

# 4-acetamido-TEMPO-mediated oxidation of wood chips and thermomechanical pulp in large scale

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Presented by David Myja

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en **écomatériaux**, **écoproduits**  
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# Why did we work with TEMPO ?

## TEMPO oxidation

Low oxidation

High oxidation

Increase paper strength  
(mainly tensile resistance)

Improve nanofiber production  
in water

New grade of use for TMP ?

Many possible applications:

- Paper reinforcement
- Medicine
- Electronic paper
- Humidity sensor
- ...

What's next ?

Scale up

How ?

Implantation at different step in the TMP  
production process

# Studied treatments

## On softwood chips:

- Impregnation with TEMPO medium (TC)
- Large batch reactor oxidation (BTC)

## Reference:

- Untreated chips (C)

## Then:

- Primary and secondary refining
  - Disc gap from 0.15 to 1.00 mm

## On softwood primary TMP:

- Replace water during the refining by TEMPO medium (TP)
- High consistency reactor treatment (HCTP)
- Large batch reactor oxidation (BTP)

## Reference:

- Untreated primary pulp (P)

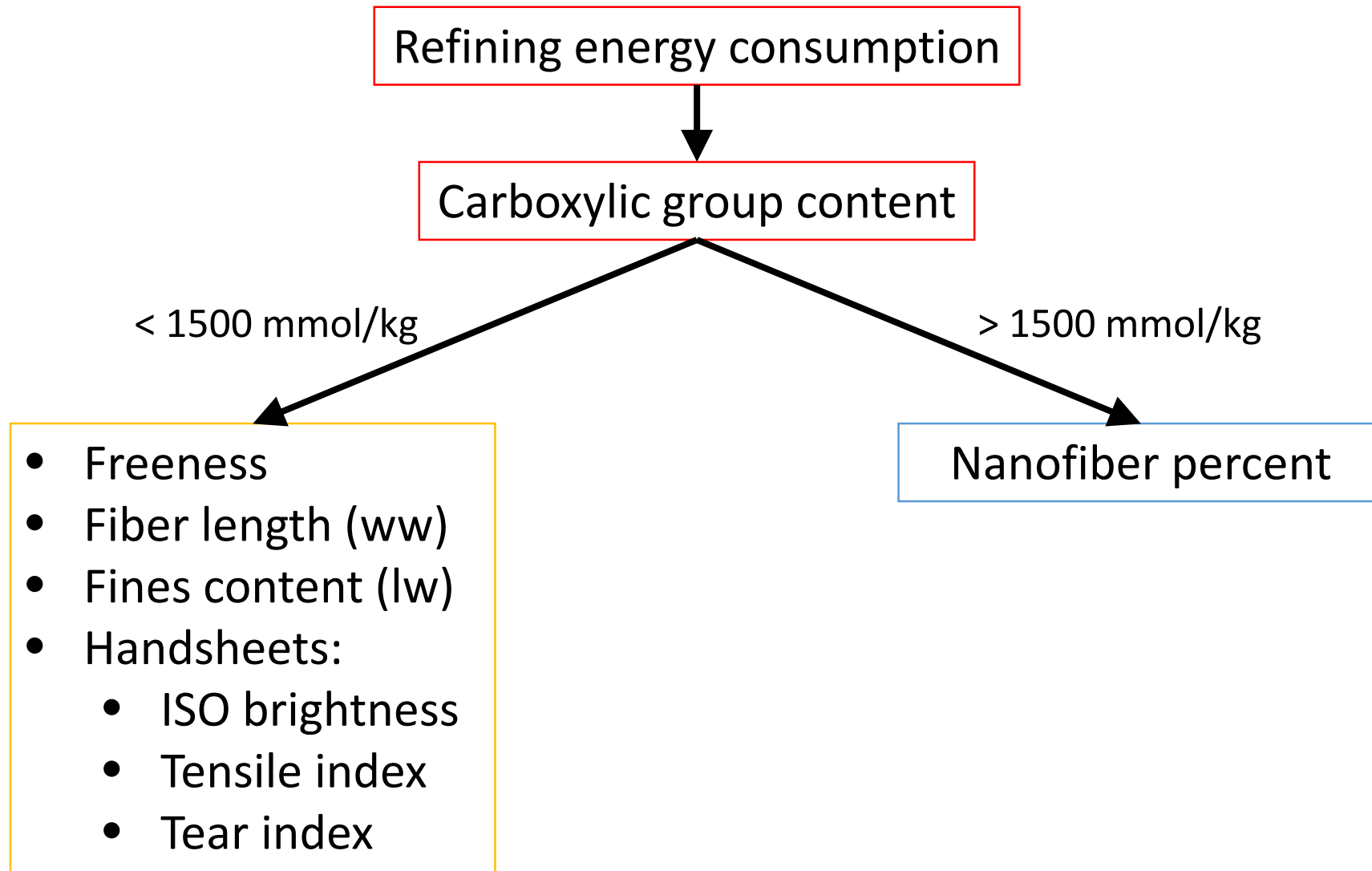
## Then:

