KU LEUVEN

MATERIALS ENGINEERING

Composite Materials Group



Improving the Durability of Bio-based composites

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Main challenges for Bio-based Composites

STRENGTHS	WEAKNESSES
Renewability	Moisture sensitivity / **Durability**
Low Carbon footprint	Variability?
Moderate cost	Not-optimized interfaces
Low abrasiveness	Thermal degradation
Healthier?	Non-linear stress-strain behaviour ?
Good specific mechanical properties (longitudinally)	Low transverse and shear properties
Acoustic and vibrational damping	Water during processing
Low CTE	Not optimized fibre preforms
Natural look and feel	Extraction without damage

Ways to improve Durability, particularly moisture sensitivity

1) Choose less moisture sensitive natural fibres, e.g. bamboo fibre
2) Lower the Equilibrium Moisture Content (EMC) of the fibres; chemical treatment
3) Reduce the swelling of the fibres; chemical treatment
4) Turn water inside the fibres into an advantage, process with water inside the fibres; pre-swollen fibres
5) Coatings (of fibres or composite)

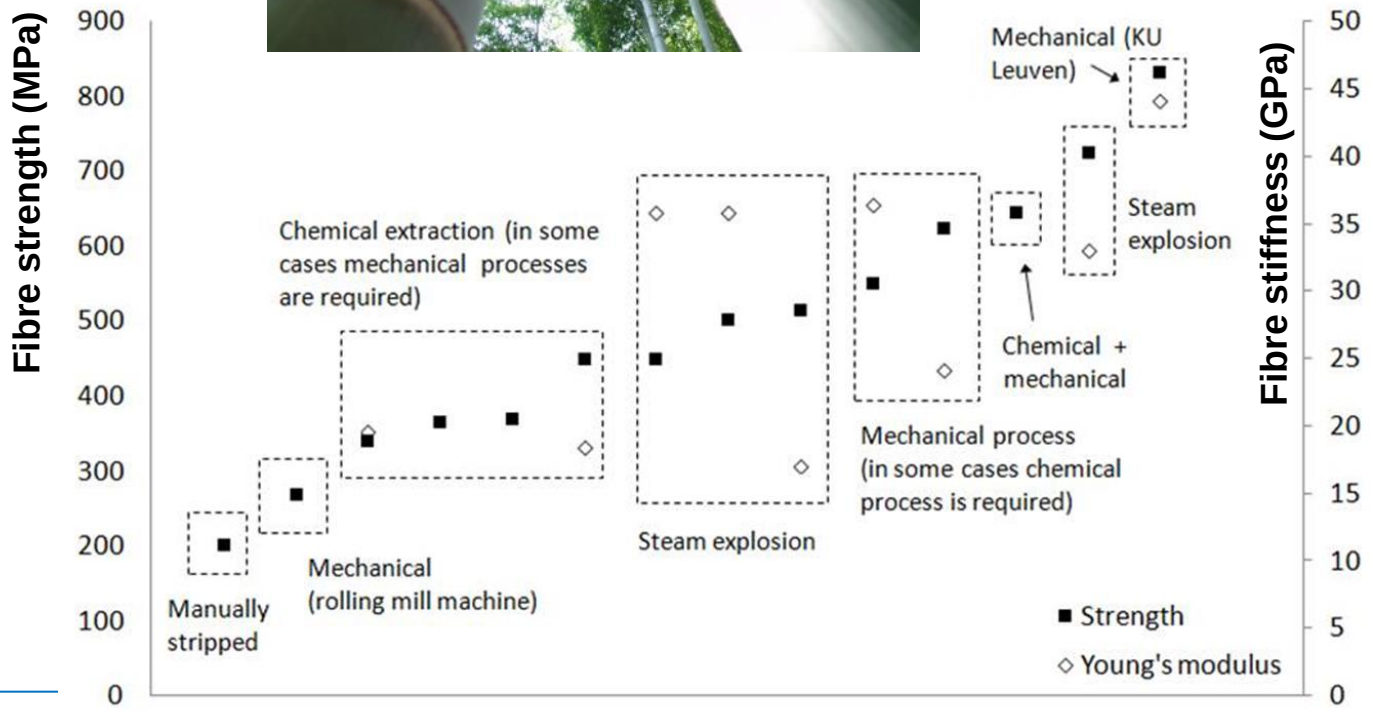
Bamboo Fibre Extraction



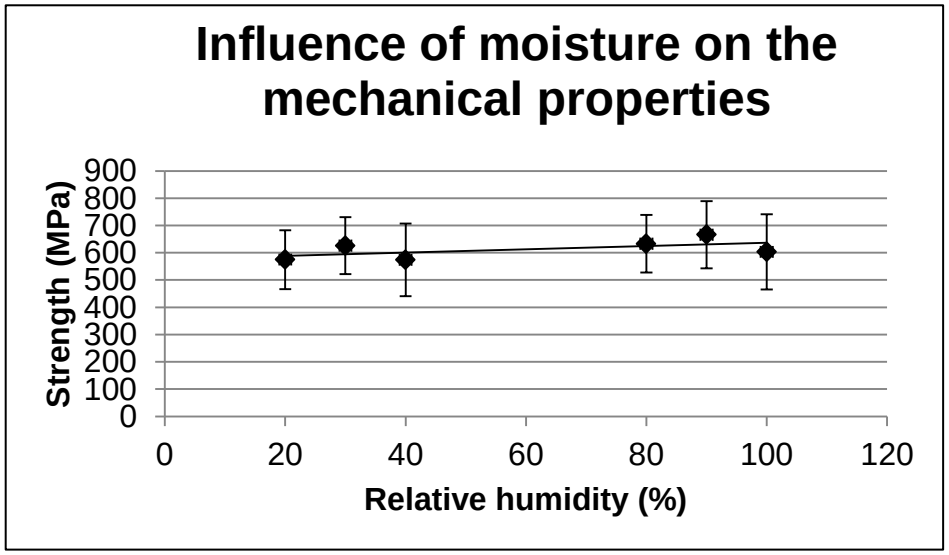


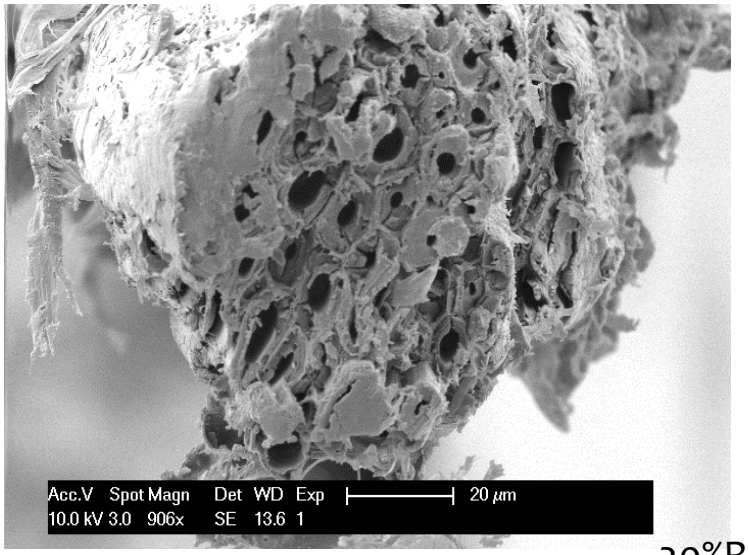




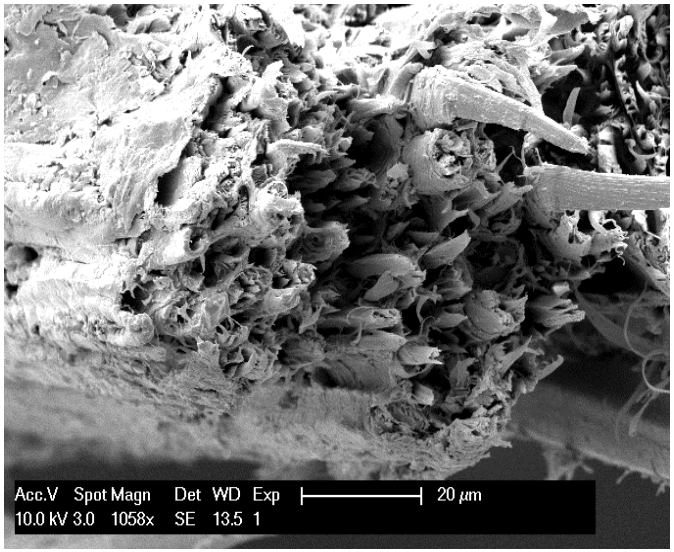


Moisture sensitivity of technical bamboo fibres

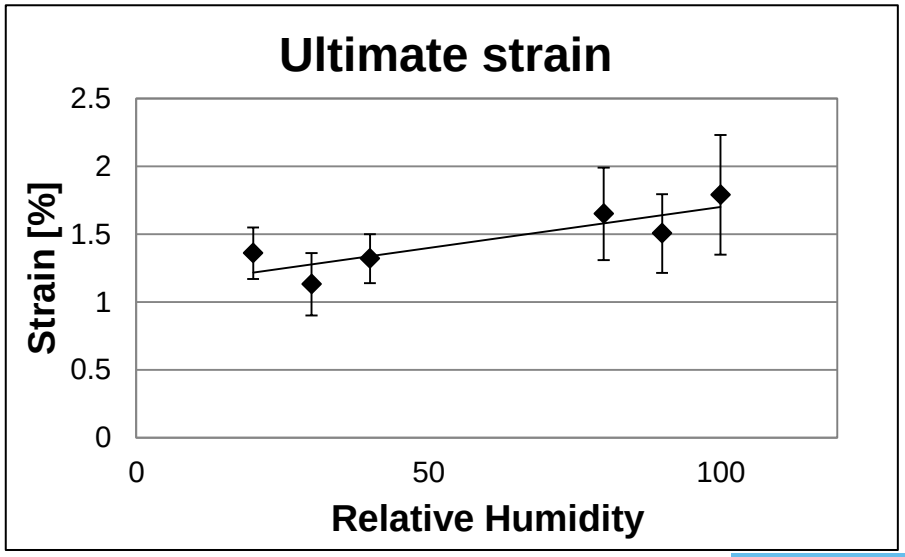




