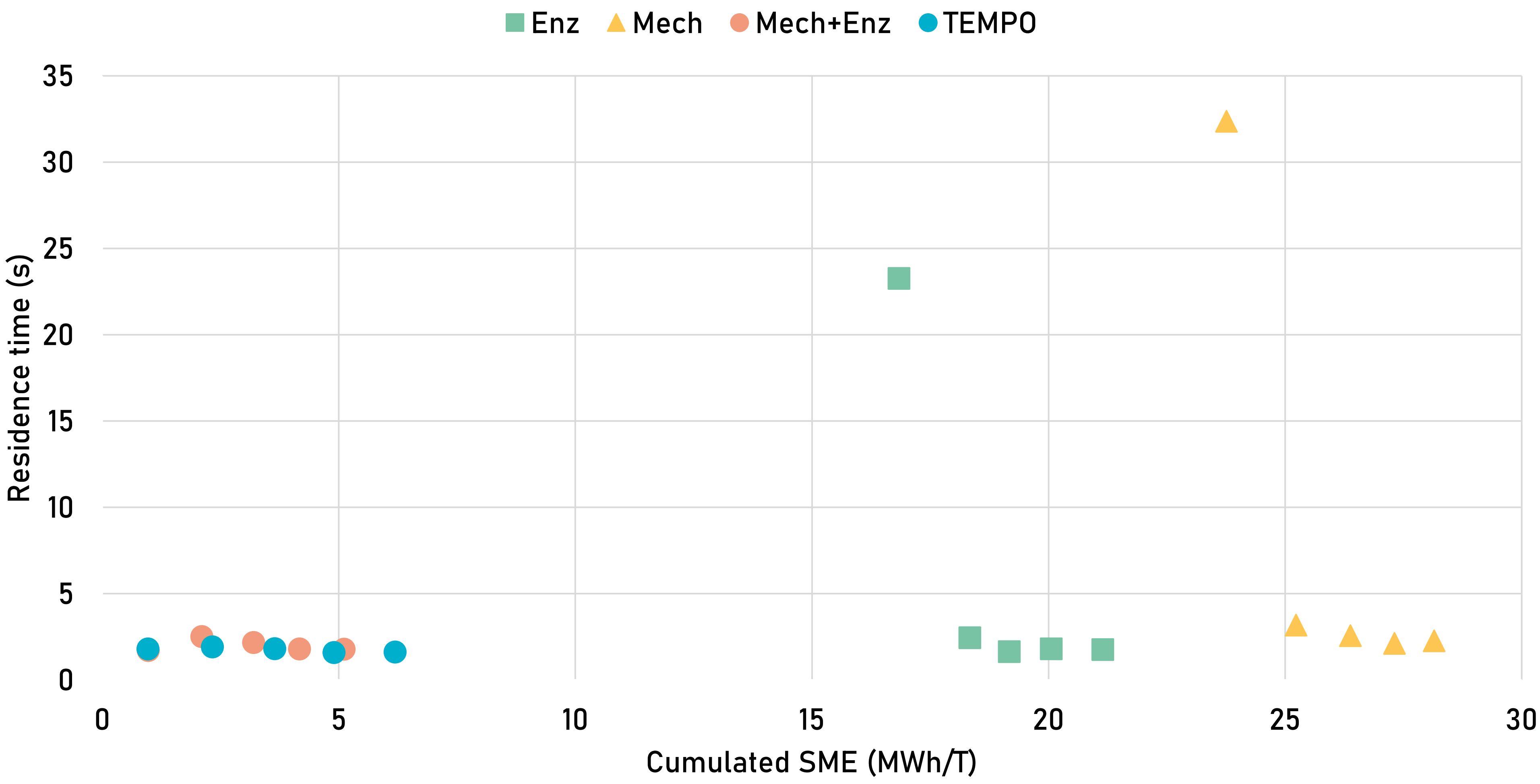


Torque stability



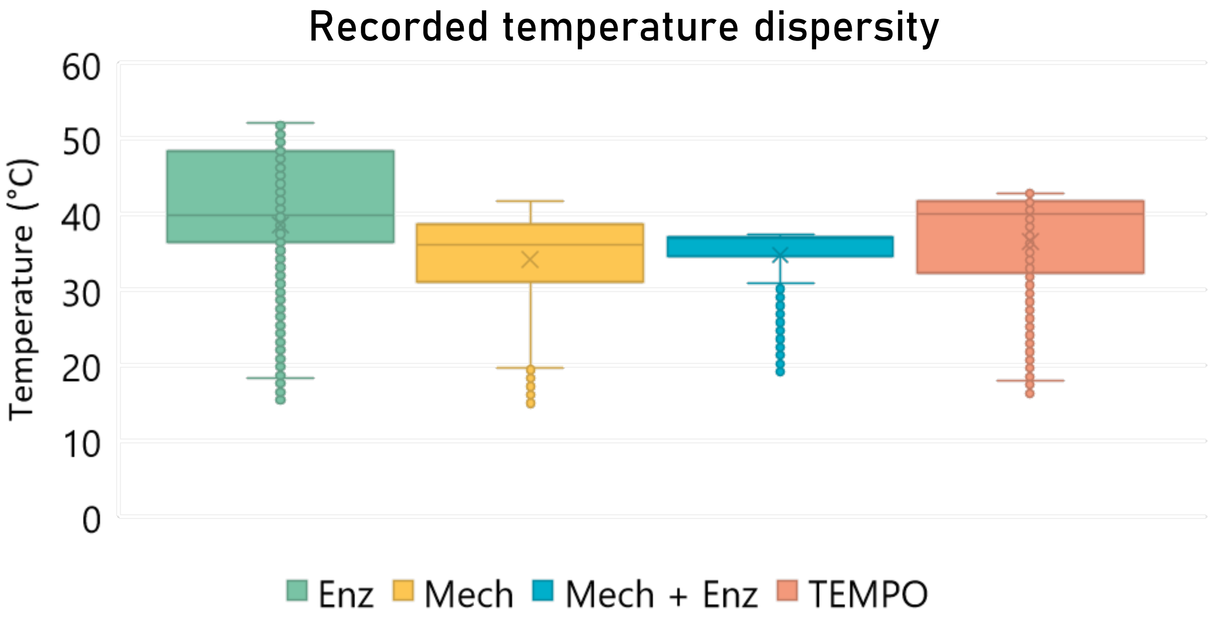
Residence time

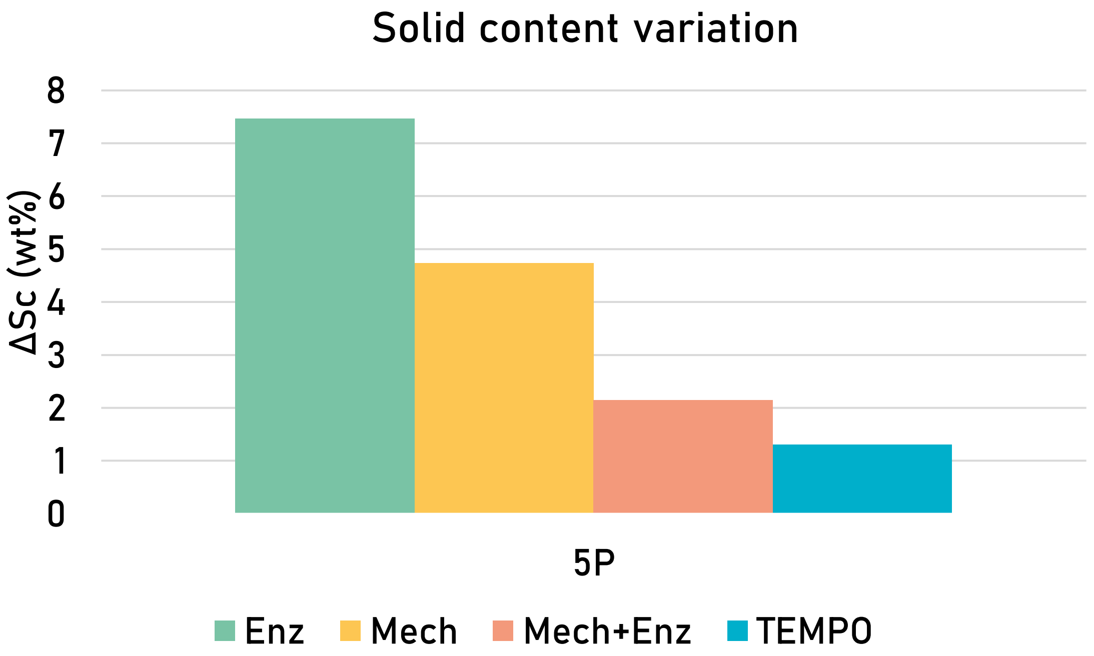
$$t_r = \frac{V_{TSE} \cdot \rho_{NP}}{q}$$ with ρ_{NP} = 1070 kg/m^3



Temperature and solid content

$$\Delta Sc = Sc_{extr} - Sc_{init}$$





→Higher T° during the 1st pass

→ Water loss due to leaks in the feeding zone

→ Water evaporation

Stability conclusions

Mech+Enz

- Lower energy consumption (1P =1000 kWh/T)