- Secondary refining
  - Disc gap from 0.15 to 1.00 mm

# Analysis on primary pulp from softwood chips

- Refining energy consumption
- Freeness
- Fiber length (ww)
- Fine content (lw)
- Shive content
- Carboxylic group content of:
  - Pulp
  - Shives
  - Fibers

# Analysis on secondary pulp



# Pulp bleaching

Secondary pulp with freeness between 100 et 200 mL

Chelation of metal ions with DTPA

Hydrogen peroxide bleaching

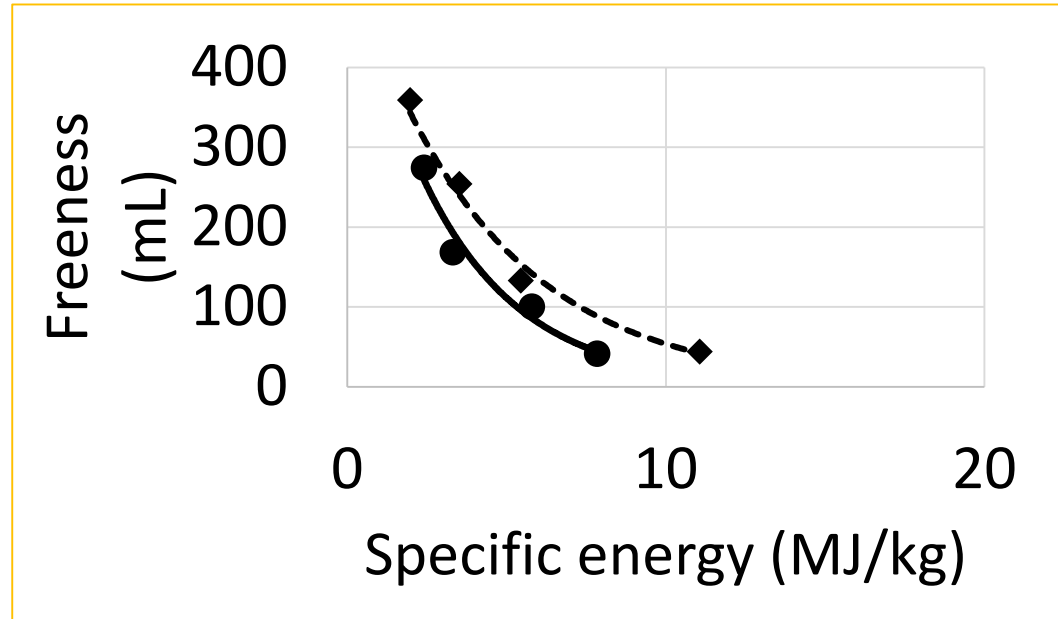
4 gram handsheets:  
ISO brightness  
Yellowness ( $b^*$ )