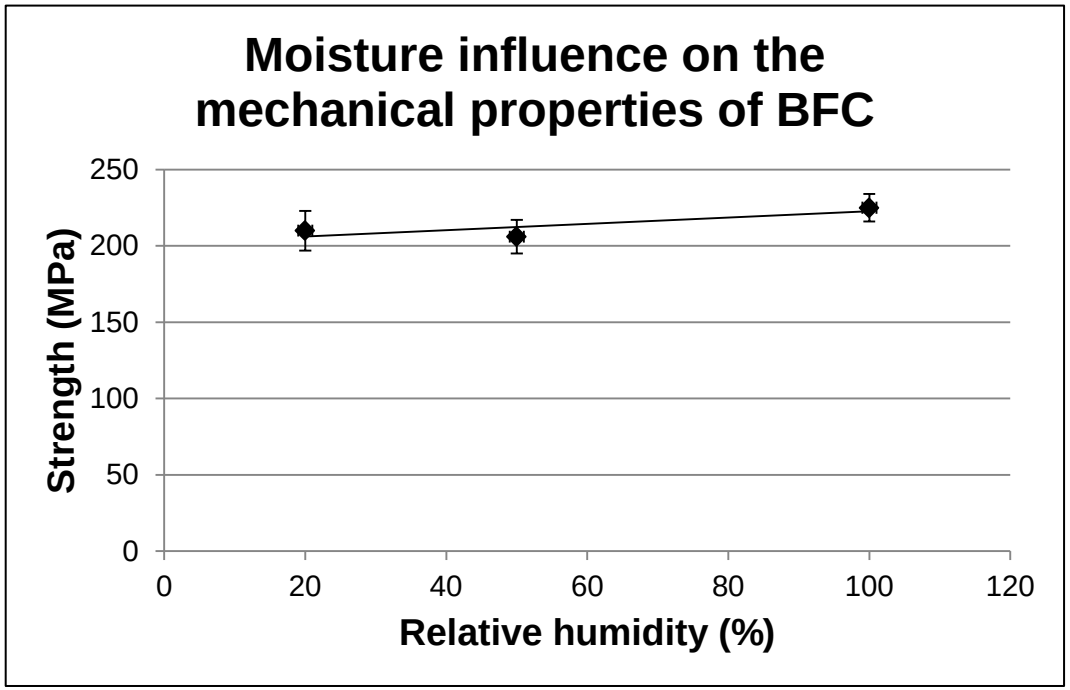
20%RH



80%RH



Moisture Sensitivity of Bamboo Composites



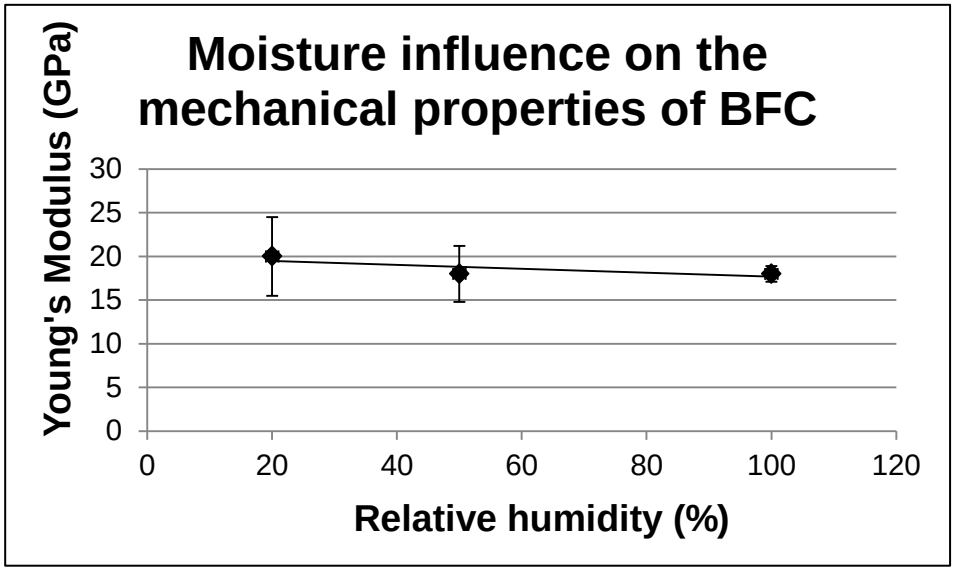


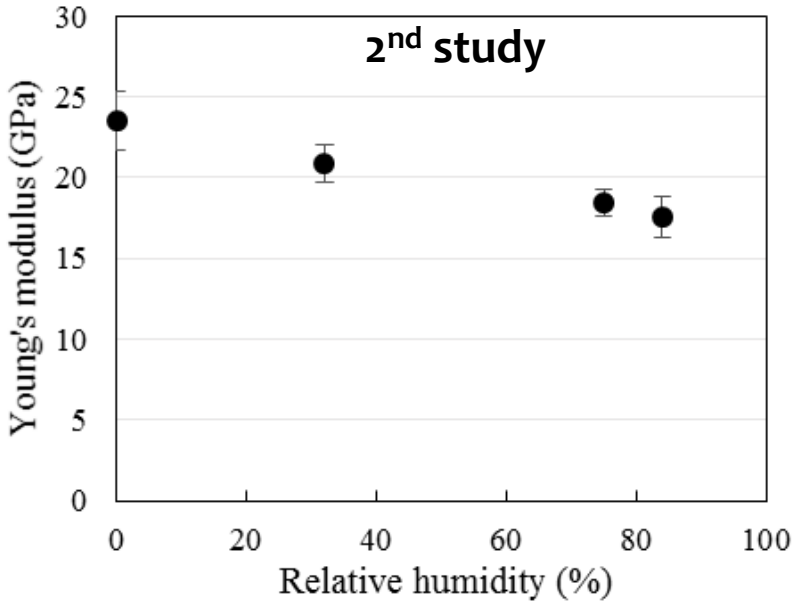


(epoxy matrix)

Moisture sensitivity of bamboo fibre composites

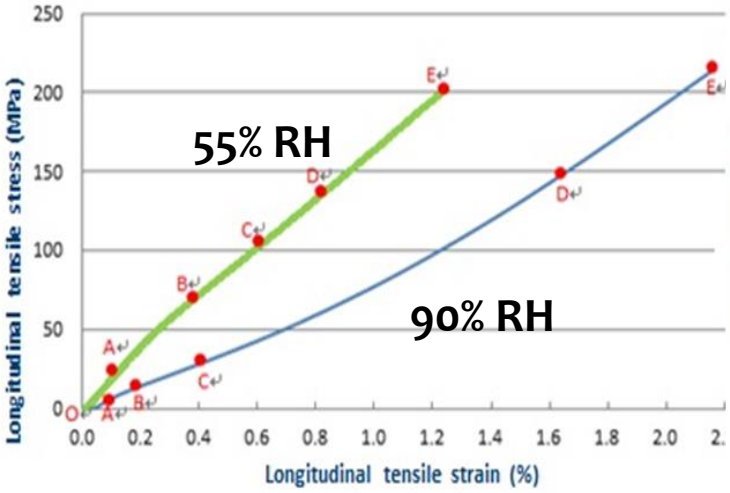
Modulus does not decrease much!

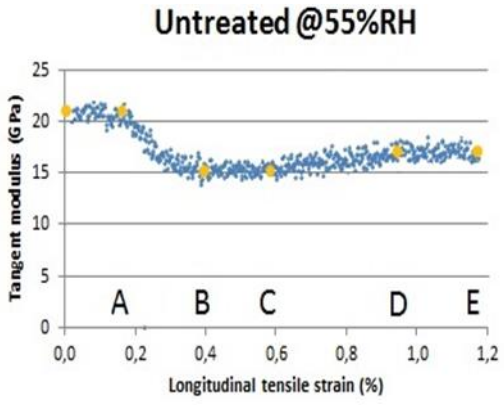


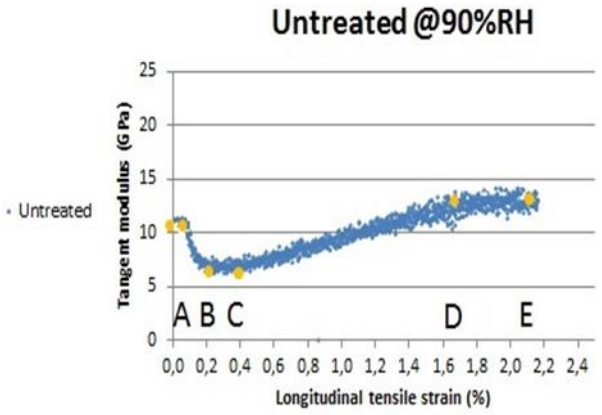


(Epoxy matrix, Vf 40%)

UD FLAX composite:





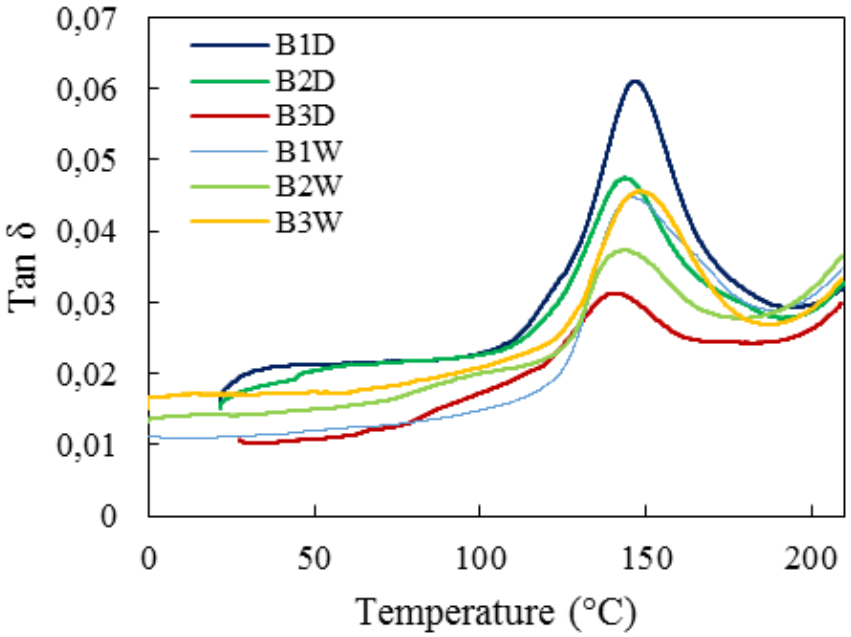


Why little drop in modulus at high humidity for BFC?

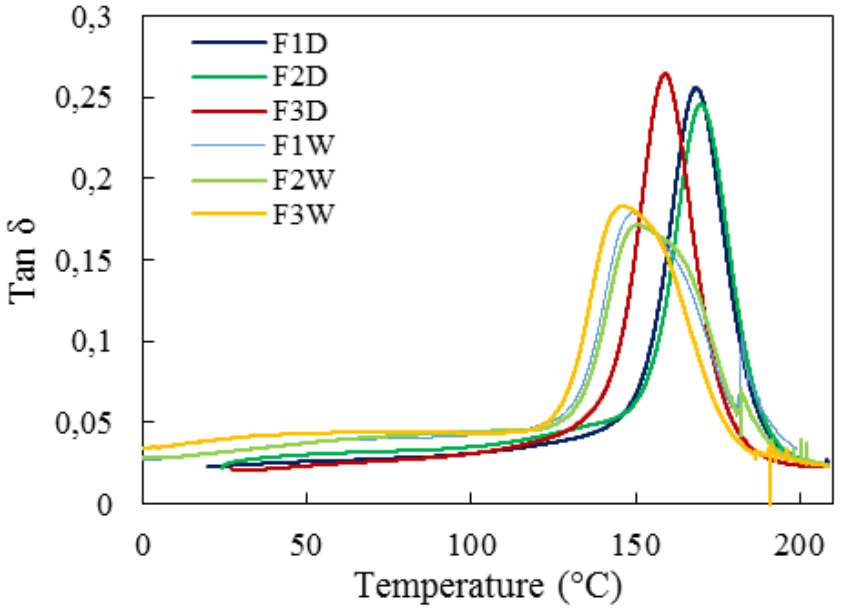
***Hypothesis*:** high lignin content of bamboo fibres ~ 30 wt%

