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Lignocellulosic fiber reinforced composites: influence of compounding conditions on defibrization and mechanical properties

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Impact of thermo-hydro environment and specific mechanical energy on defibring using flow modelling and extrusion

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(Fractionation of lignocellulosic resources and Environment)



Lignocelluloses defibring

We know: final composite properties = f (initial fibre quality & comportment under processing conditions)



Wood



/ long fibres

Extrusion

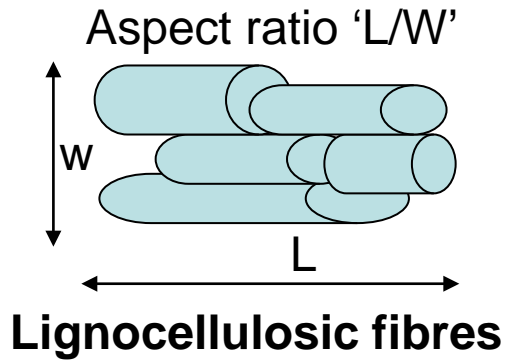
Transformation

+ Polymer matrix



Compound/Composite material

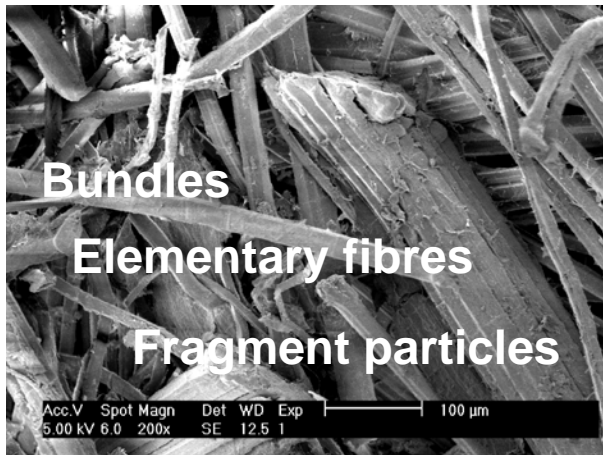
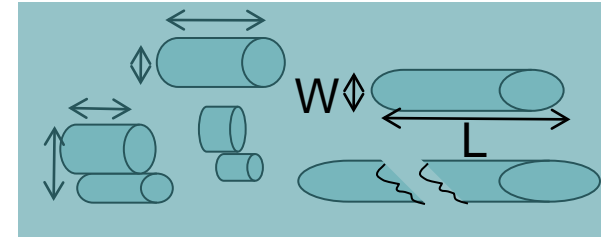
Lignocelluloses defibring



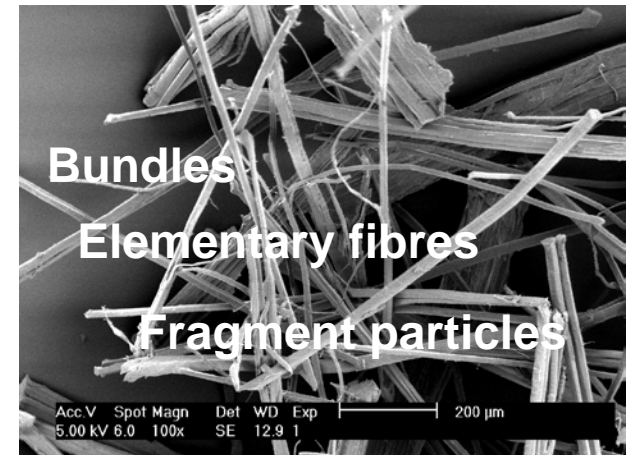
Individualisation

Extrusion

And Fragmentation



Δ Distribution profile

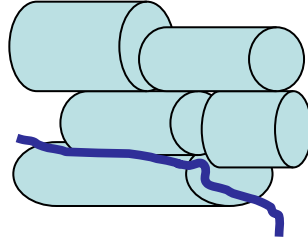


We know: Aspect ratio 'L/W' and size influence composite end uses properties

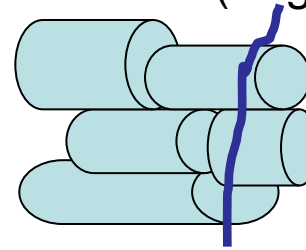
Is it possible to control the defibring in order to enhance the mechanical properties of composites?

Control of the defibring : hypothesis about fibre' ruptures locations?

Inter fibres (individualisation)



Intra fibres (fragmentation)



Structure

decohesion

dammage

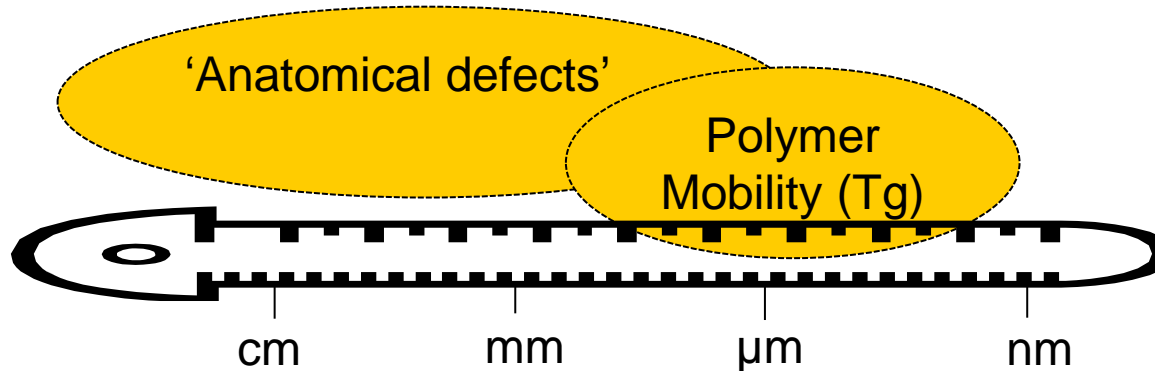
Theoretical L/W



Theoretical material reinforcement



origins?