# Observations on primary pulp from chips

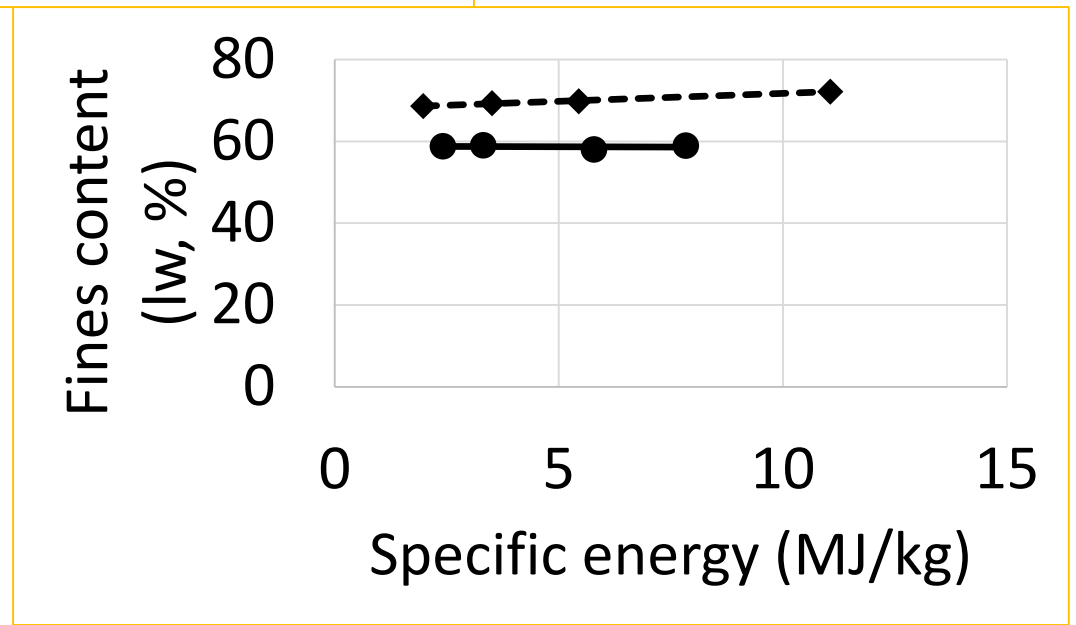
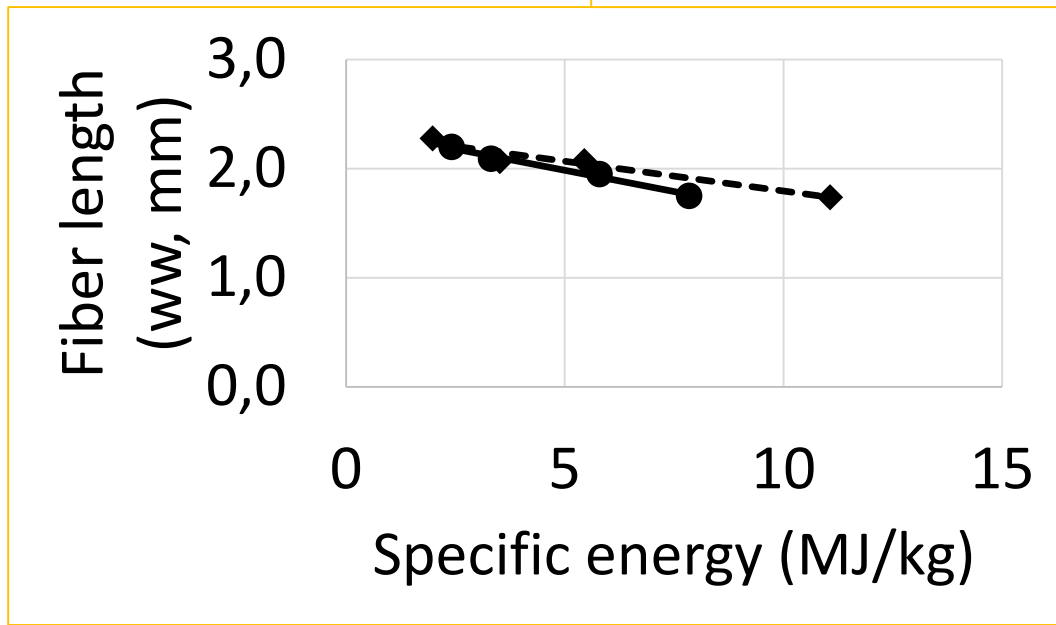
Pulp	Disc gap	Specific energy	Freeness	Fiber Length (ww)	Fines content (lw)	Shives content	Carboxylic group content		
							Pulp	Shive	Fiber
							mmol/kg		
<b>C1</b>	1.00	2.27	715	2.21	61.0	24.1	120	41	145
<b>TC1</b>	1.00	2.43	702	2.25	61.8	27.1	220	45	285
<b>BTC-1</b>	1.00	1.13	425	1.11	69.5	47.5	545	89	958
<b>BTC-2</b>	0.70	2.19	297	1.21	71.0	40.9	605	69	977
<b>BTC-3</b>	0.15	4.27	217	1.62	64.3	21.2	675	58	841