DMA measurements for Tg:

Dry (0%RH) versus Wet (80%RH) BFC

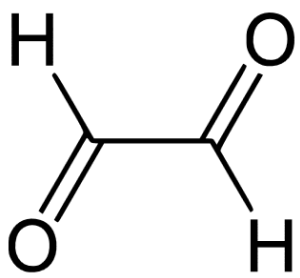


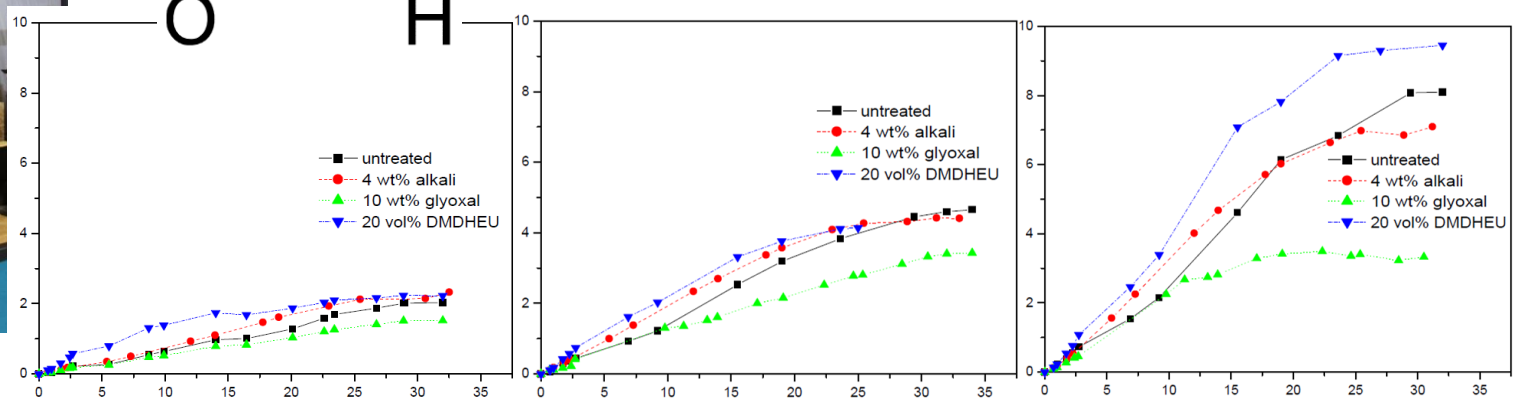
Dry versus Wet FFC



Flax fibre composites; treatments to lower the EMC



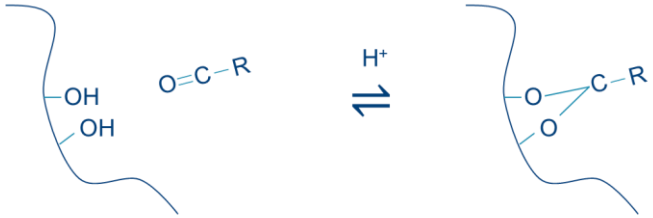




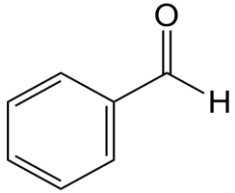
	50% RH	75% RH	90% RH
untreated	2%	4%	8%
4 wt% alkali	2%	4%	7%
10 wt% glyoxal	1.5%	4%	3.5%
20 vol% DMDHEU	2%	3%	10%

Aldehydes: A “new” class of treatments

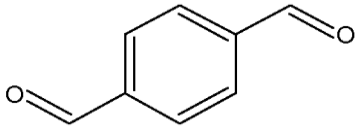
Acetal bond formation



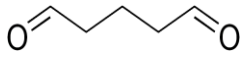
Benzaldehyde



Terephtaldehyde



Glutaraldehyde



Crosslinkers

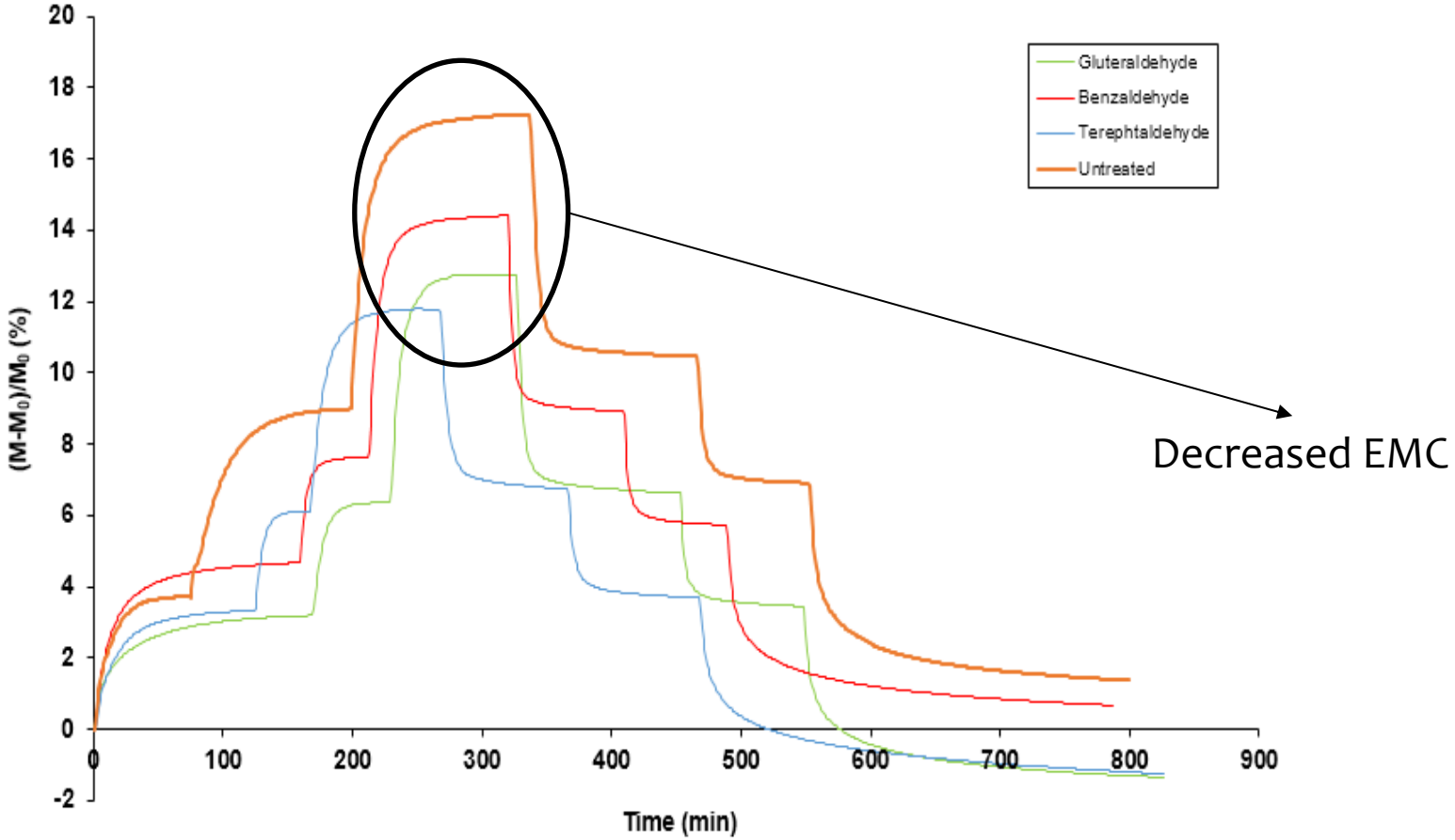
Conditions:

- 10 wt% in THF @ room temp.
- Fibres soaked for 10 min
- Dried in oven at 80°C

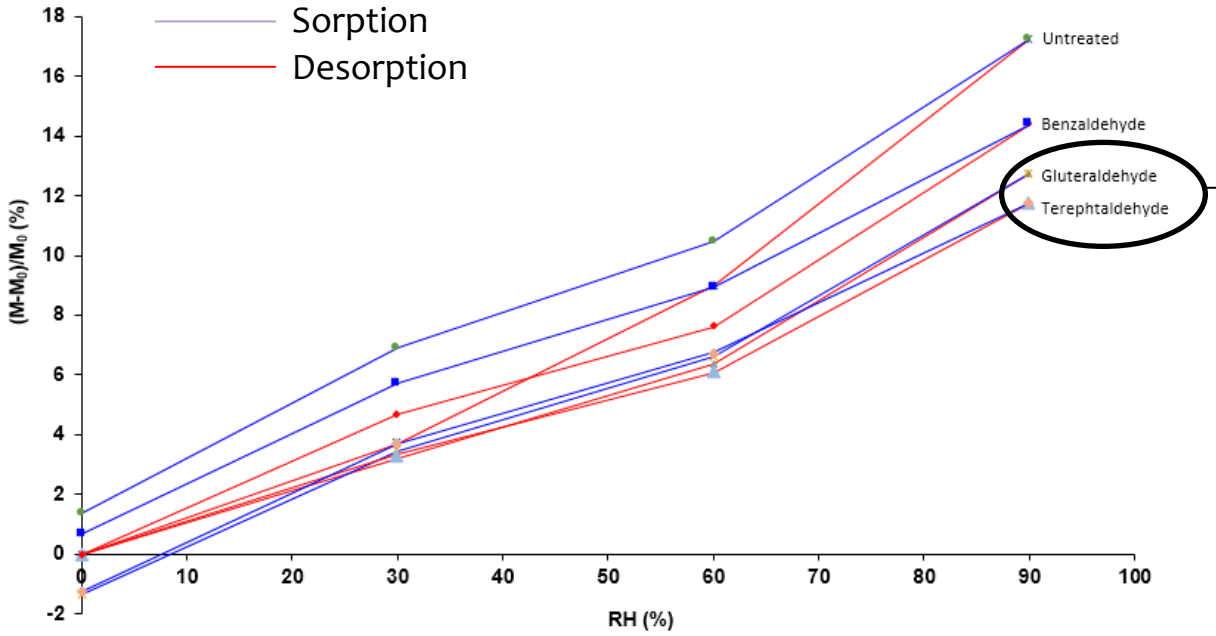
10

Dynamic vapour sorption – EMC

Di-aldehyde cross-linkers are more efficient



Dynamic vapour sorption – isotherms



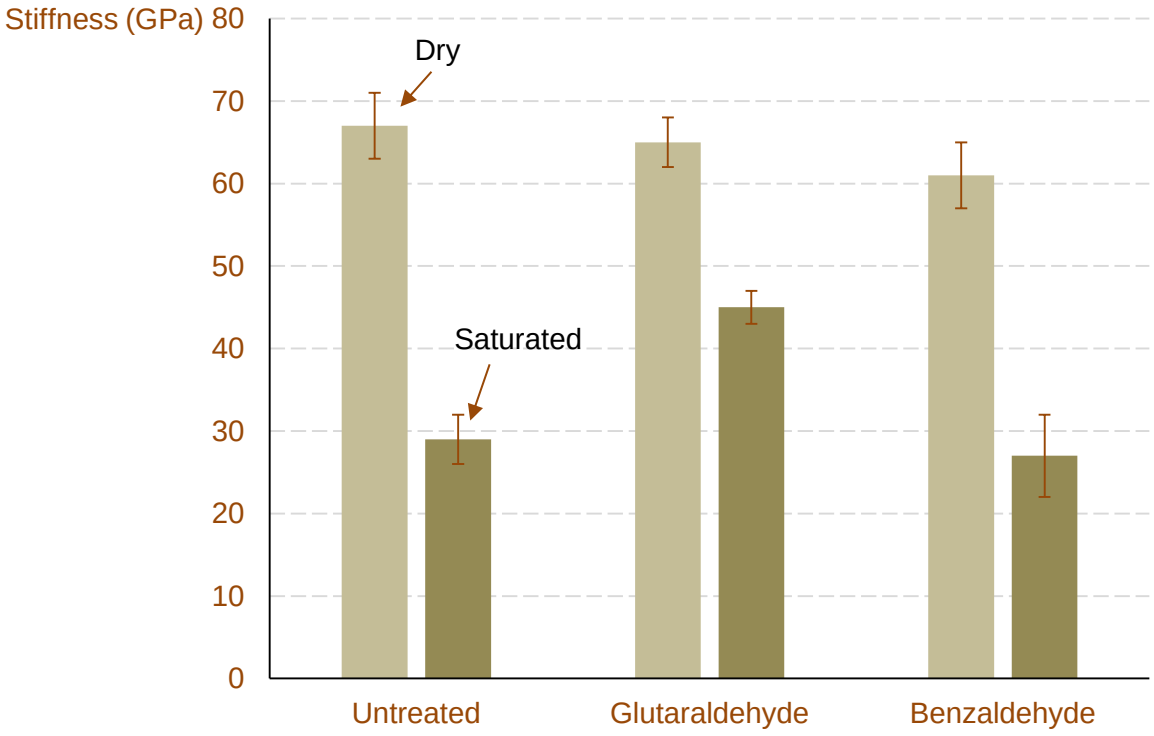
Hysteresis reduction

For cross-linkers

Fibre swelling is restricted ?

