

Evaluation of Resizing Carbon Fibers for Reuse in Thermoset Composite Manufacturing: a study of individual fibers



MORPHO project workshop

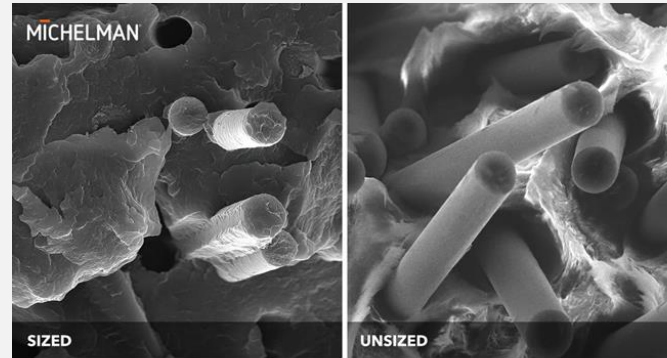
Novembre 26th

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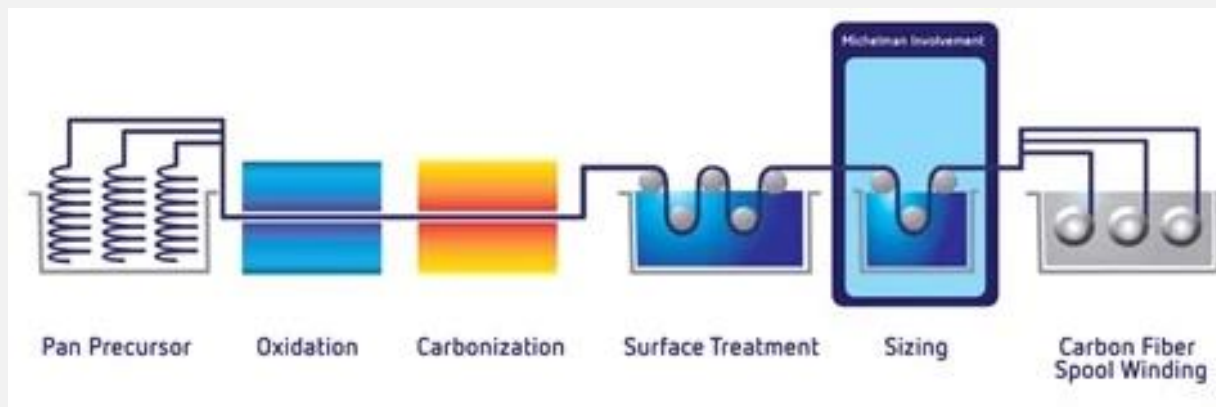


Background

What is a Fibre Sizing ?



<https://www.michelman.com/markets/reinforced-plastic-composites/fiber-sizing/>



- ✓ A thin, homogenous coating
- ✓ To protect the filaments
- ✓ Improved adhesion between the fiber and the matrix
- ✓ Increase the mechanical properties, chemical or water resistance, and thermal stability

Background

Recycled carbon fibre (rCF)

Mechanical processing
After tearing



Solvolysis



Pyrolysis



Problem:

During recycling the original sizing in most cases is removed

- Adhesions problems with matrix
- Bad composite quality (low mechanical properties)

Solution :