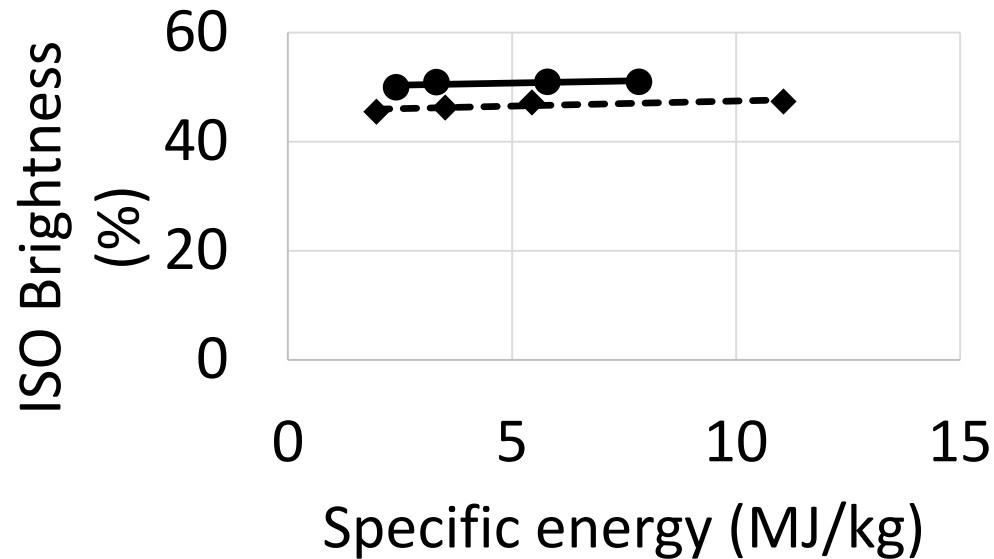
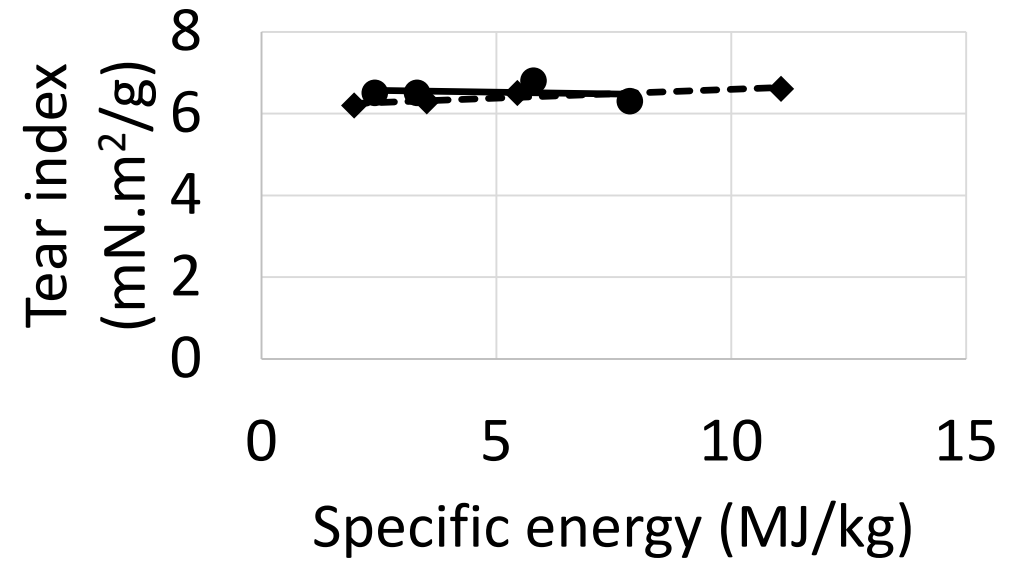
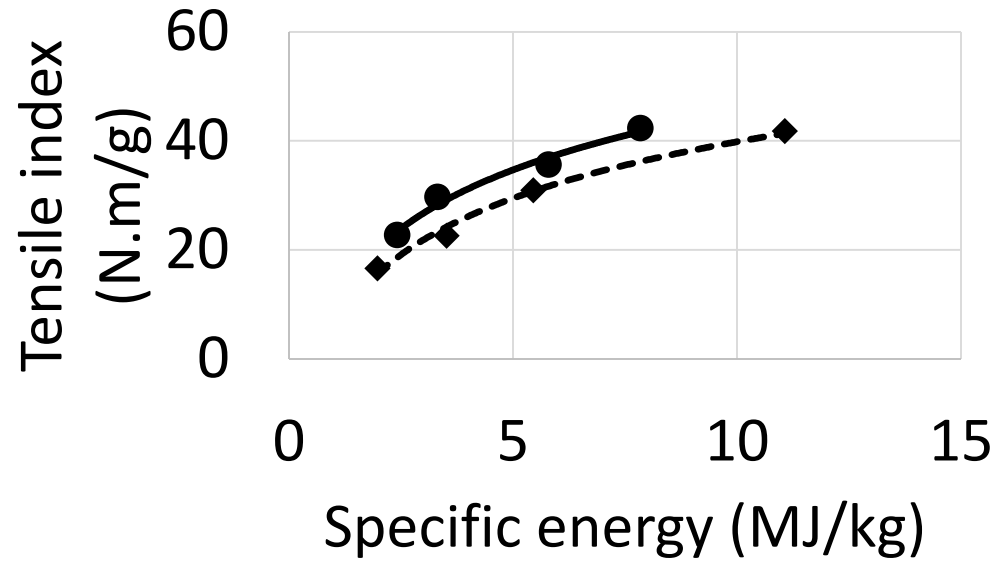
# Observations on secondary pulp from chips