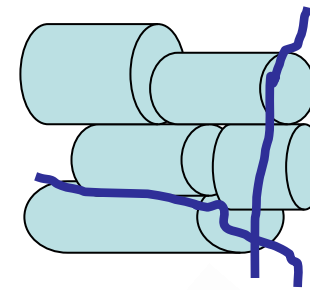
First strategy : playing on fiber polymer mobility



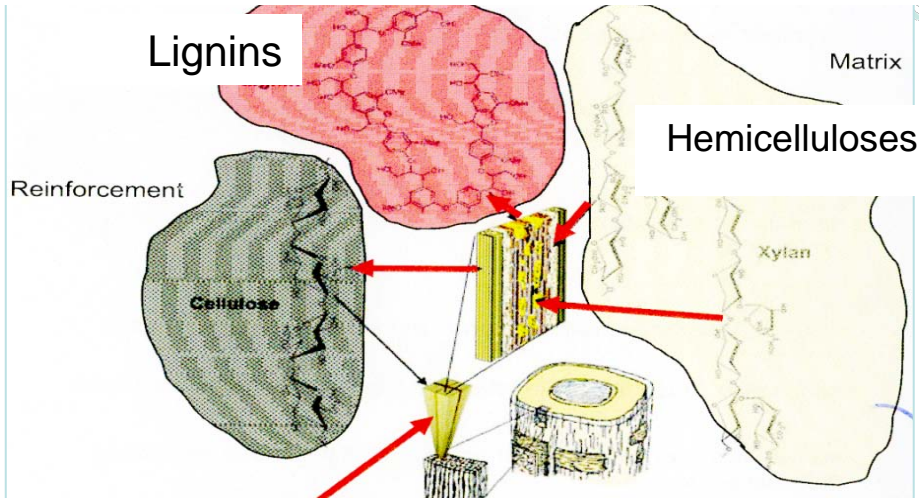
Macroscopic impact



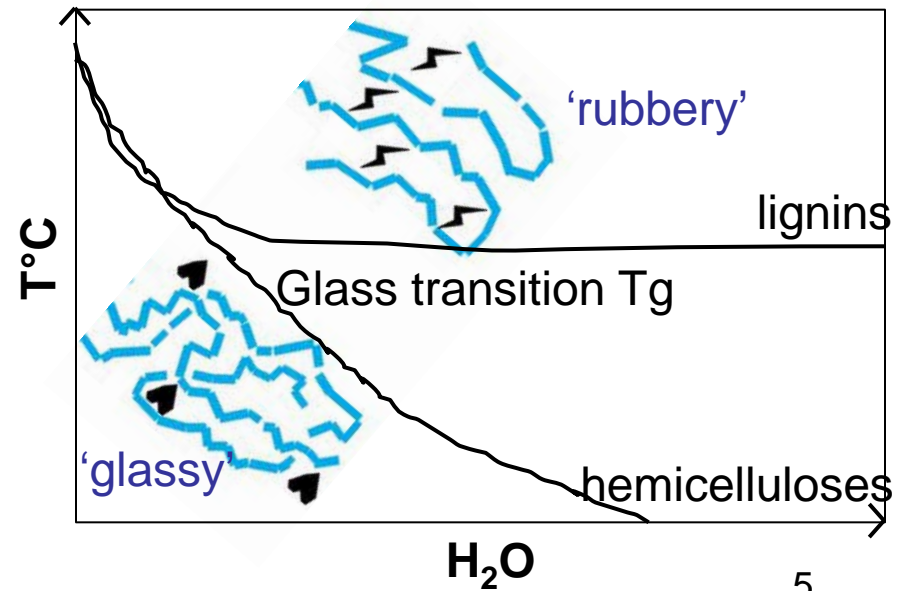
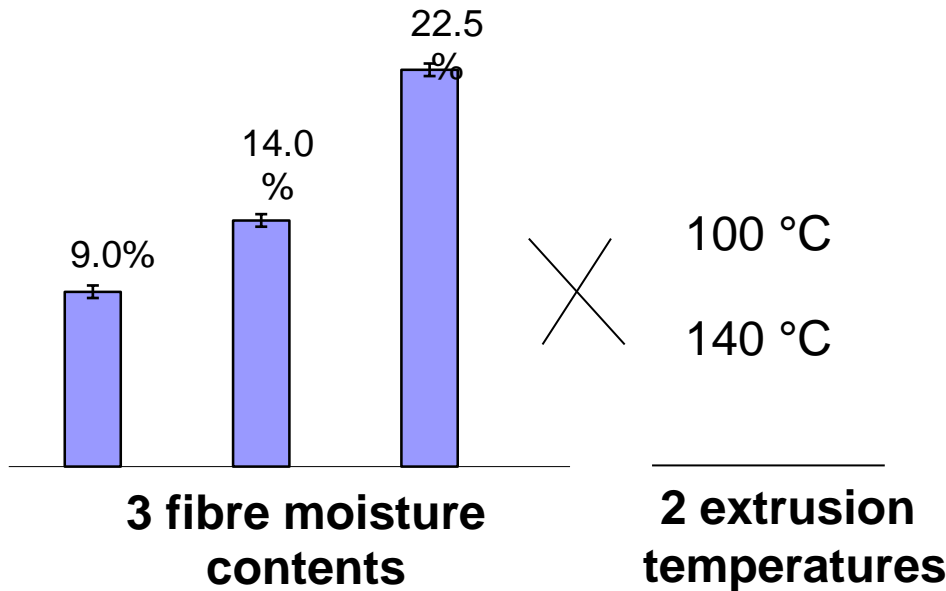
Rupture location



Spatial arrangement of Lignocellulosic polymers



From Salmén and Olsson 1998



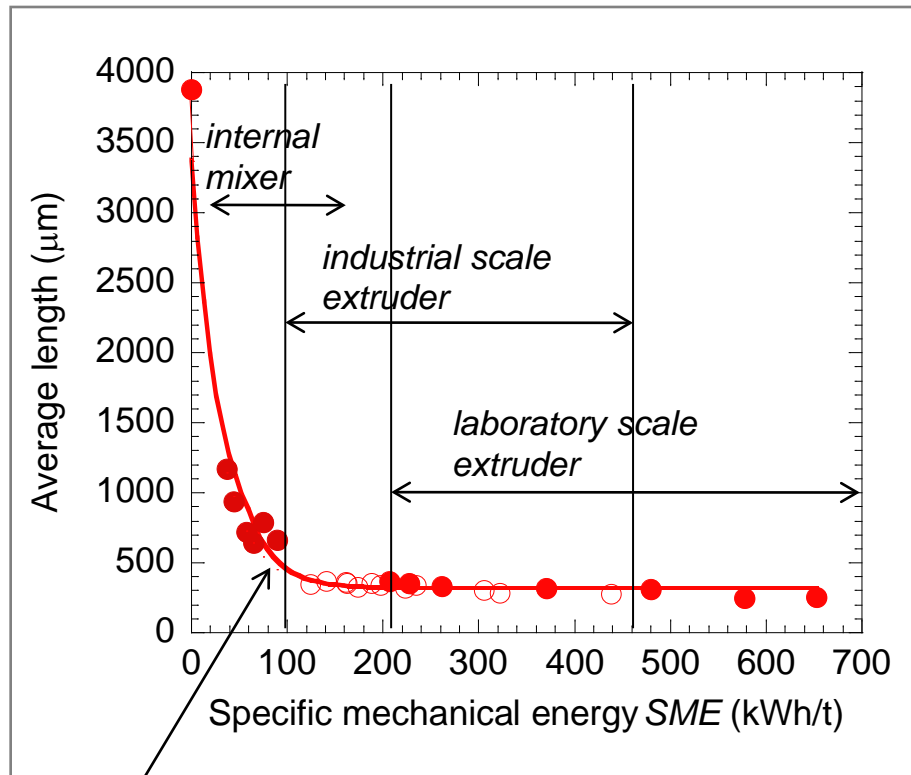
Second strategy : playing on extrusion process

Taken from glass fibre

Transposable to natural fibres?