Resizing, a new coating on the fibres

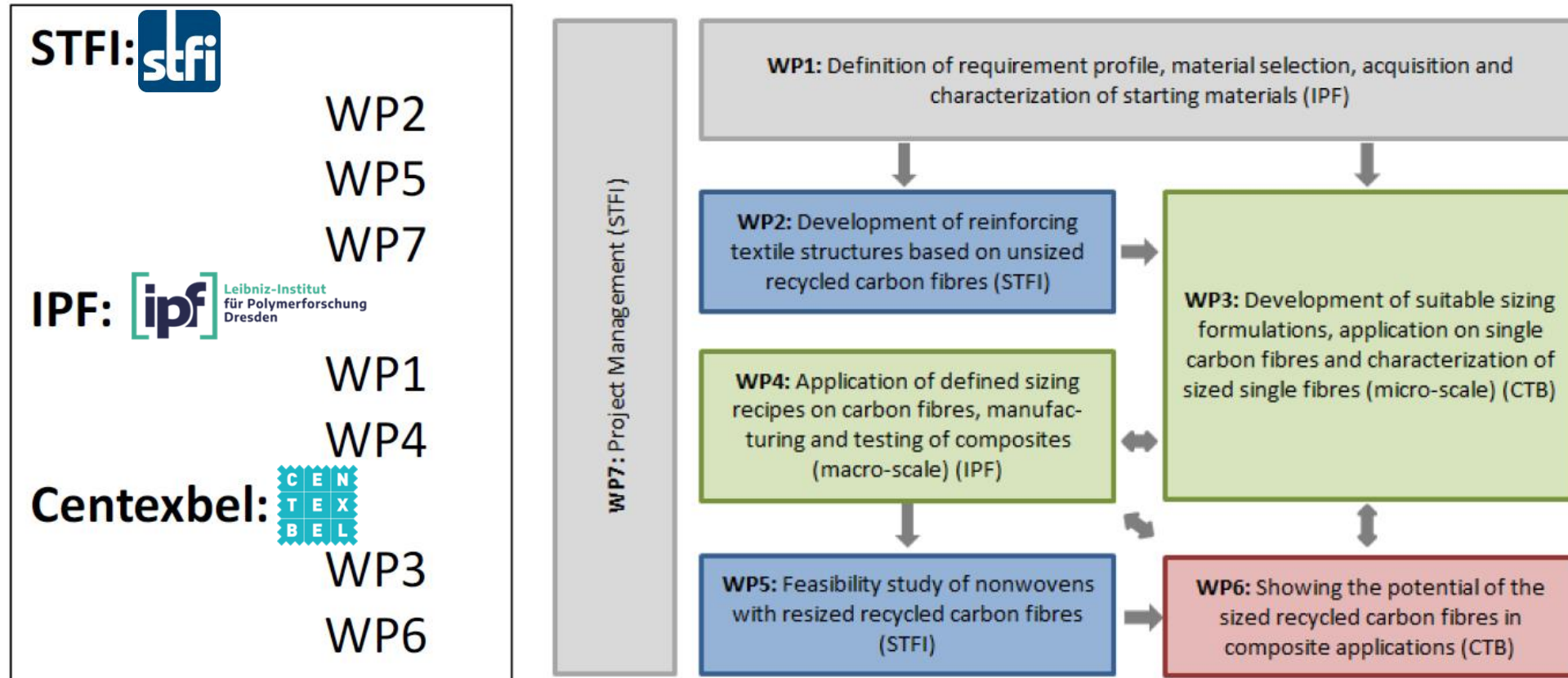


Different matrix => different sizing formulations

Special formulation for rCFs

RecarboSize

Resizing recycled carbon fibers to optimize **adhesion** with polymer matrices and improved **processing behavior** for composite manufacturing



Outline