- Constant and low torque among passes
- Stable residence time and mass flow
- No water losses
- Controlled temperature process

Agenda

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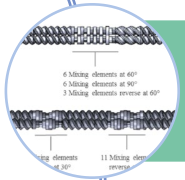

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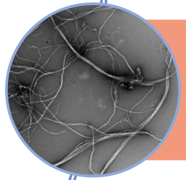

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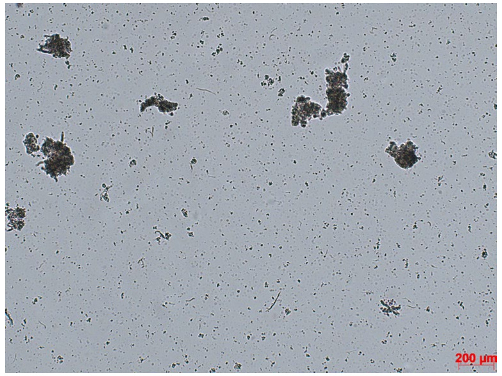
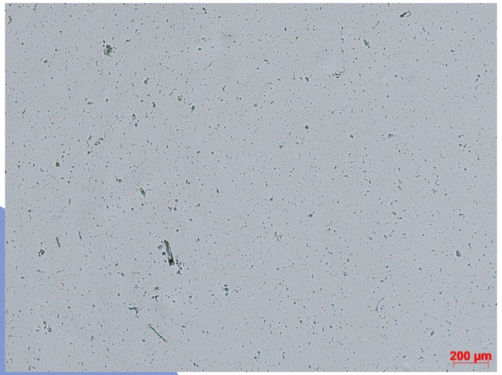
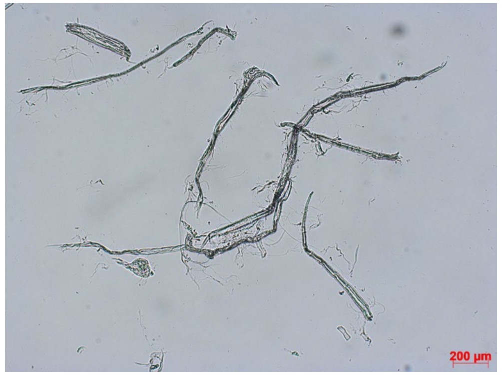
Optical microscopy