SME Specific Mechanical Energy (kWh/t)
intensity of the thermomechanical treatment

$$SME = \frac{1}{Q} \frac{N}{N_{max}} r \frac{C}{C_{max}} W$$



$$L_n(t) = [L_0 - L_\infty] \exp(-k' SME) + L_\infty$$

Where:

Q is the mass feed rate,

N is the screw speed,

N_{max} the maximum screw speed (680 rpm),

r the motor efficiency (0.93),

C the torque,

C_{max} the maximum torque,

W the nominal power (9.2 kW)

Materials and methods

❑ Materials

ϵ -caprolactone $\rightarrow T_{melting} : 60^{\circ}\text{C} - M_w : 80.000 \text{ g/mol}$

Hemp bast fibre 20 % (w/v) \rightarrow 2 cm length on average



❑ Extrusion

Laboratory scale twin-screw extruder (Clextral BC 21)



❑ Trials

Compounding caprolactone / fibres in different conditions

Analyze of fiber L/W in compounds

Analyze of mechanical properties Young modulus and Yield_{\max} (tensile tests)

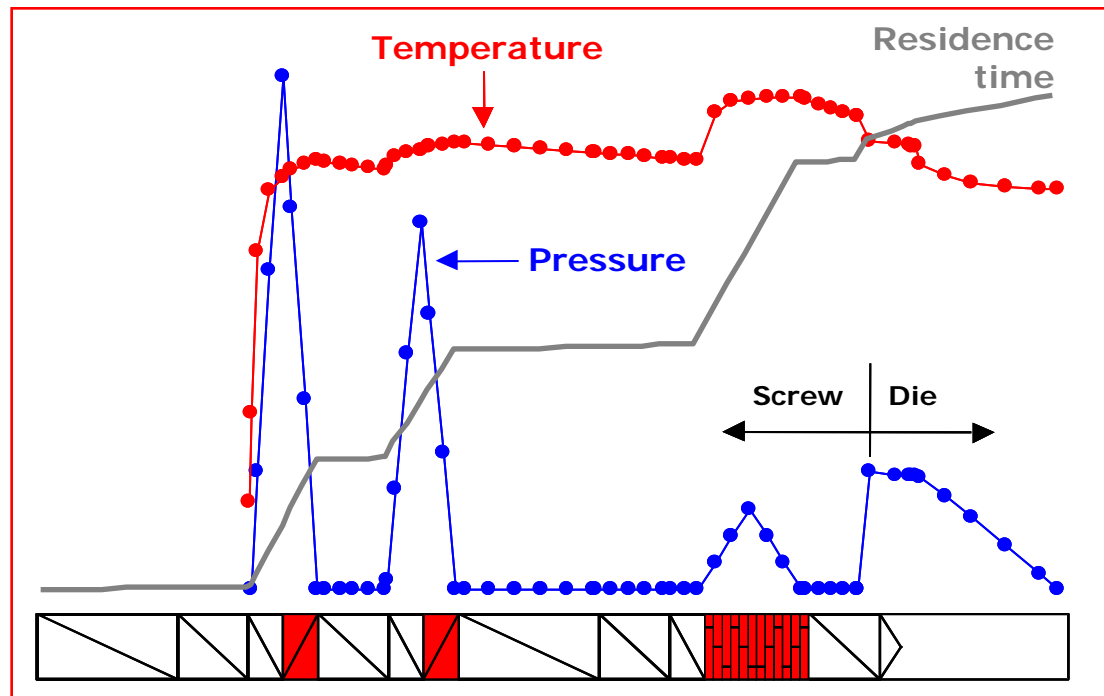
❑ Simulation/modeling of the fibre' thermomechanical history

Twin-screw extrusion modeling software

- LUDOVIC[©] : global model based on mass and thermal balance equations allowing to calculate the principal flow parameters

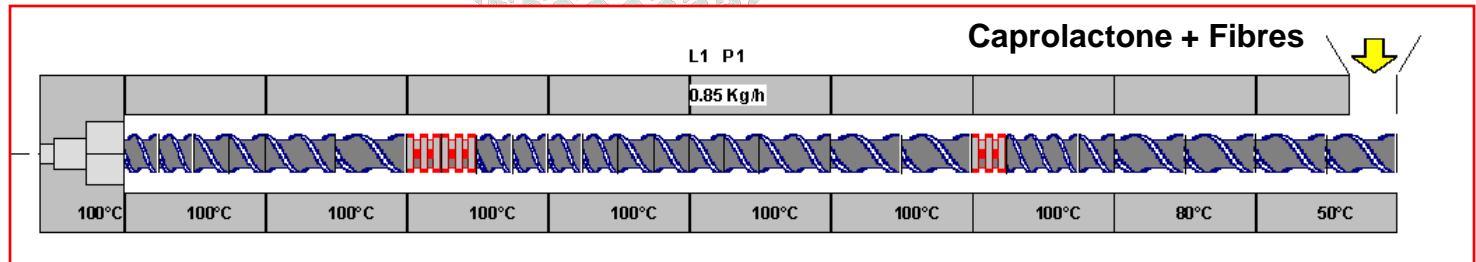
Vergnes et al., *Polym. Eng. Sci.*, 1998 <http://www.siconsultants.com>

- Pressure
- Shear rate
- Temperature
- Residence time
- Filling ratio...



Extrusions set up

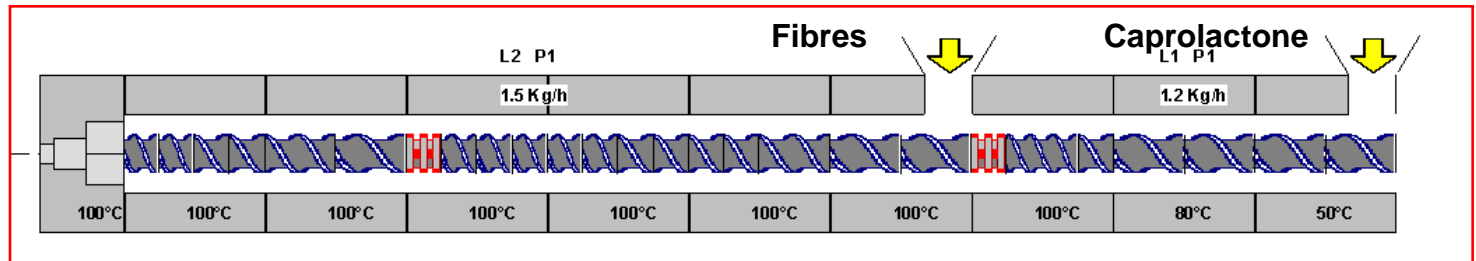
Screw profile 1 'hard'