- C2
- ◆ TC2



# Observations on secondary pulp from chips



- C2
- ◆ TC2

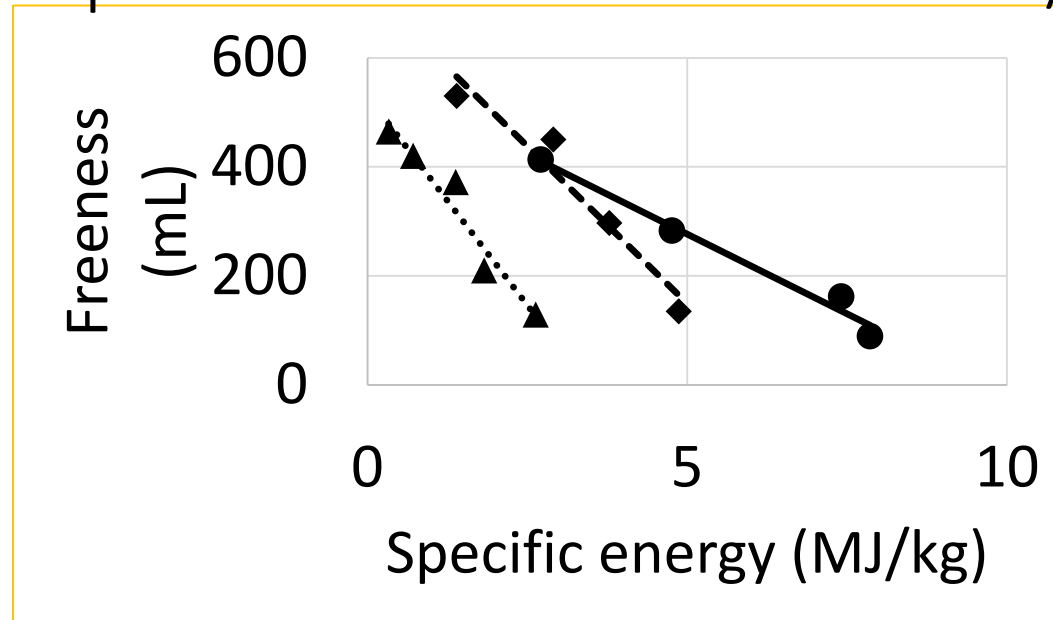
# Bleaching effect on untreated and impregnated with TEMPO medium chips

<b>Pulp</b>	<b>Before hydrogen peroxide bleaching</b>		<b>After hydrogen peroxide bleaching</b>	
	ISO Brightness (%)	Yellowness (b*)	ISO Brightness (%)	Yellowness (b*)
<b>C2-2</b>	51.8	13.3	72.2	11.3
<b>TC2-3</b>	47.2	14.2	61.8	14.7