12

Backcalculated stiffness of UD treated fibre composites



Saturation @ 80%RH and 80°C

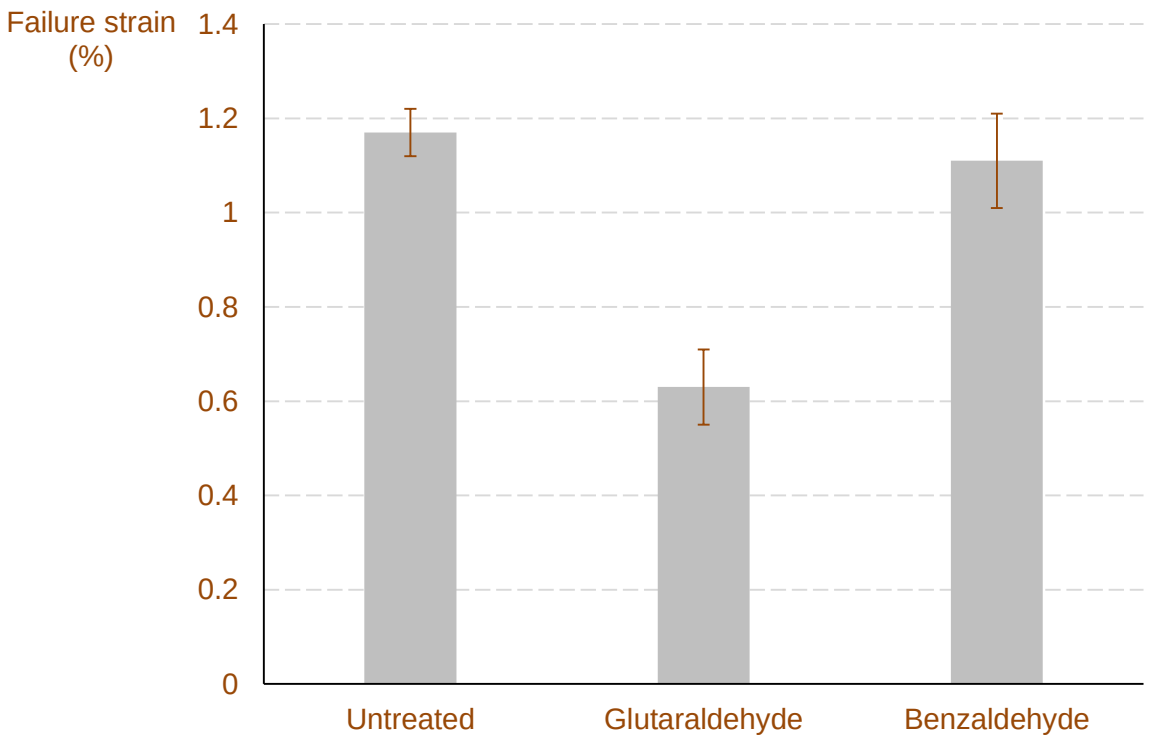
Stiffness decrease = 55% for untreated fibres

Only 30% decrease for gluteraldehyde → crosslinking partially counteracts plasticization due to water absorption

Benzaldehyde (not a crosslinker) does not have an effect on plasticization

13

Embrittlement due to crosslinking



Failure strain of the composites severely decreased by glutaraldehyde

Fibres are likely embrittled due to crosslinking

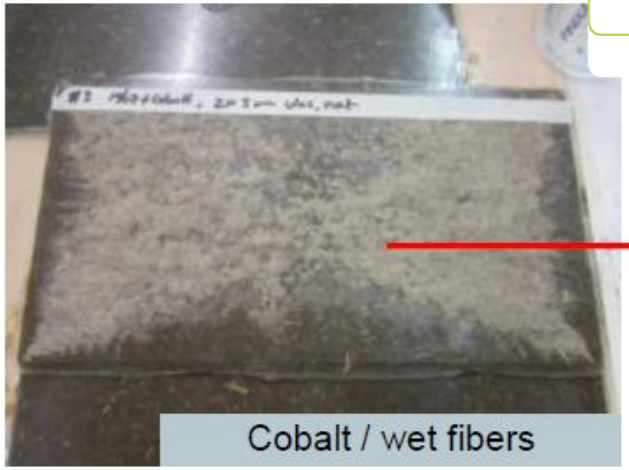
14

Next: non-dried natural fibres

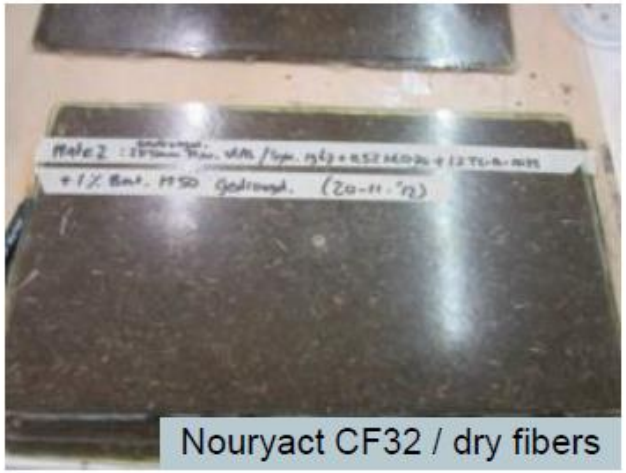
Novel water-insensitive curing systems for biofiber reinforced polyester composites

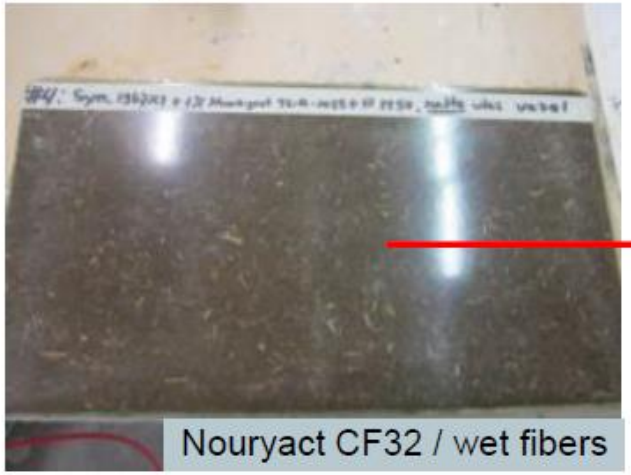
With Ssuchy partners Akzo Nobel and NPSP (Netherlands)