Study of formulation (at constant screw speed 250 rpm and feed rate 0.85 kg/h)

- Influence of fibre water content: 9 – 14 – 23 (%)
- Influence of temperature: 100 – 140 (°C)

Screw profile 2 'soft'



Study of extrusion conditions (at constant humidity 50% and temperature 100°C)

- Influence of screw speed: 100 - 200 - 300 - 400 (rpm)
- Influence of feed rate: 0.85 – 1.5 (kg/h)

Characterization – Extruded hemp fibres

- Fibres morphology (L/W)

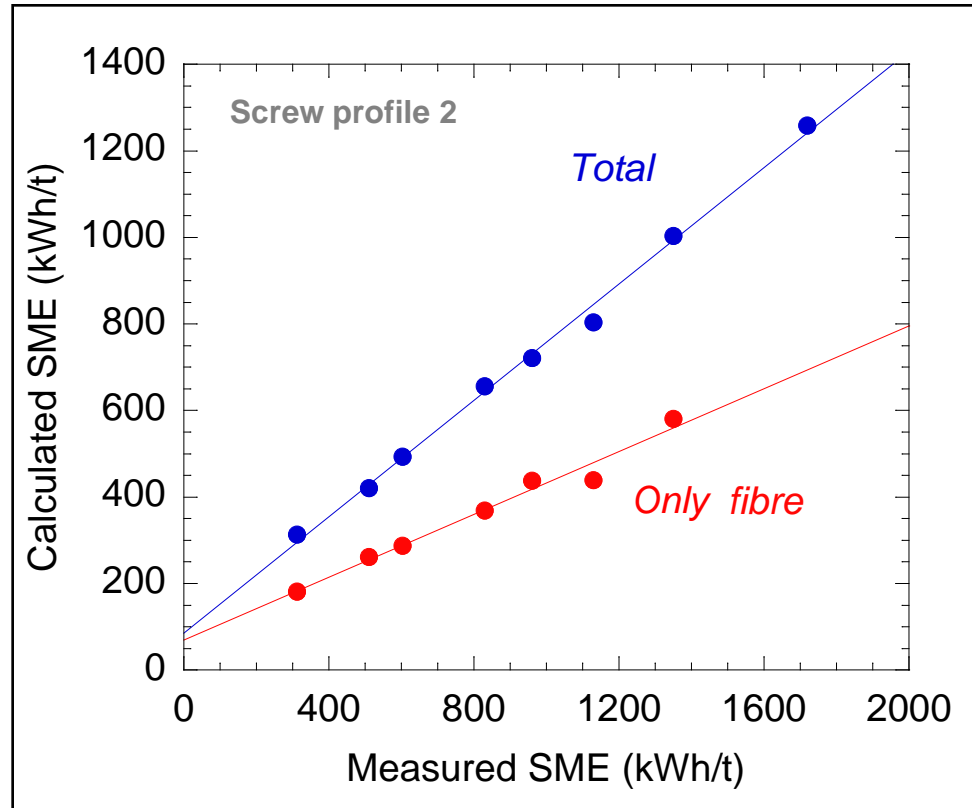


Optical Image Analysis



Results: effects of extrusion conditions

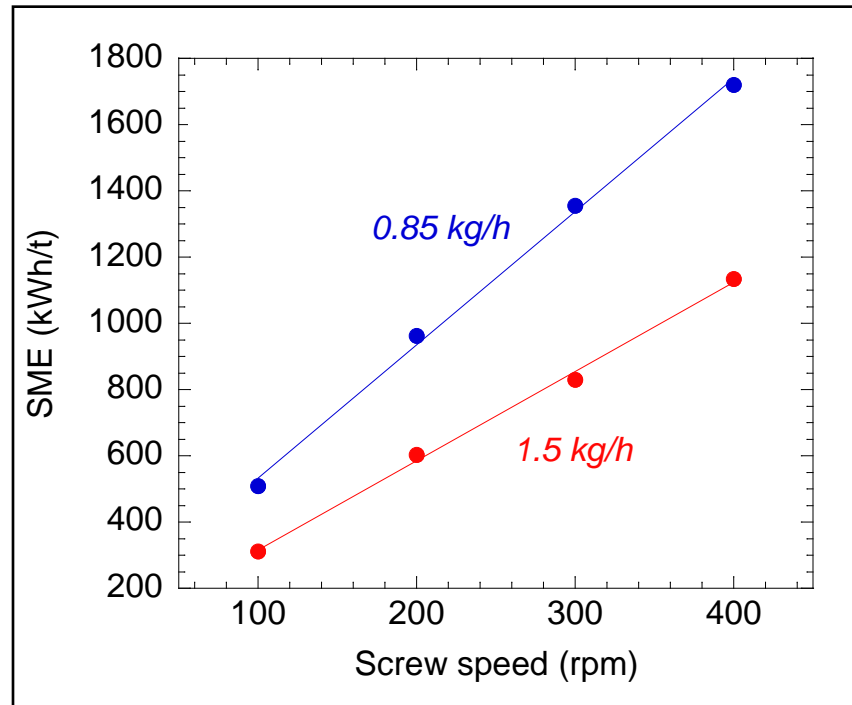
- Computation of the total SME and SME received by fibers



- Good fitting of experimental and simulated SME for total energy
- Slight underestimation of SME calculated by LUDOVIC[©]

Results: conditions of twin-screw extrusion

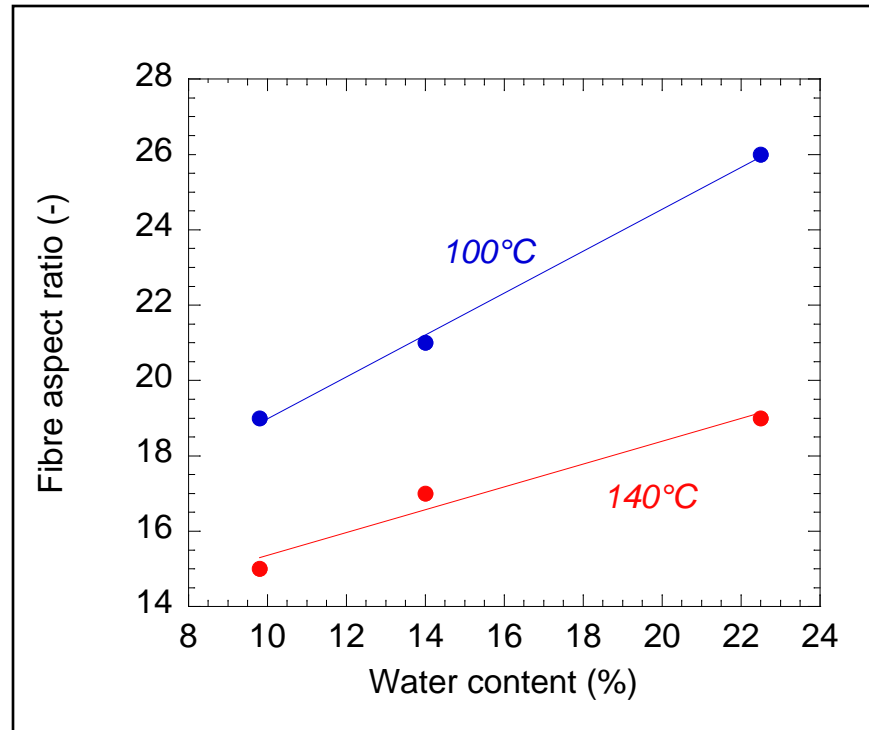
- Effects of screw speed and feed rate (profile 2)