Development of sizing formulations for epoxy matrix



Characterization of single carbon fibres



Feasibility study of reuse of resized rCFs in composites

Characterization of single recycled carbon fibre

* Favimat test

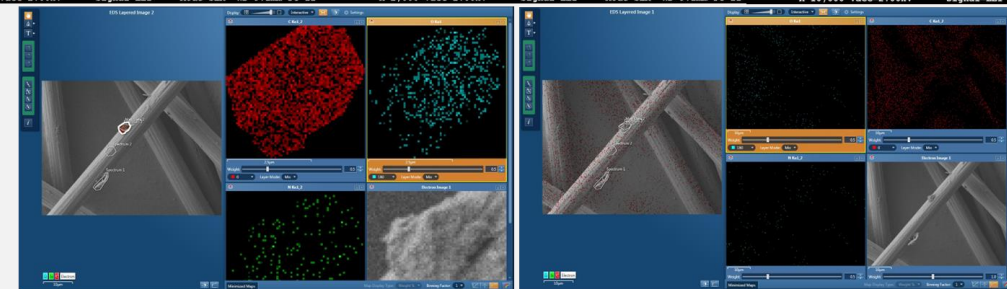
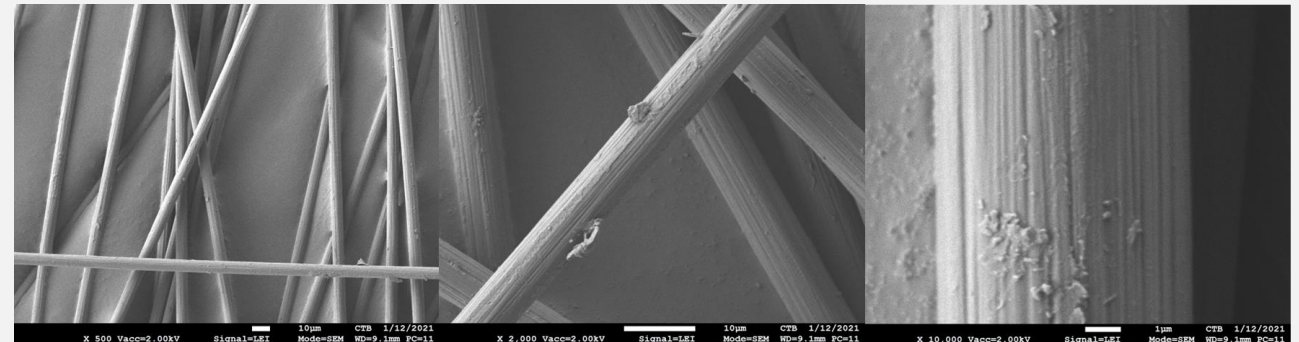
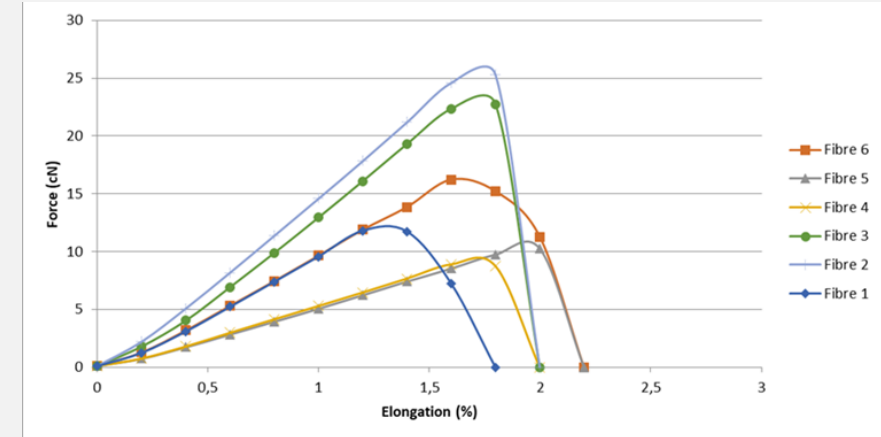
Tensile strength
Young's module