Cobalt / dry fibers


Cobalt / wet fibers

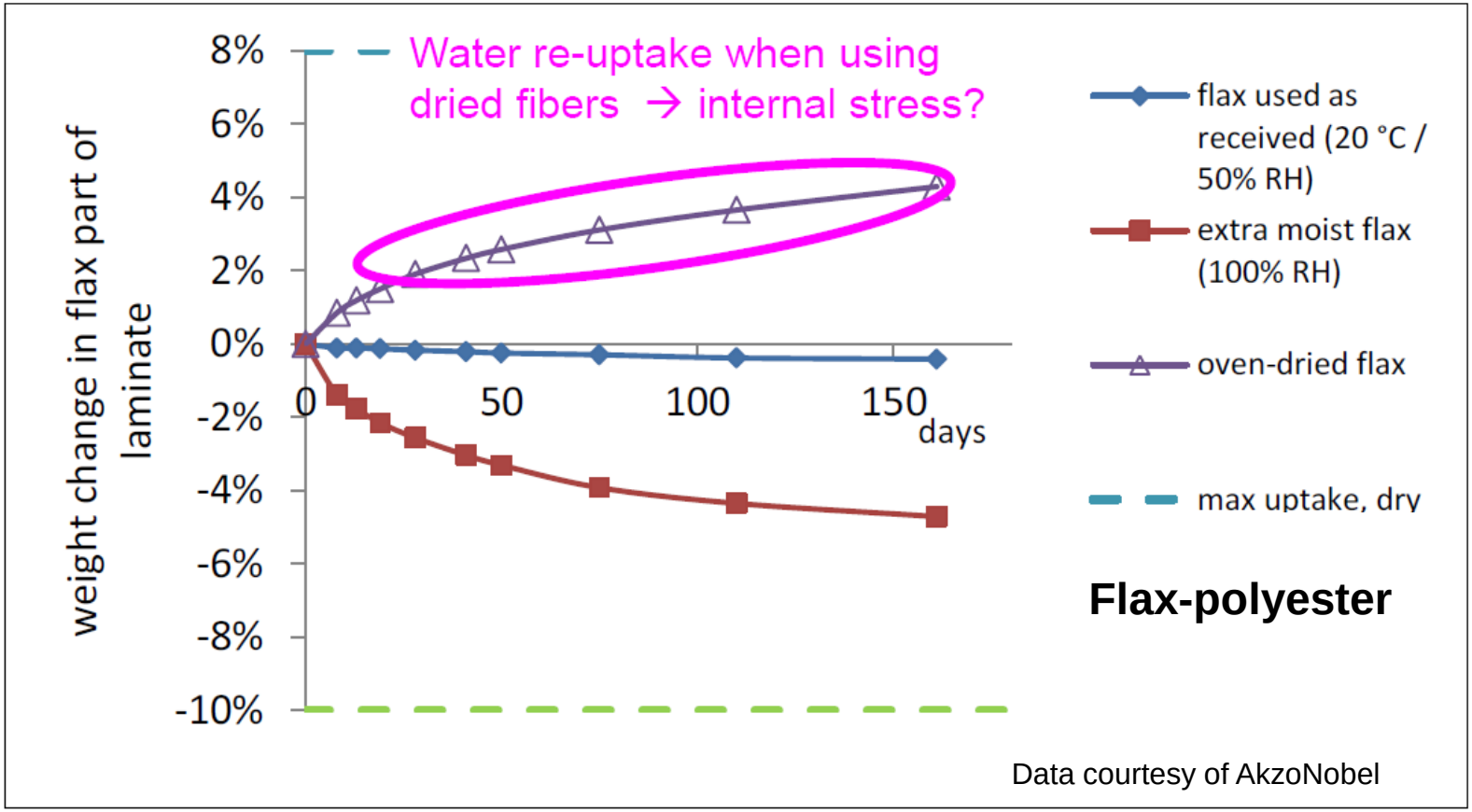
Hardly any cure


Nouryact CF32 / dry fibers


Nouryact CF32 / wet fibers

Excellent cure

New Perspective on Water Uptake

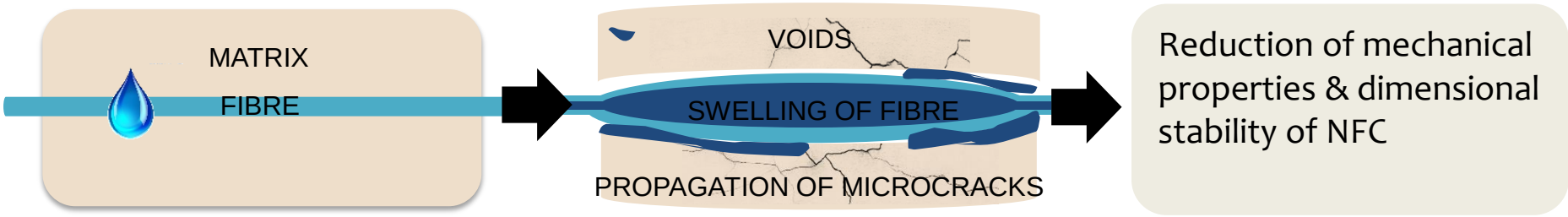


Long term average indoor conditions 20°C and 50%RH

Hypothesis: Durability better for damp natural fibres equilibrated at Equilibrium Moisture Content of average environment: pre-swelling

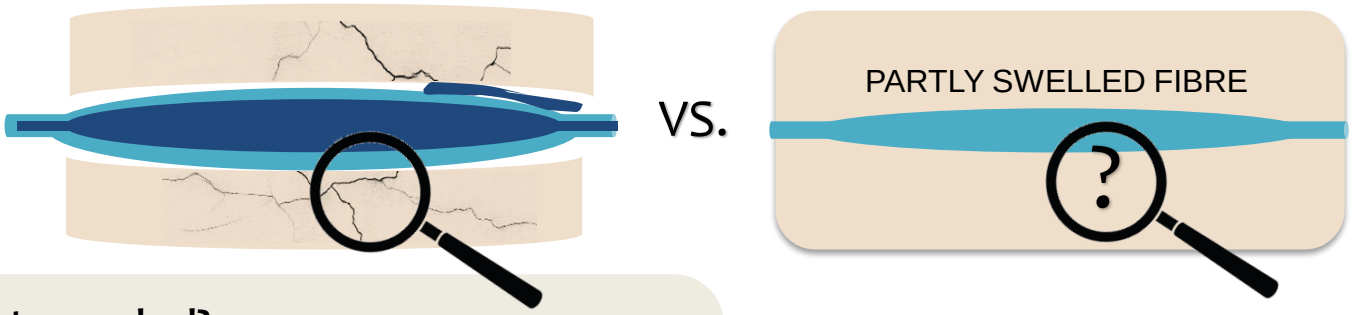
* Less swelling, shrinkage, cracking

EFFECTS OF MOISTURE SORPTION ON DURABILITY OF NATURAL FIBRE COMPOSITES



What's new in this study!

In this study we will use **undried fibre** as reinforcement in composites to take advantage of the moisture present in the fibre to enhance the water-resistant property of composites.



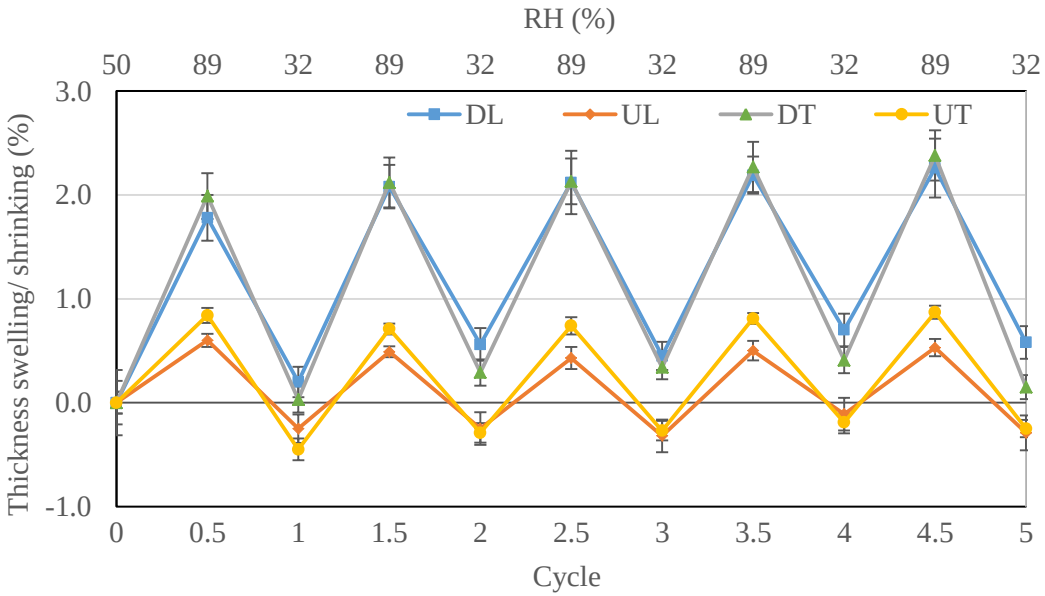
Less moisture sorbed?