- SME ↗ if screw speed ↗
- SME ↘ if feed rate ↗

Results: characterization of fibres

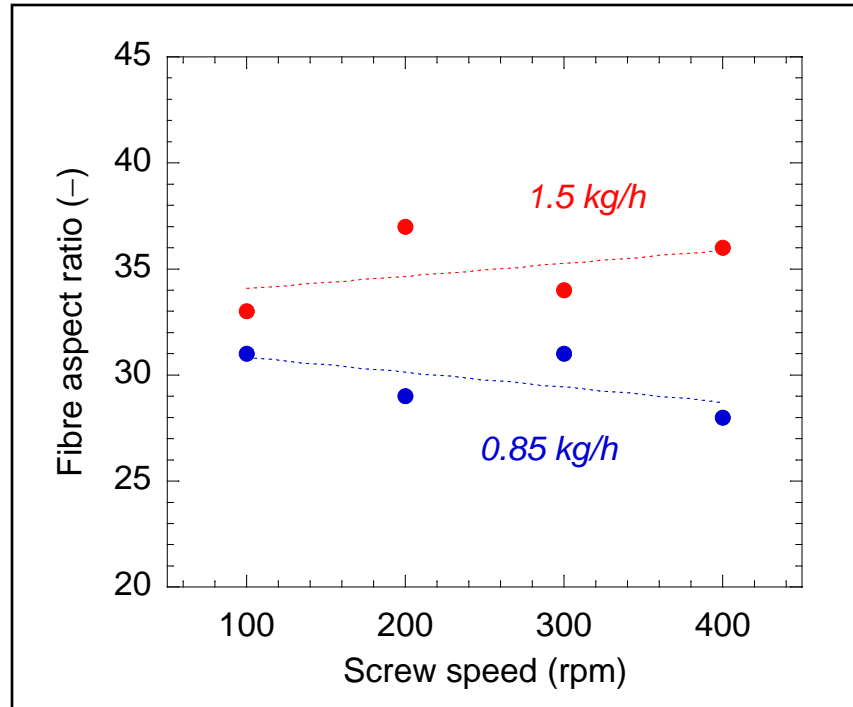
- Effects of formulation (profile 1, 250 rpm, 0.85 kg/h)



- Fibres water content ↗ → L/W ↗
- Extrusion temperature ↗ → L/W ↘

Results: characterization of fibres

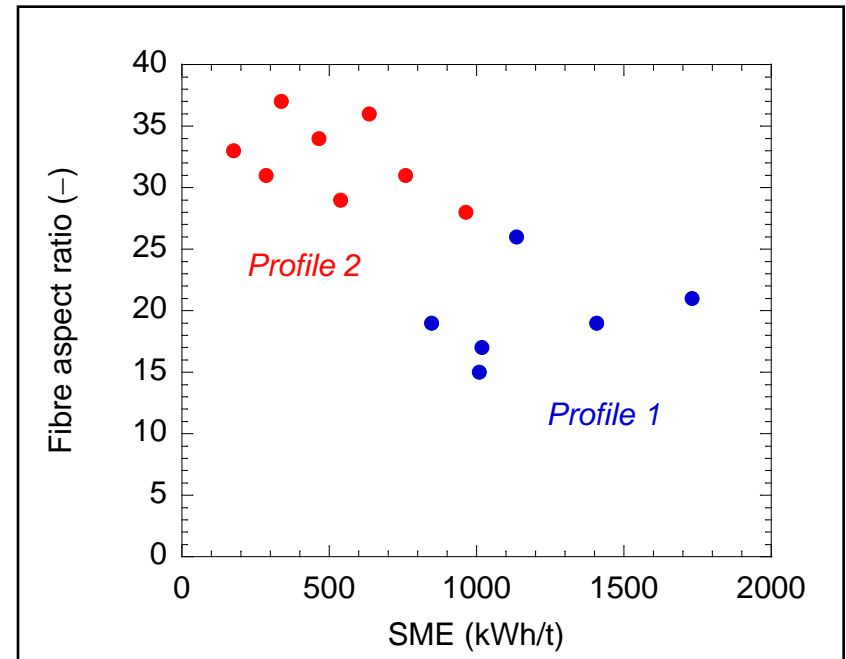
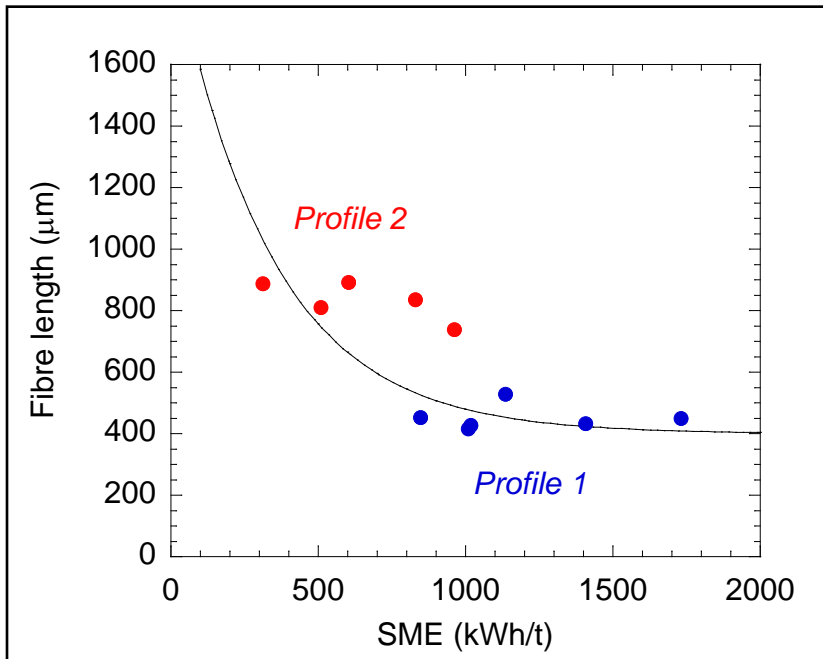
- Effects of extrusion conditions (profile 2)