* SEM & SEM-EDX

Diameter of the fiber
Structure of the surface
Chemical composition

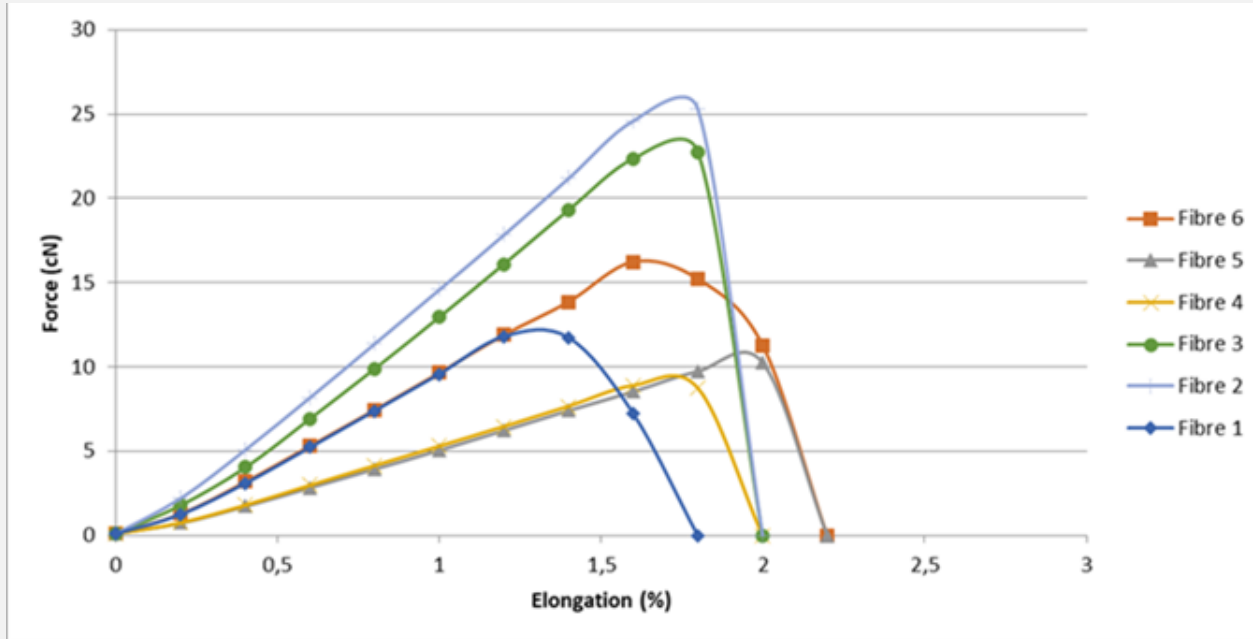
* TGA

Measure the amount and rate of change in the mass of a sample as a function of temperature and time in a controlled atmosphere.