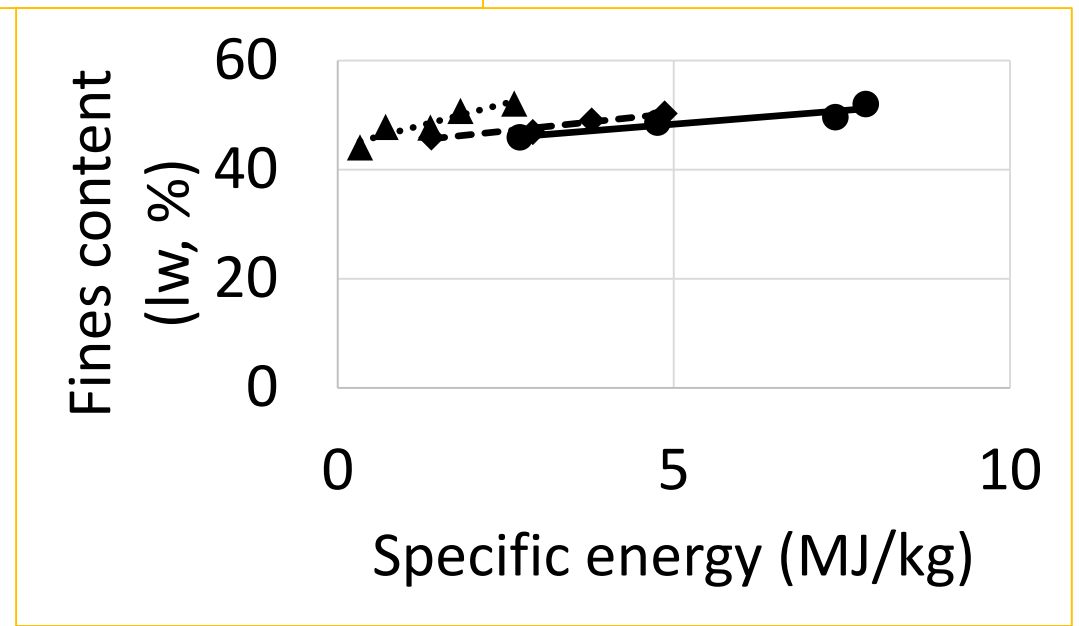
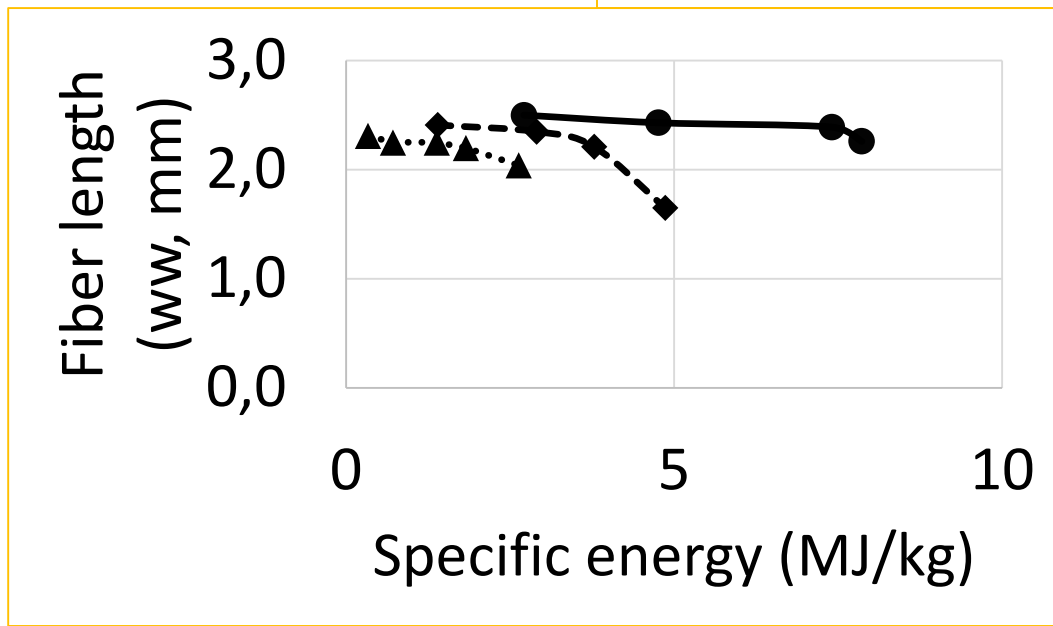
# Conclusions on chips treatments