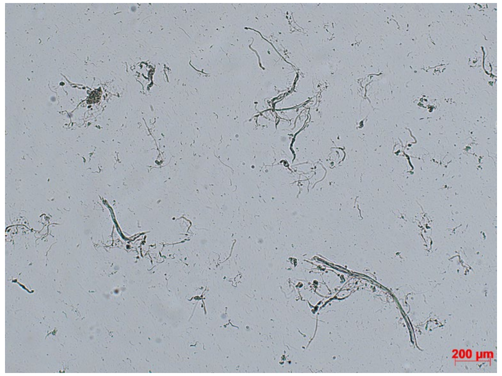
Enz



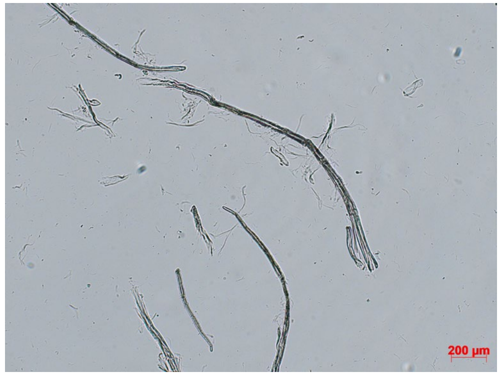


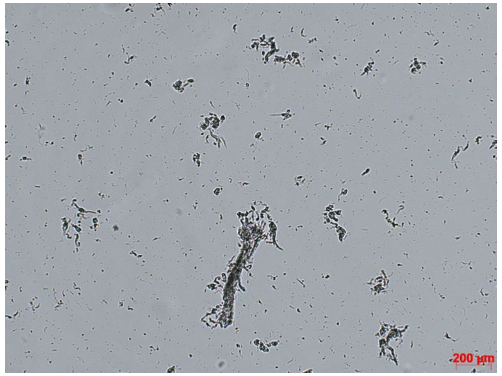
Mech

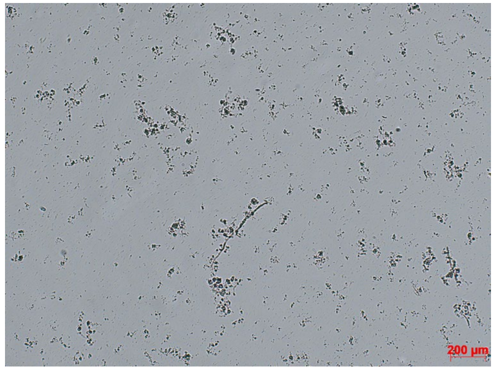




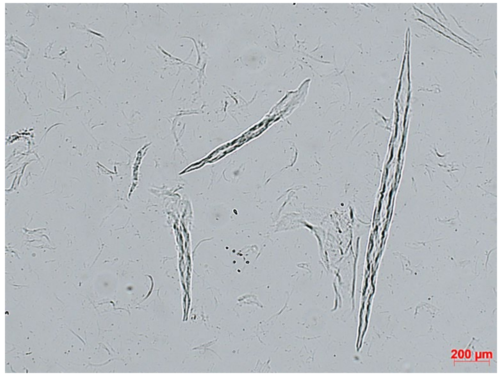
Mech + Enz

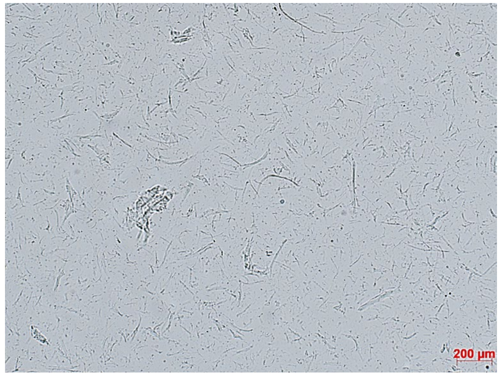






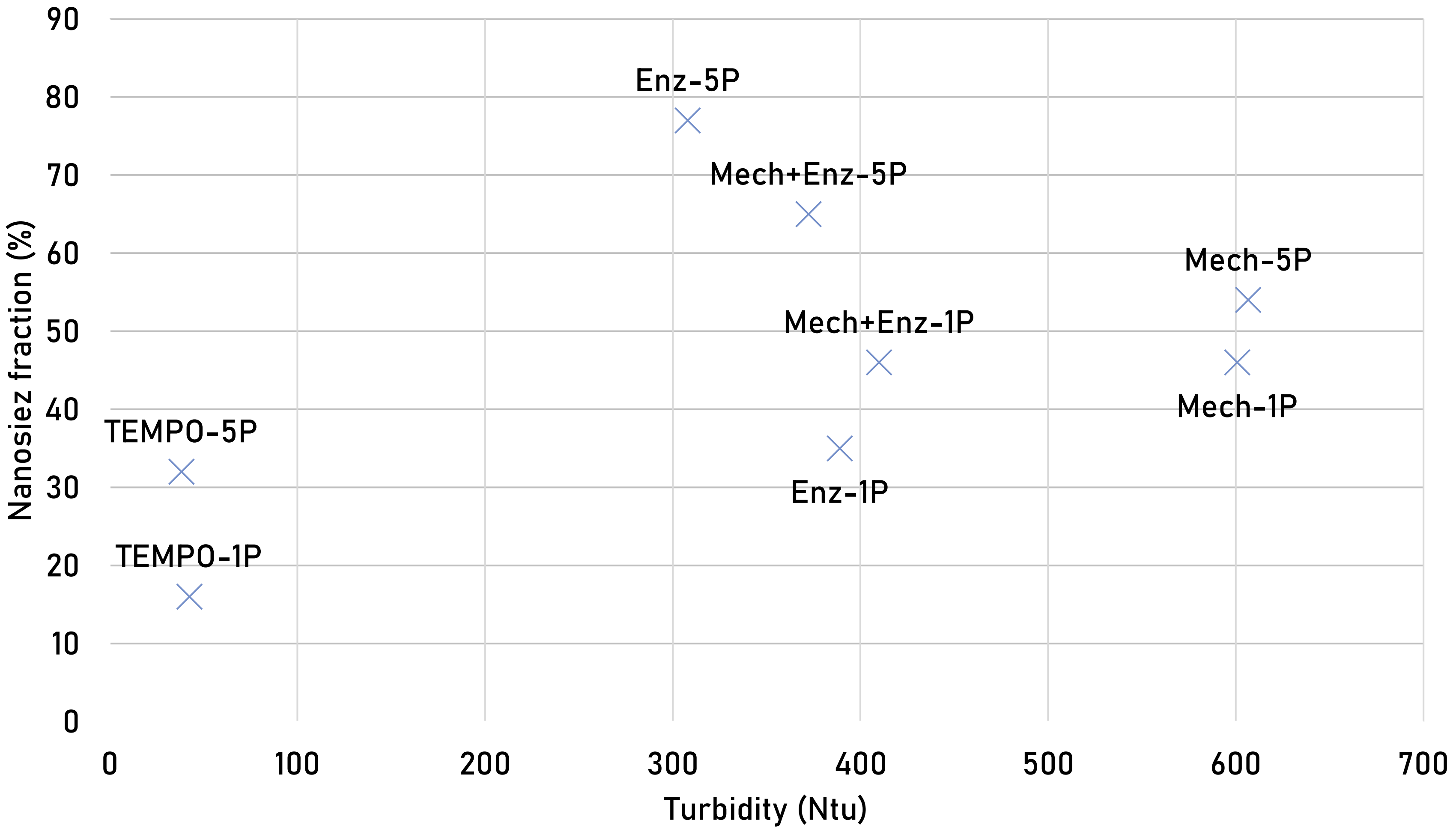
TEMPO