SEM images of solvolyzed fibers
Diameter: 6.52 μm

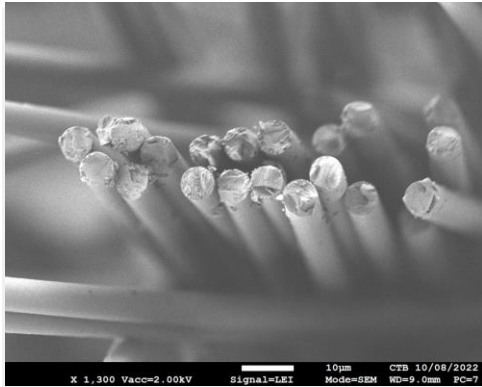
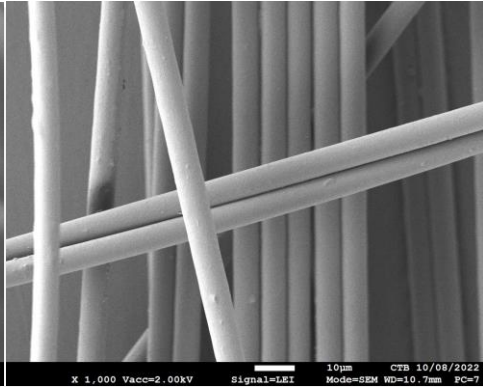
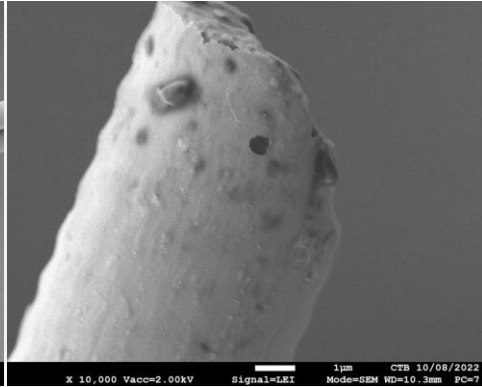
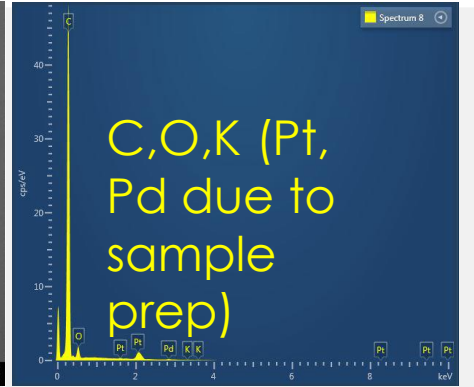
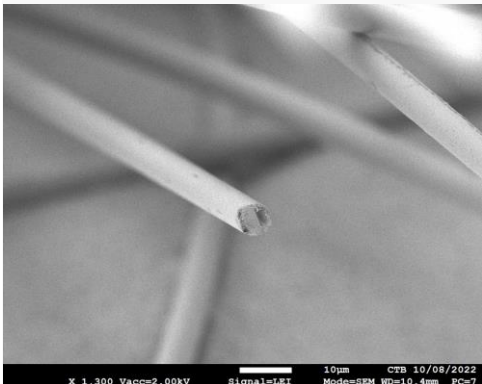
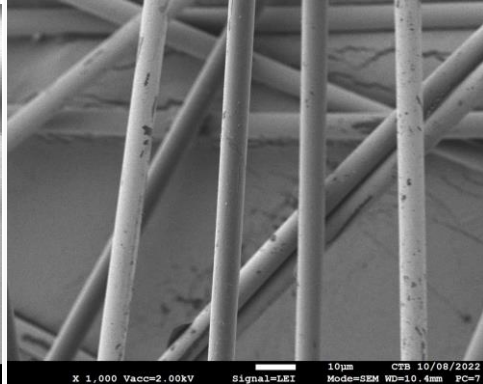
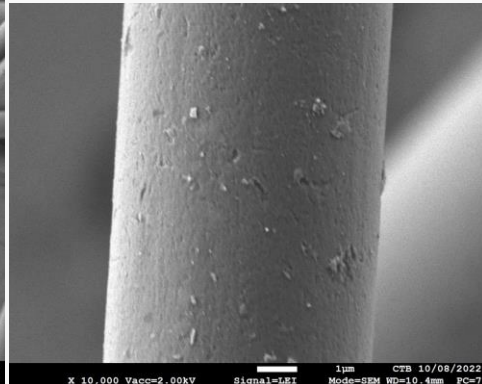
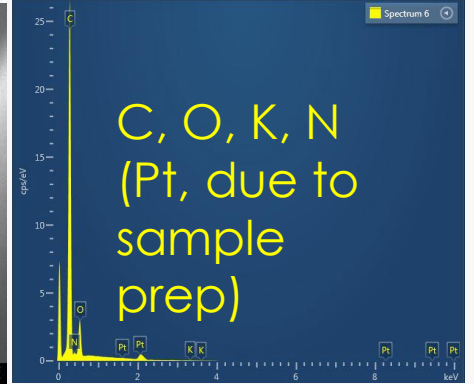
Favimat test on individual fibre