- Screw speed ↗ → $L/W \approx$
- Feed rate ↗ → $L/W \nearrow$

Results: influence of extrusion conditions

○ Effects of SME



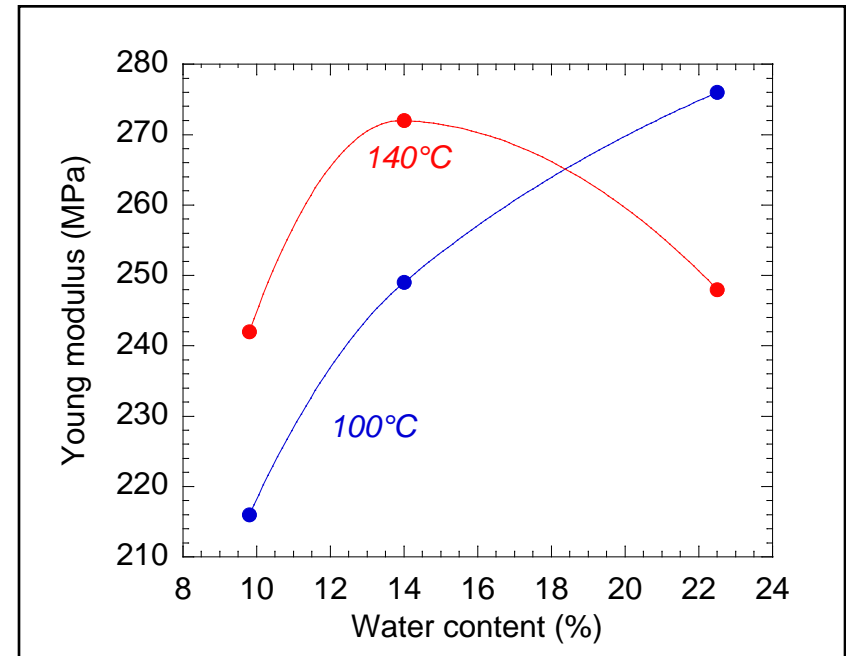
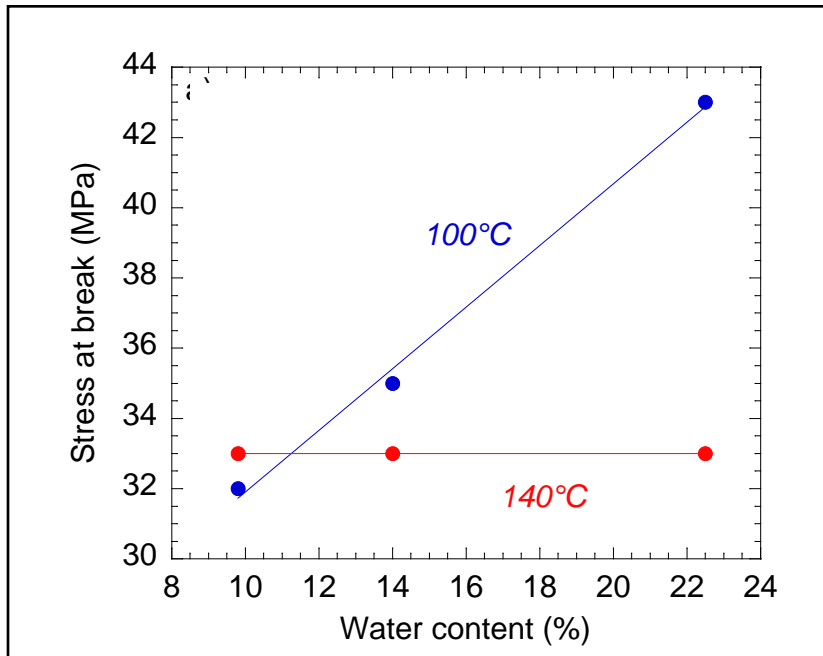
□ $L = L_{\infty} + (L_0 - L_{\infty}) \exp(-K \cdot SME)$
□ $L_0 = 2000 \text{ mm}, L_{\infty} = 400 \text{ mm}$ and $K = 0.003 \text{ (kWh/t)}^{-1}$

□ L and L/W \searrow with SME

□ L and L/W \searrow with profile 1

Results: mechanical properties

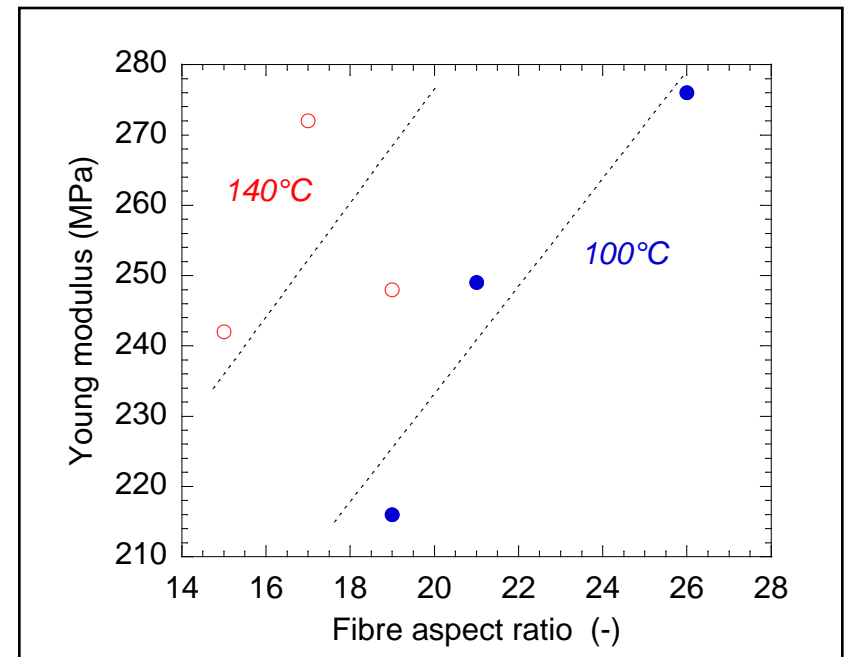
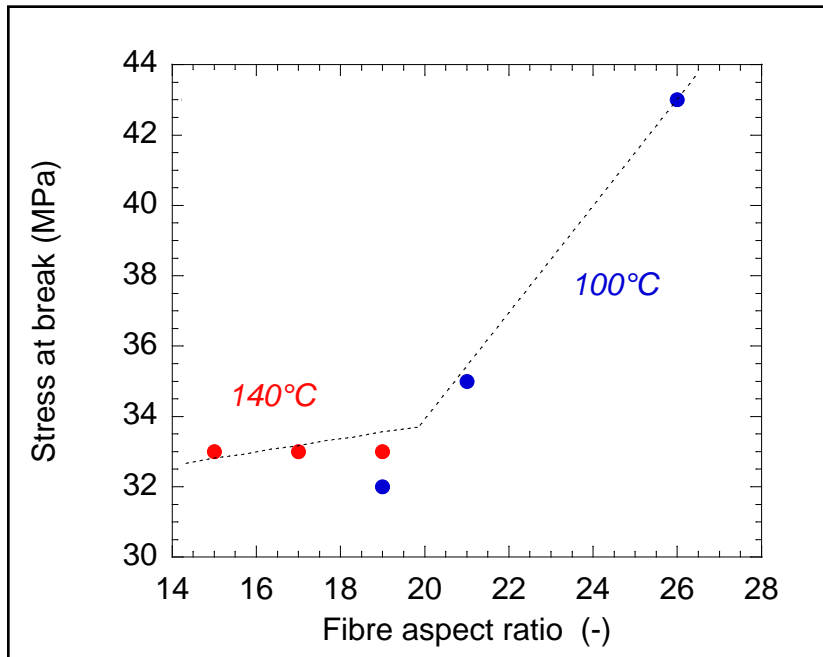
- Effects of formulation (profile 1, 250 rpm, 0.85 kg/h)