Nanosize characterization

0,02 wt%
1000 g
15min
4°C



0,1 wt%, 860nm

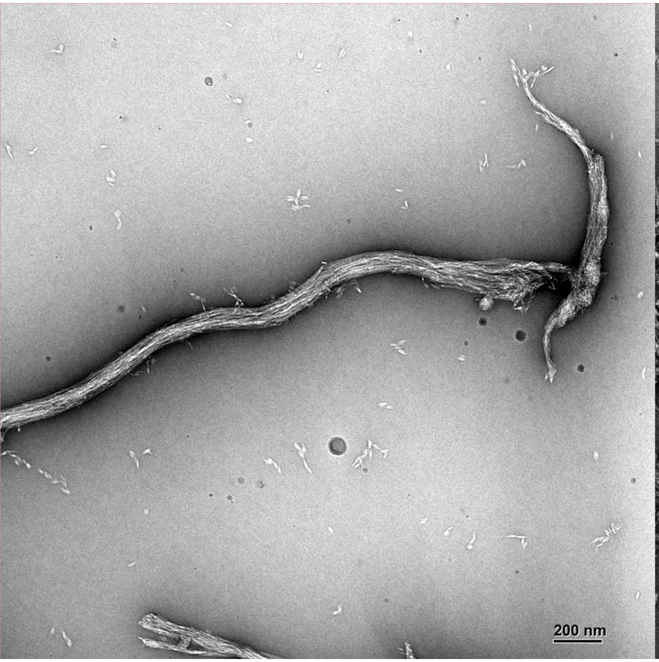
Lower tubidity → higher NF

TEMPO better dispersity → lower turbidity

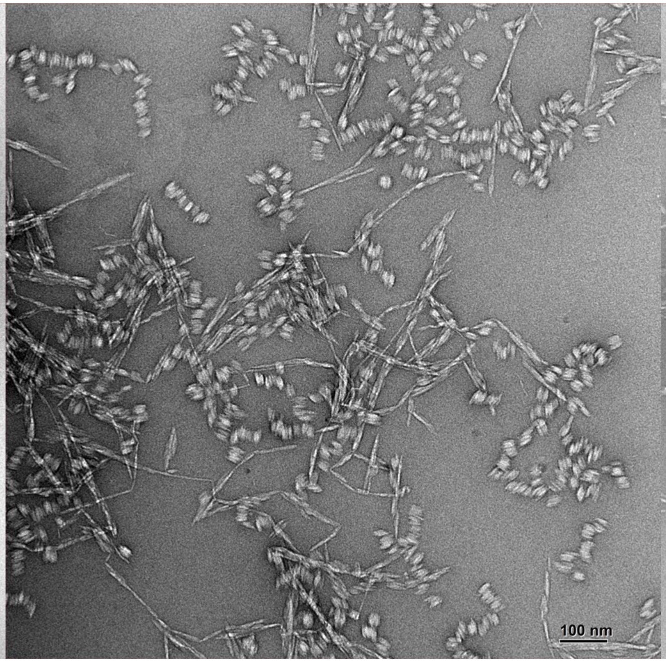
Only one characterization is not enough

TEM

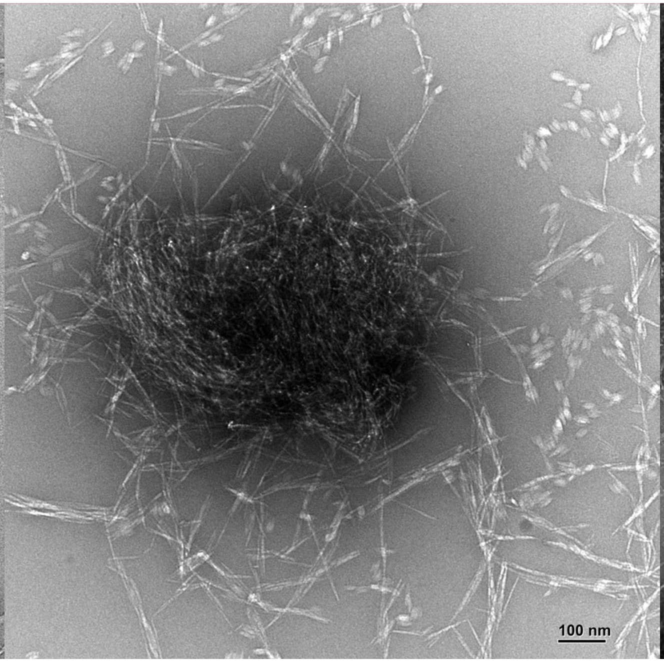
Enz



Mech



Mech + Enz