- Harmfull effect of chips impregnation with TEMPO mixture  
→ Not recommanded
- Chips batch oxidation effective and change refining effects  
→ Further investigation needed

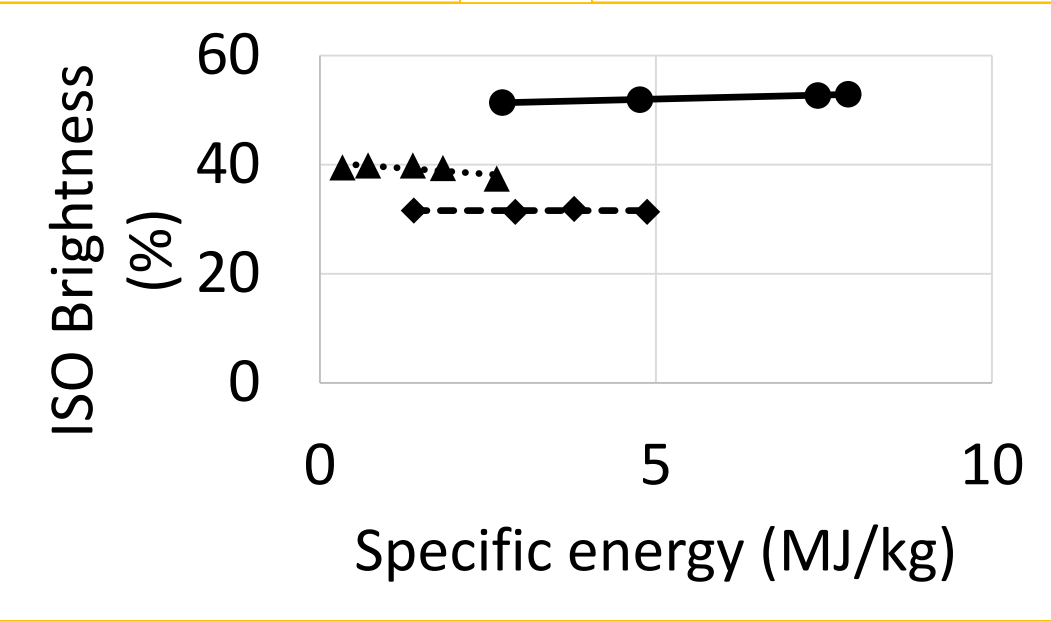
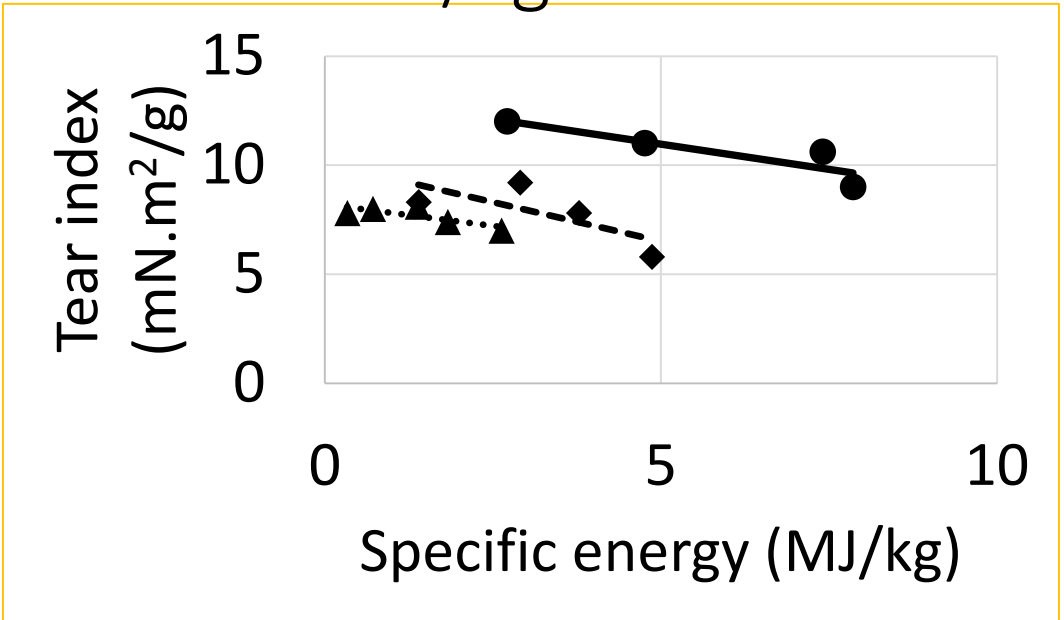
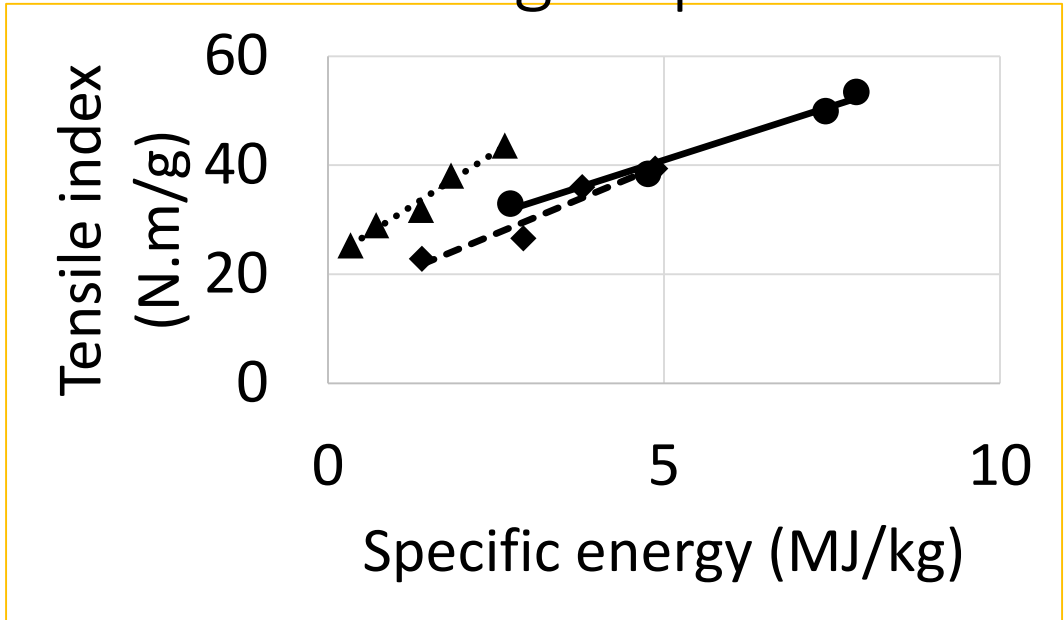
# Observations on secondary pulp from primary TMP with a carboxylic group content under 1500 mmol/kg