Individual carbon fiber force-strain curves, these curves indicate linear and elastic behavior to break for carbon fiber:

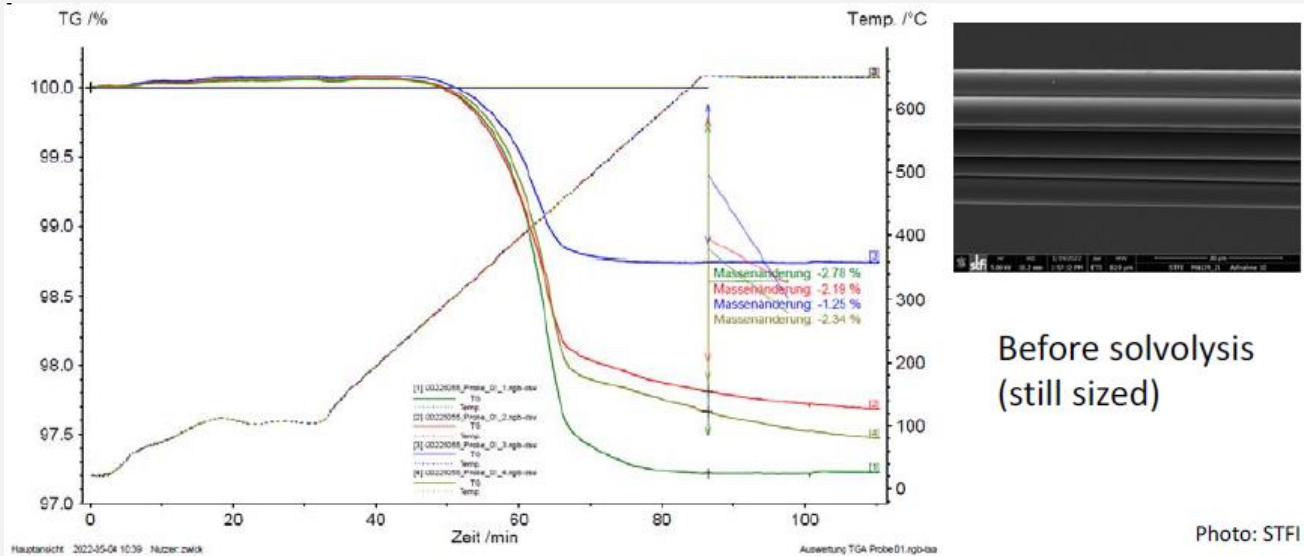
- Tensile strength
- Young's module

SEM on recycled fibers before and after pyrolysis

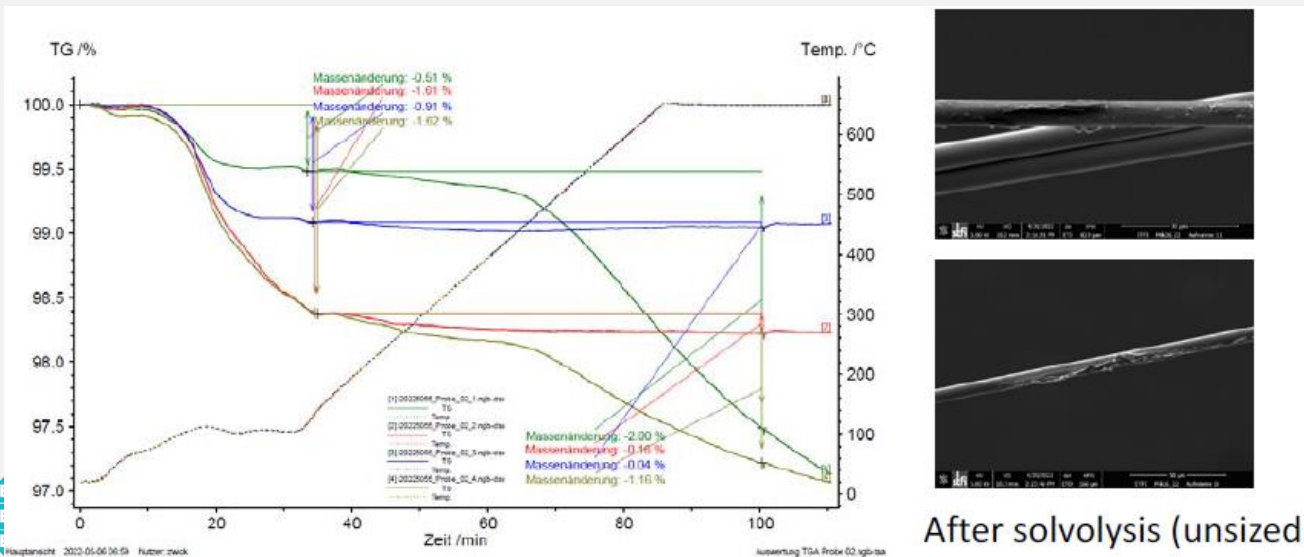
	x1300	x1000	x10000	Chemical composition
Before pyrolysis	 <p>X 1,300 Vacc=2.00kV Signal=LEI Mode=SEM WD=9.0mm PC=7</p>	 <p>X 1,000 Vacc=2.00kV Signal=LEI Mode=SEM WD=10.7mm PC=7</p>	 <p>X 10,000 Vacc=2.00kV Signal=LEI Mode=SEM WD=10.3mm PC=7</p>	 <p>Spectrum 8</p> <p>C, O, K (Pt, Pd due to sample prep)</p>