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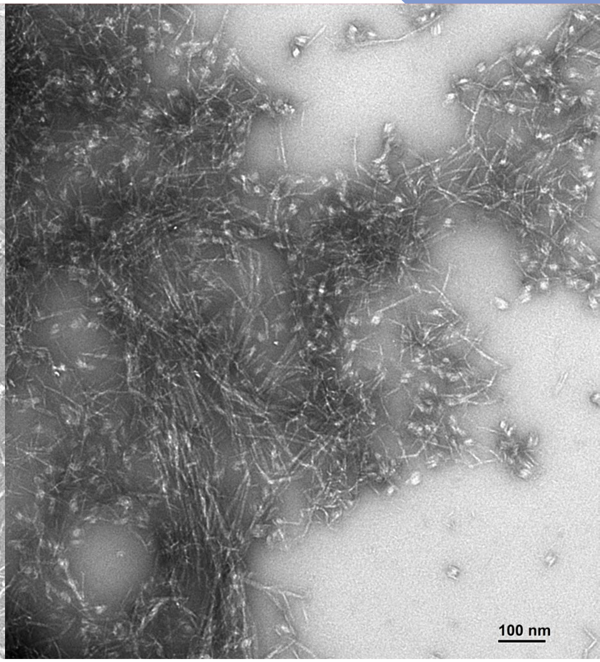
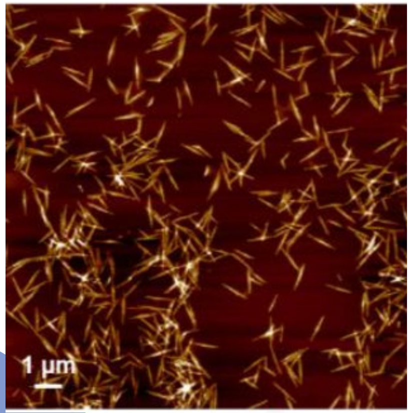
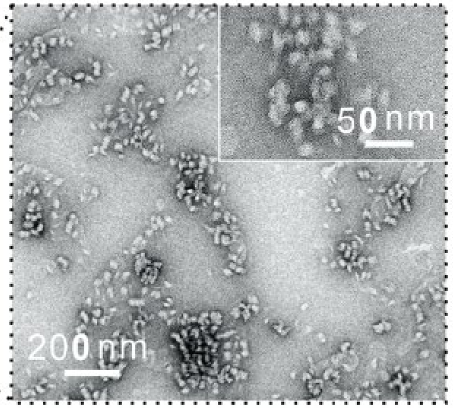


1.



Xylan Nanocrystals ?

2.



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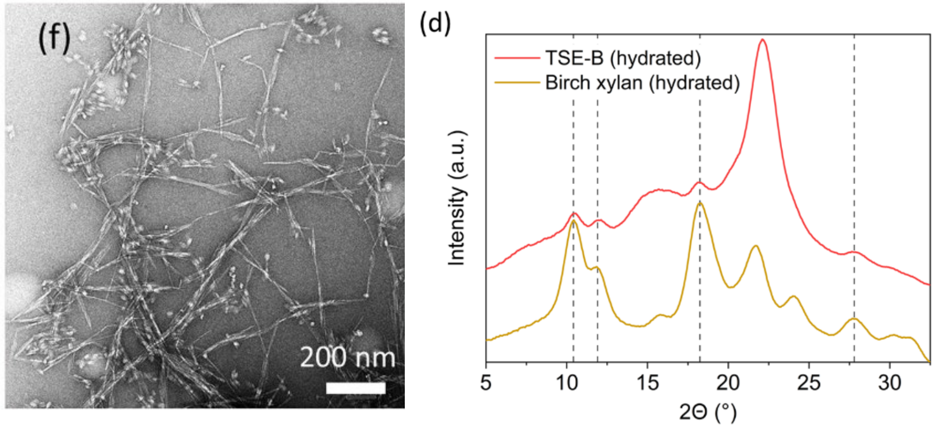
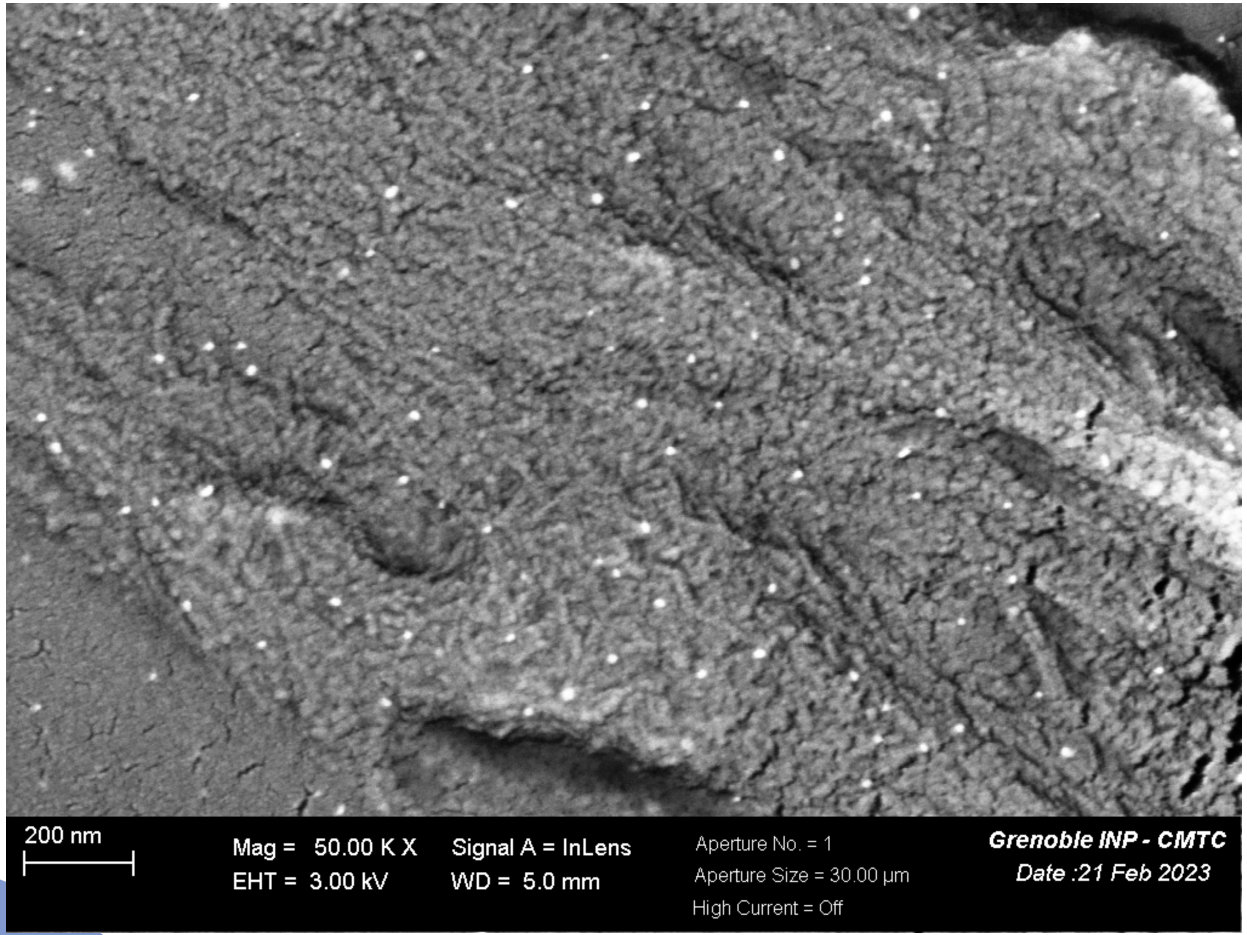