- P
- ◆ TP
- ▲ HCTP



# Observations on secondary pulp from primary TMP with a carboxylic group content under 1500 mmol/kg



- P
- ◆ TP
- ▲ HCTP

# Bleaching effect on untreated, refined with TEMPO medium and oxidized in high consistency reactor primary TMP

Pulp	Before hydrogen peroxide bleaching		After hydrogen peroxide bleaching	
	ISO Brightness (%)	Yellowness (b*)	ISO Brightness (%)	Yellowness (b*)
P-3	55.3	13.0	75.2	11.0
TP-4	30.8	21.4	43.8	24.9
HCTP-5	38.1	17.3	52.1	19.8

# Observations on secondary pulp from primary TMP with a carboxylic group content higher than 1500 mmol/kg

<b>Pulp</b>	<b>Disc gap</b>	<b>Specific energy</b>	<b>Pulp carboxylic group content</b>	<b>Nanofiber percent</b>
	mm	MJ/kg	mmol/kg	%
<b>BTP-1</b>	1.00	0.63	1840	12.3
<b>BTP-2</b>	0.50	3.21	2005	23.8
<b>BTP-3</b>	0.15	4.80	2095	27.2
<b>BTP-IKA</b>	/	57.11	1975	19.9



# Conclusions on primary pulp treatments

- High consistency treatment is better than to replace water in the refiner but harshly affect the paper optical properties
  - Further investigation needed (Better control of the pH?)
- High efficiency dispersion when refining highly oxidized pulp
  - Recommended to be used as disperser

# Global conclusions

- Highly oxidized pulp obtained only with batch treatment of primary TMP
- Batch treatment of chips should be further investigate
- Difficulties to control oxidation conditions during impregnation, inside refiner or high consistency reactor treatments decrease their efficiency
- Refiner is clearly viable for large scale nanofiber producer

# Thank you

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