After pyrolysis	 <p>X 1,300 Vacc=2.00kV Signal=LEI Mode=SEM WD=10.4mm PC=7</p>	 <p>X 1,000 Vacc=2.00kV Signal=LEI Mode=SEM WD=10.4mm PC=7</p>	 <p>X 10,000 Vacc=2.00kV Signal=LEI Mode=SEM WD=10.4mm PC=7</p>	 <p>Spectrum 6</p> <p>C, O, K, N (Pt, due to sample prep)</p>

Some darker areas, small amounts of residue

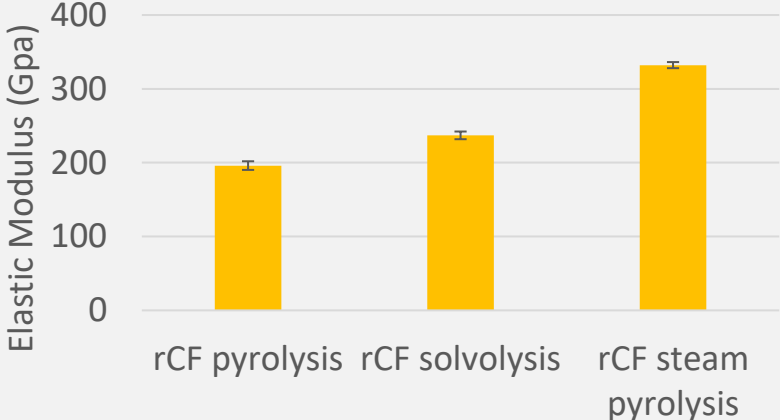
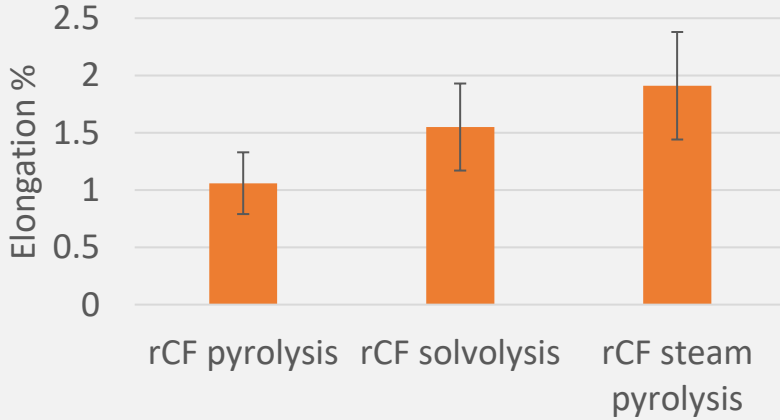
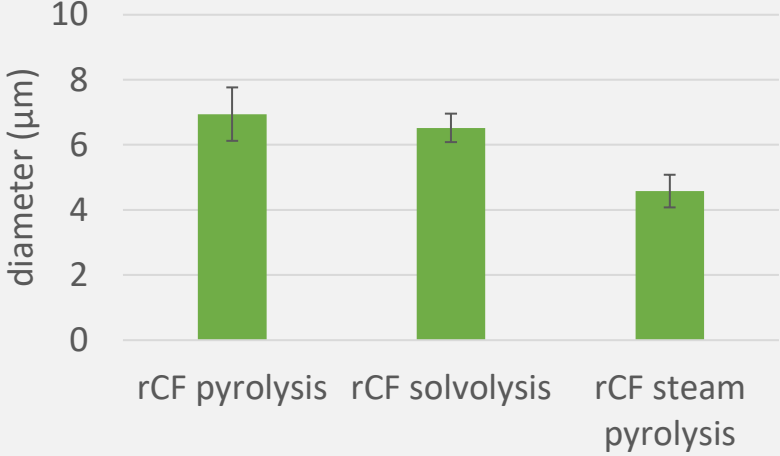
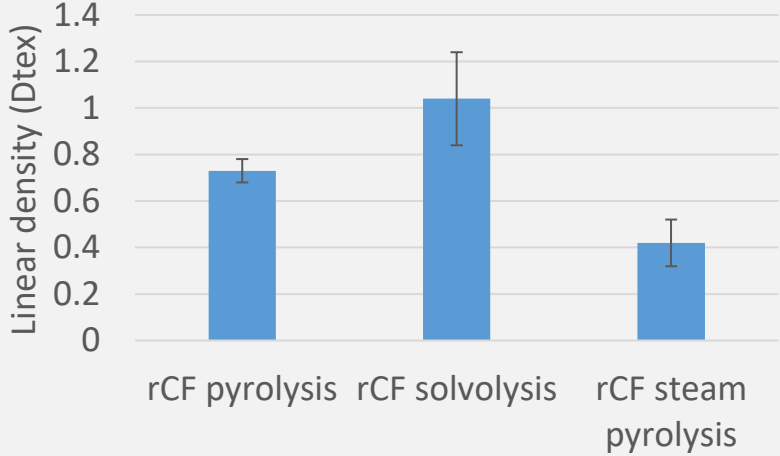
TGA and SEM on recycled fibers before and after solvolysis