- Water content \nearrow \rightarrow Stress and Young modulus \nearrow at 100°C
- Different behavior at 140°C

Results: mechanical properties

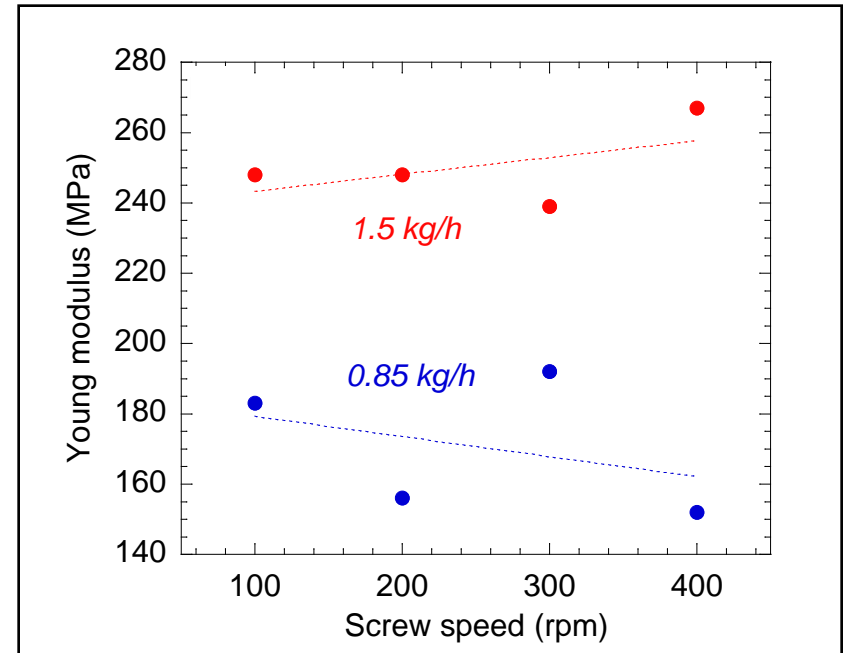
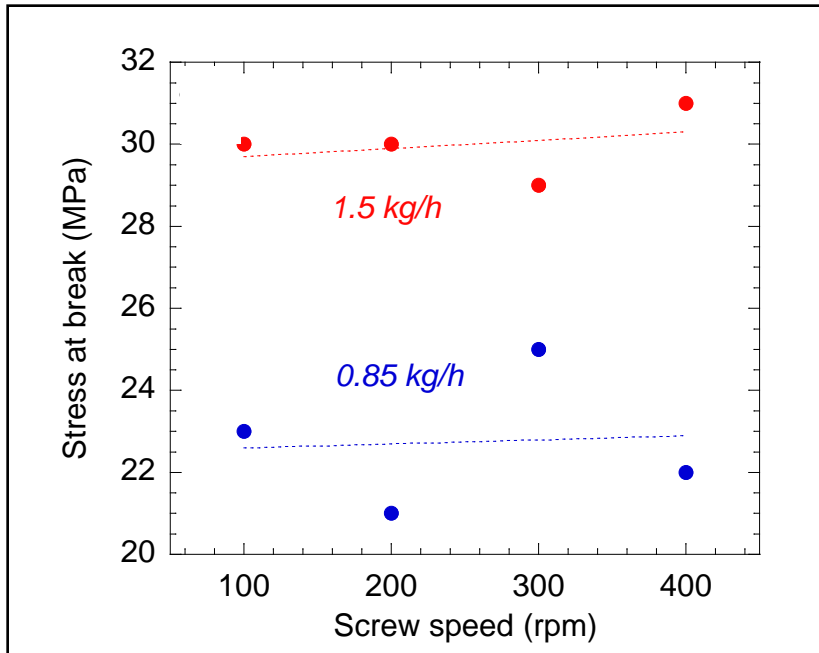
- Effects of fiber aspect ratio (profile 1, 250 rpm, 0.85 kg/h)