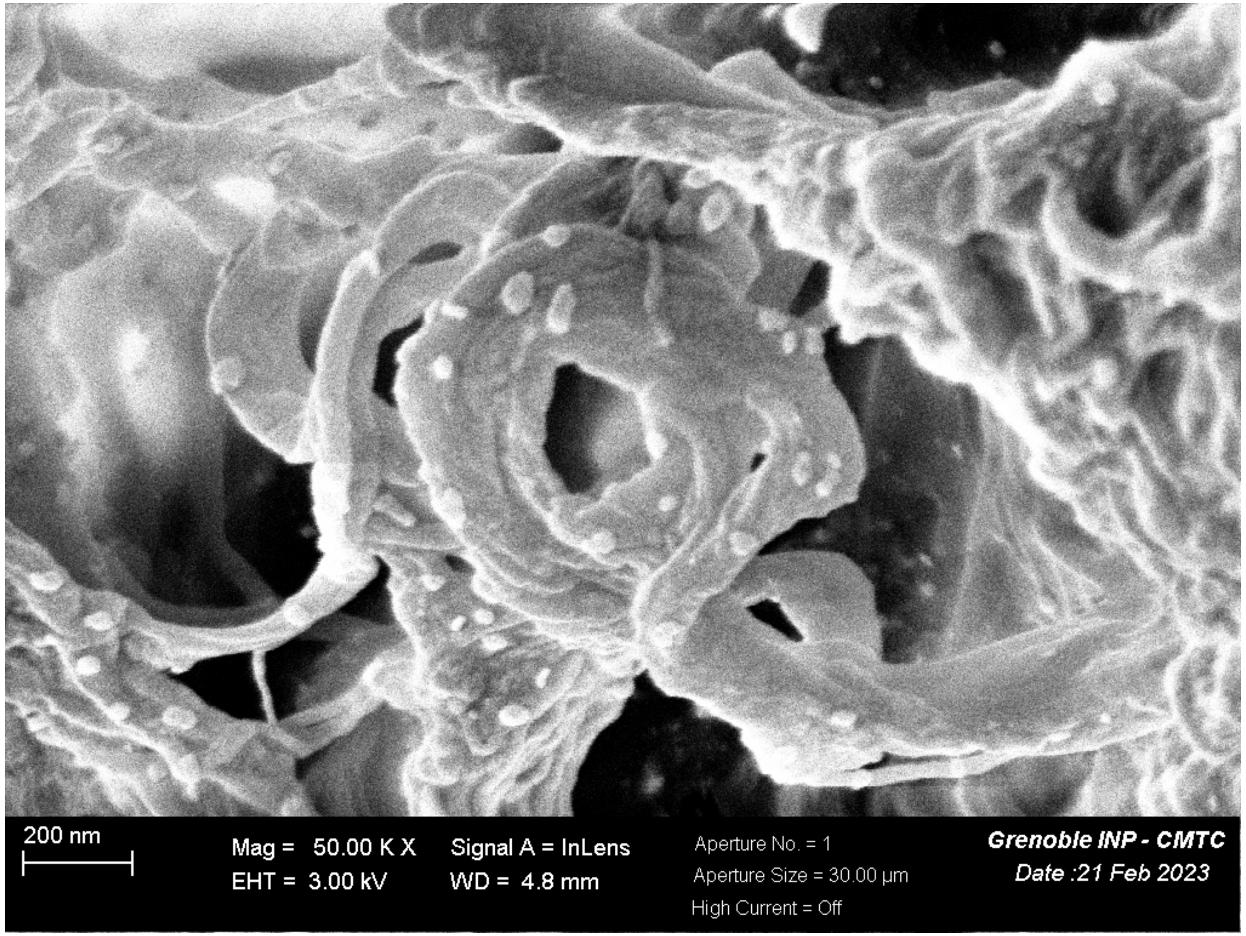
1. Zhuojun *et al.* « Bottom-up Construction of Xylan Nanocrystals in Dimethyl Sulfoxide ». *Biomacromolecules*, 2021
2. Wang *et al.* « Repurposing Xylan Biowastes for Sustainable Household Detergents ». *ACS Sustainable Chemistry & Engineering* 2023.
3. Banvillet *et al.* « Cellulose Nanofibril Production by the Combined Use of Four Mechanical Fibrillation Processes with Different Destructuration Effects ». *Cellulose* 2023

SEM

Enz


200 nm
Mag = 50.00 K X
EHT = 3.00 kV
Signal A = InLens
WD = 5.0 mm
Aperture No. = 1
Aperture Size = 30.00 µm
High Current = Off
Grenoble INP - CMTC
Date :21 Feb 2023