Less microcracks?

Less voids?

Will there be less damage at the interface?

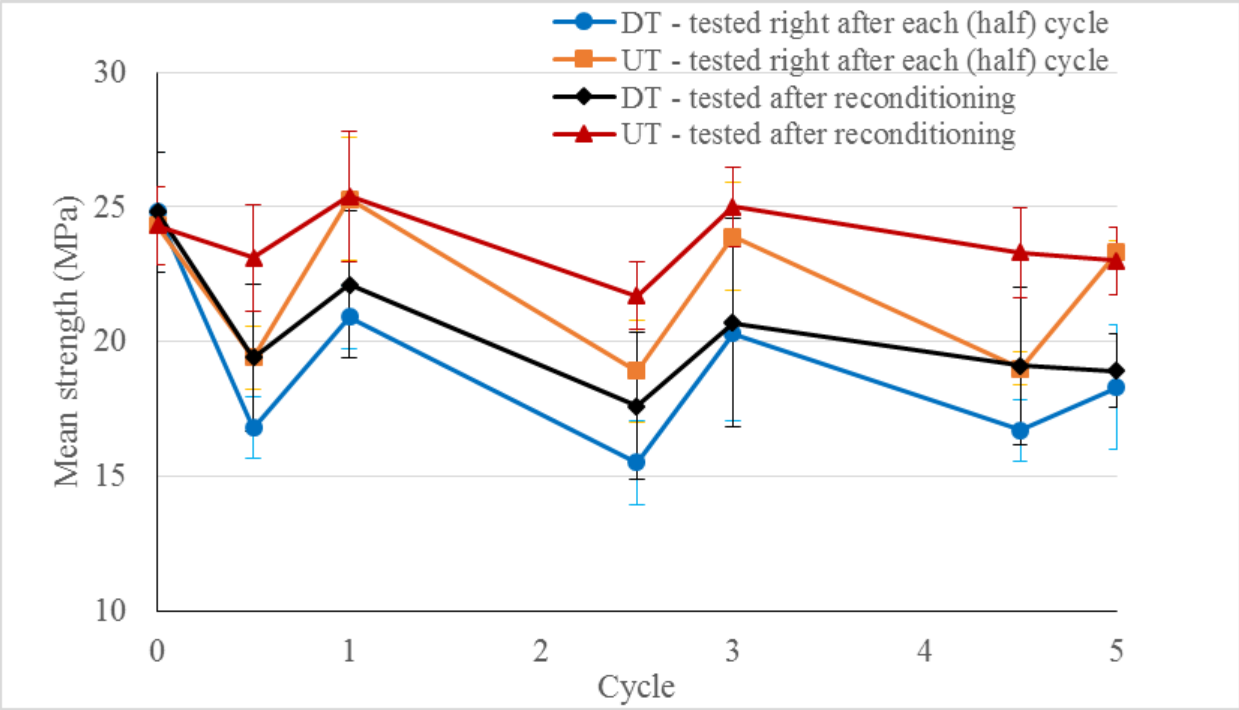
THICKNESS SWELLING/SHRINKING OF THE COMPOSITES



In the graph, the thickness of samples after preconditioning at 22 °C and 50% RH or prior to the artificial ageing experiment are shown as zero thickness swelling/shrinking.

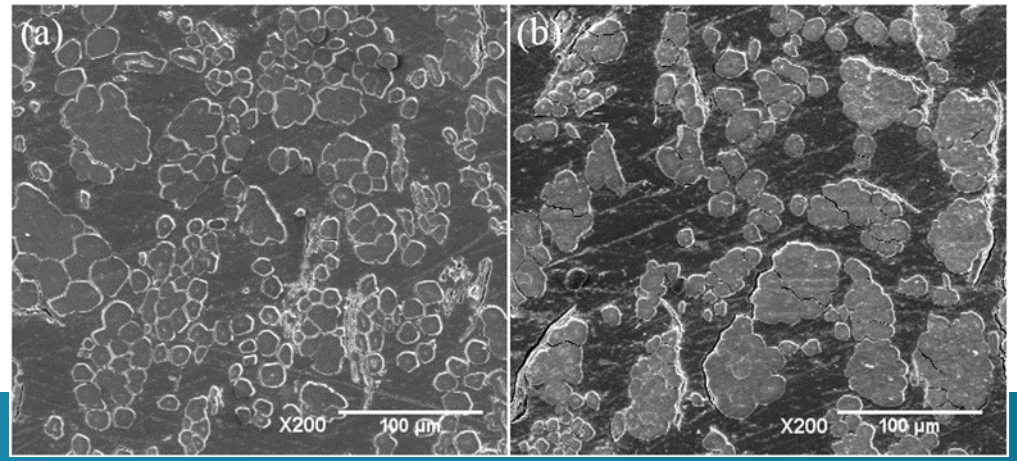
The degree of swelling and shrinking of UL and UT is lower compared to DL and DT

Do un-dried fibres have higher durability?



Images after half cycle at high humidity

UT **DT**



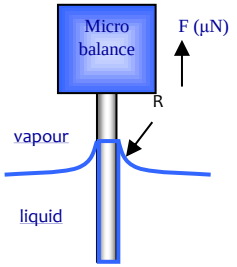
Already cracks for dried fibres, also inside bundles!

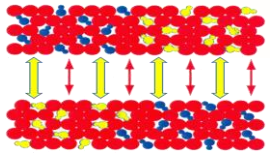
CMG expertise: Studying and improving the Fibre Matrix Interface; Durability enhancement

- An integrated physical-chemical-micromechanical approach

FibreNet

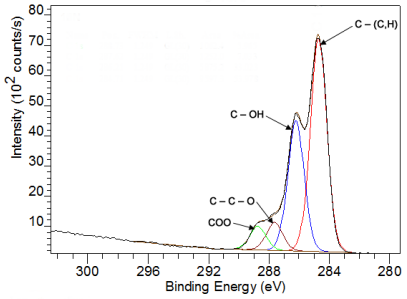
1) Physical characterization: surface energy components from contact angle measurements





$$\gamma_{L,i}(1+\cos\theta_i) = 2\left(\sqrt{\gamma_{L,i}^{LW}\gamma_S^{LW}} + \sqrt{\gamma_{L,i}^{+}\gamma_S^{-}} + \sqrt{\gamma_{L,i}^{-}\gamma_S^{+}}\right)$$

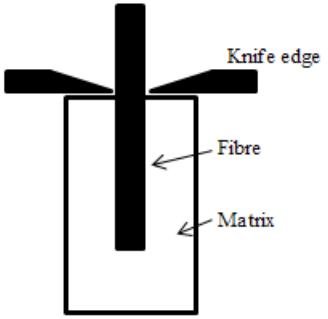
2) Surface chemistry: XPS, ToF-SIMS



X-ray photoelectron spectroscopy (XPS)

3) (Micro)-mechanical testing of adhesion

(single fibre pull-out & transverse UD 3-point bending)



Tools for selecting fibre treatment or matrix modification

For improvement of wetting and adhesion

Conclusions

1) Bamboo fibres loose much less stiffness in wet condition than flax fibres
2) Di-aldehyde treatments show significantly lower EMC for flax fibres, but embrittle the fibres
3) Making composites with undried, pre-swollen fibres, gives significant improvements in durability for flax fibre composites

   * Plus save energy and time for not having to dry

Acknowledgements:

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