- Aspect ratio ↗ → Stress and Young modulus ↗
- Influence of temperature

Results: mechanical properties

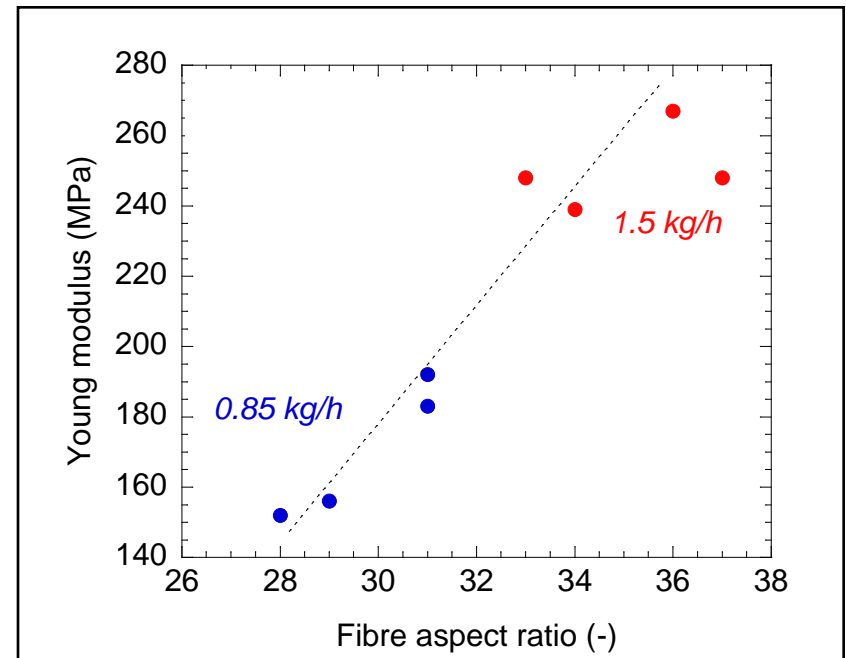
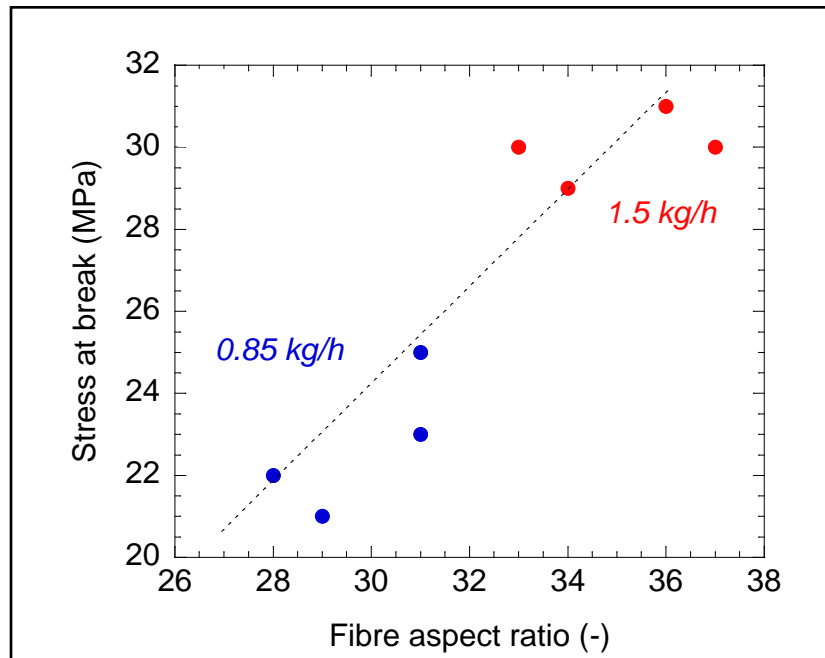
- Effects of extrusion conditions (profile 2)



- Low or no effect of screw speed
- Feed rate ↗ → Stress and Young modulus ↗

Results: mechanical properties

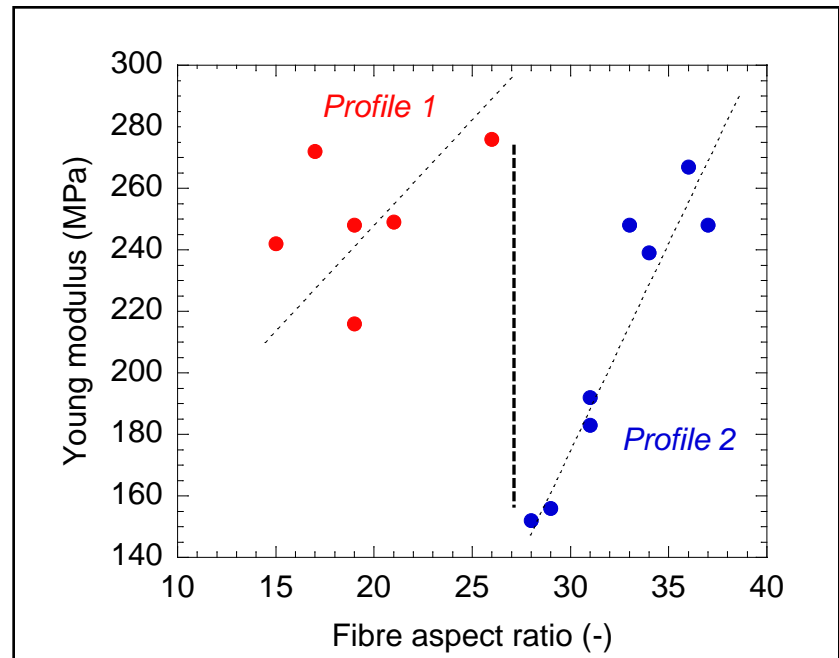
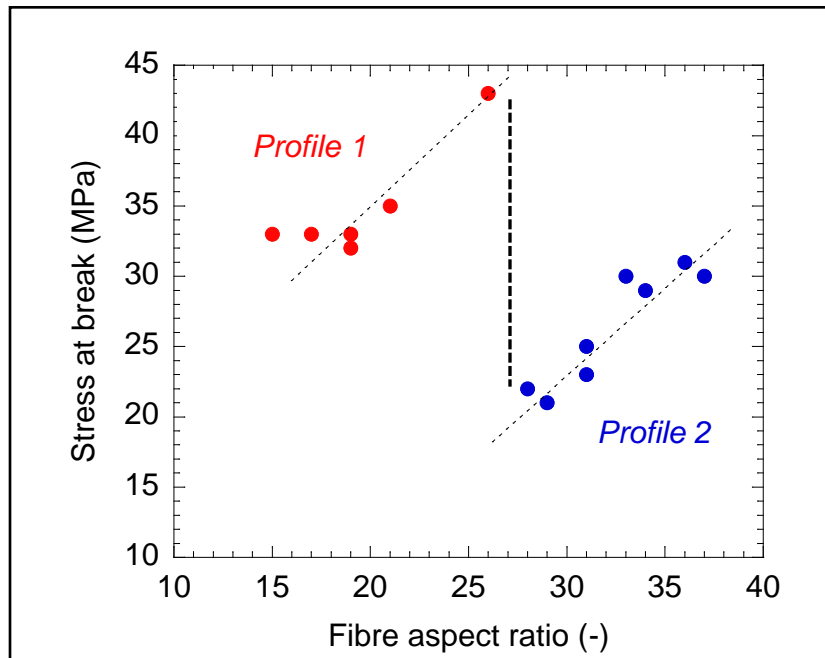
- Effects of fibre aspect ratio (profile 2)



- Mechanical properties (stress at break and Young modulus) are affected by L/W

Results: properties – morphology correlation

- Effects of fibre aspect ratio (profiles 1 and 2)



- No identical properties between screw profiles 1 & 2
- Problem: at identical L/W ratio → ≠ material properties

Is it possible to control the defibring in order to enhance the mechanical properties of composites?

- Yes, partial control of the defibring can be done and useful for mechanical properties enhancement
 - Fiber defibring depends on extrusion conditions (rotation speed and feed rate) through SME
 - To favour \nearrow L/W: twin-screw extrusion at high feed rate (low SME) as for glass fiber (limit breakages)
- No simple relation between L/W and mechanical properties: other influences (size and fibre adhesion...)

Contacts:

Physical and chemical transformations of lignocelluloses (fibres and composites):

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Engineering of Agricultural Products