Mech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EHT = 3.00 kV
Signal A = InLens
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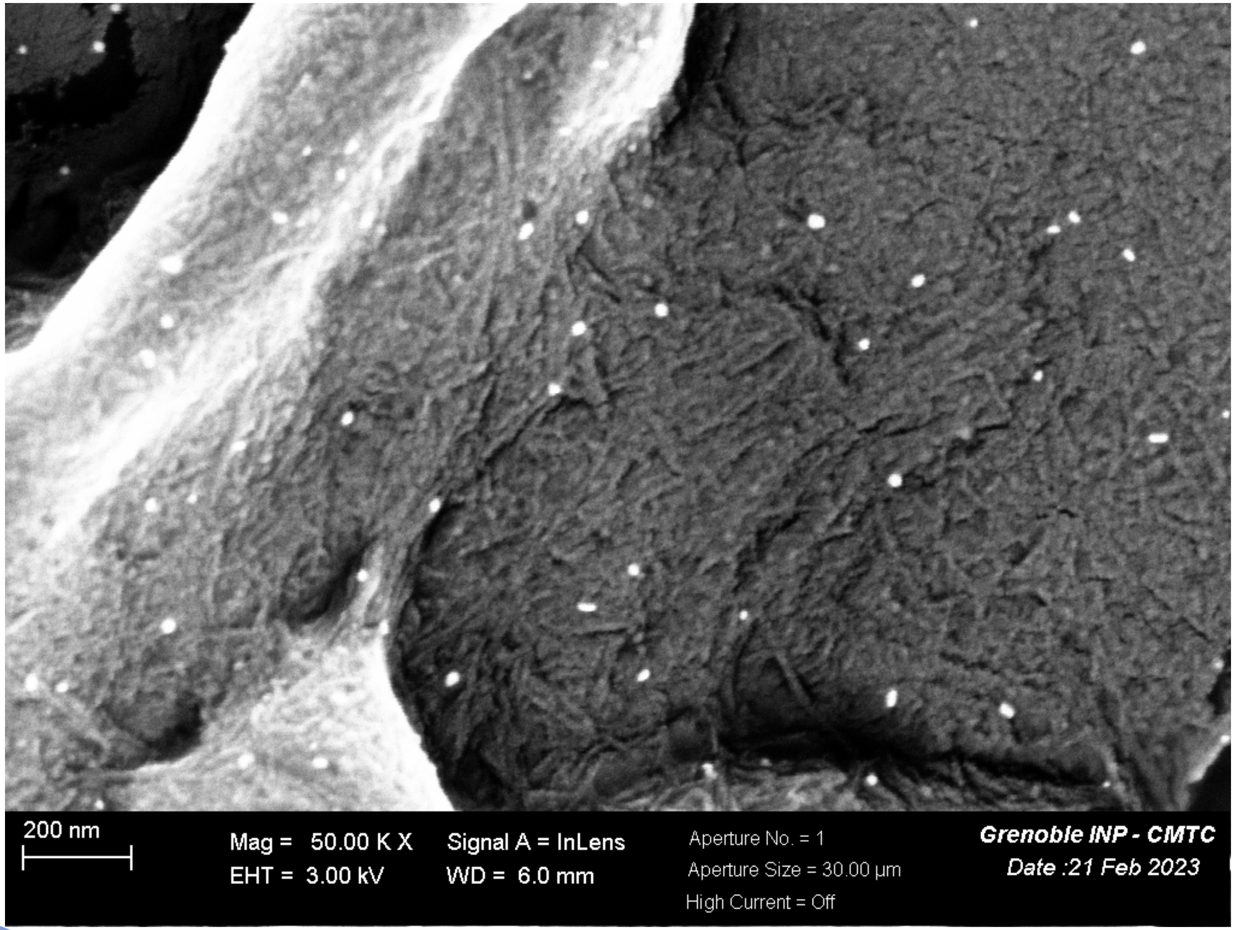
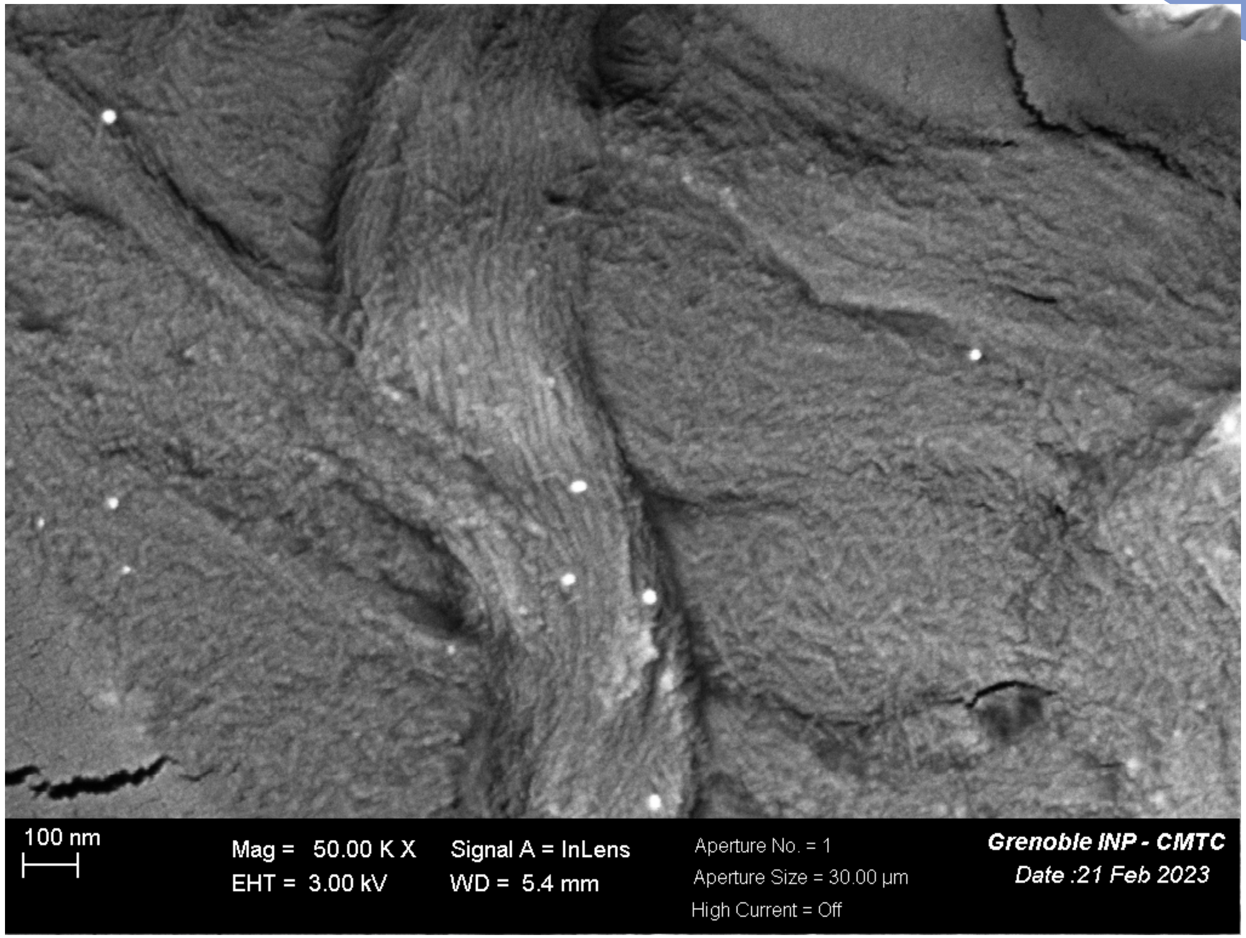
Aperture Size = 30.00 µm
High Current = Off
Grenoble INP - CMTC
Date :21 Feb 2023

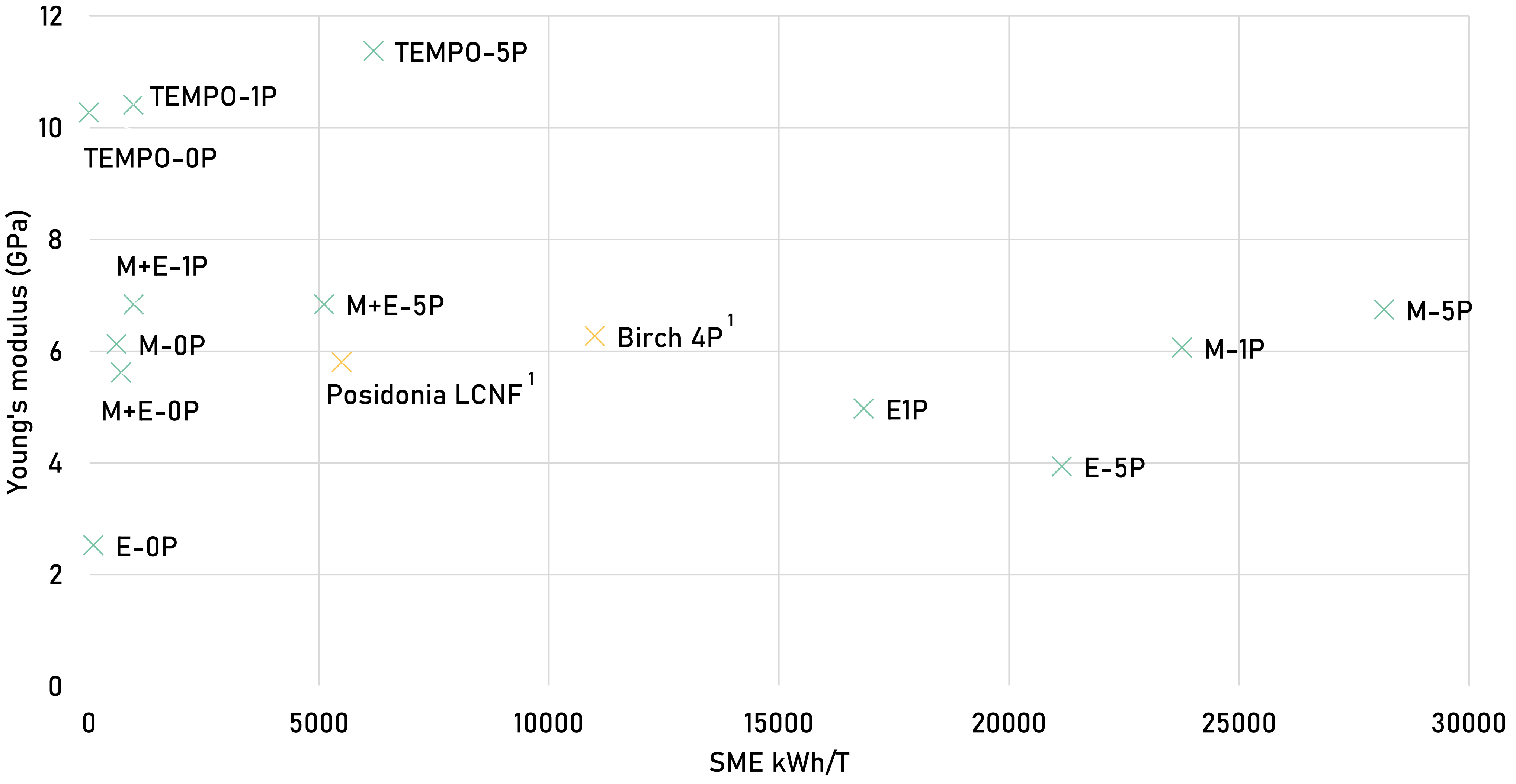
Mech + Enz



TEMPO



Mechanical properties



1. Khadraoui Malek, et al. *Industrial Crops and Products*, 2022
2. Banvillet Gabriel et al *Cellulose*, 2023

Conclusion

- Mech + Enz → Stable TSE process (SME, C, t, q, Sc, T°)
- High NF after 5P → XNC creation ?
- Poor mechanical properties → dispersion / CNF morphologies / Sugars

Acknowledgment

- TEM: Jean Luc PUTAUX, CERMAV
- SEM-FEG: Francine ROUSSEL, CMTC

Thank you for your attention

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