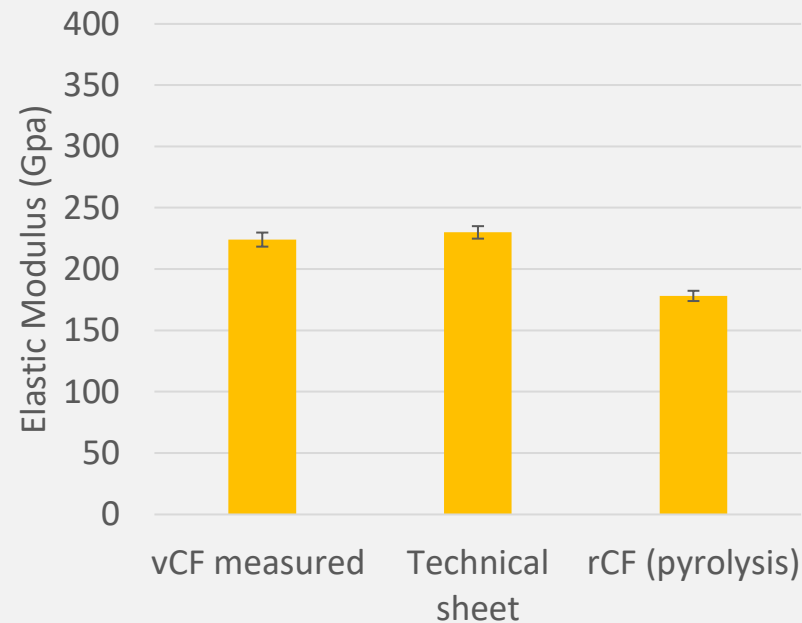
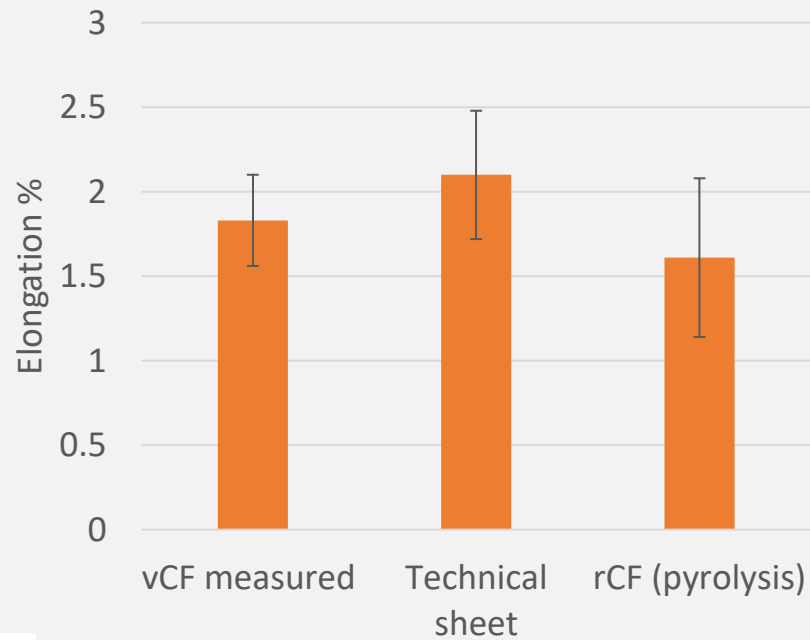
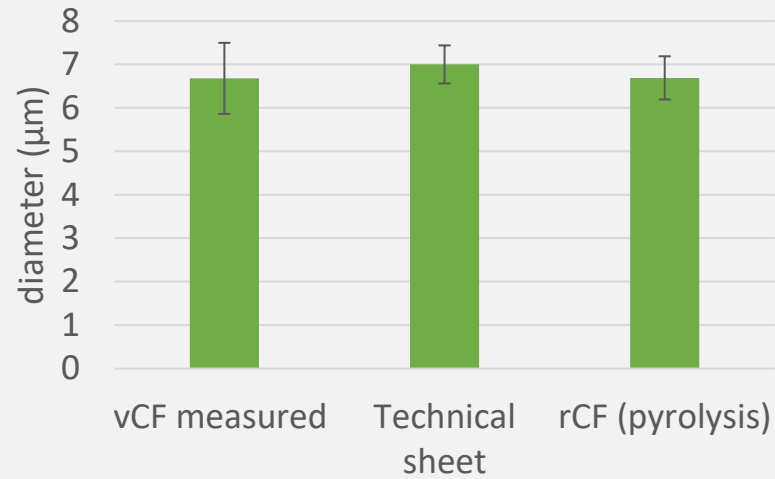
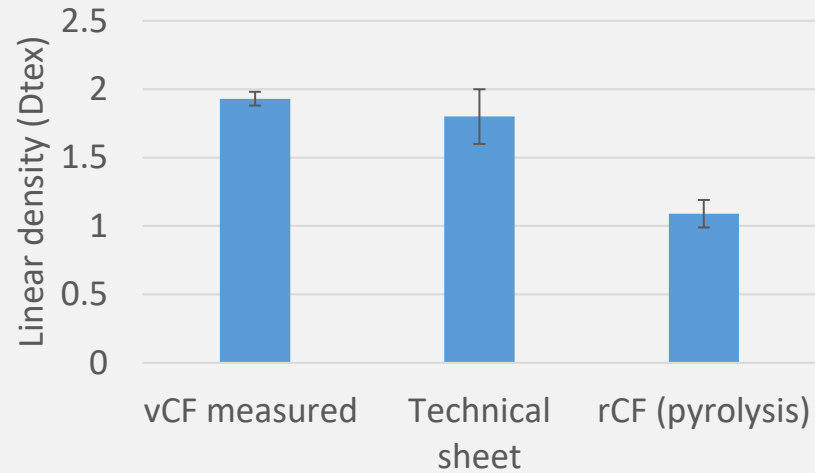
- Residue on the surface is visible
- The mass change is visible
- High variance in results before (amount of sizing from 1.25 to 2.78wt%) and after solvolysis



Comparison of various rCFs in the market



Comparison of vCF and rCF

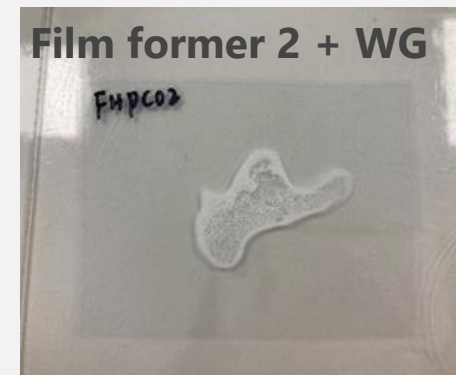
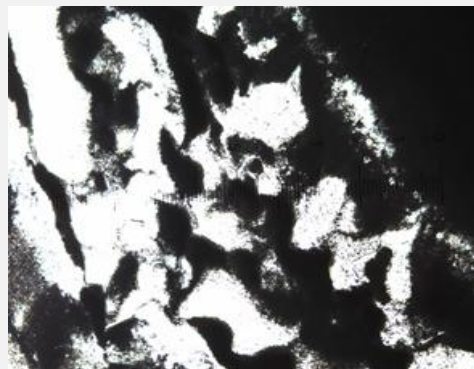
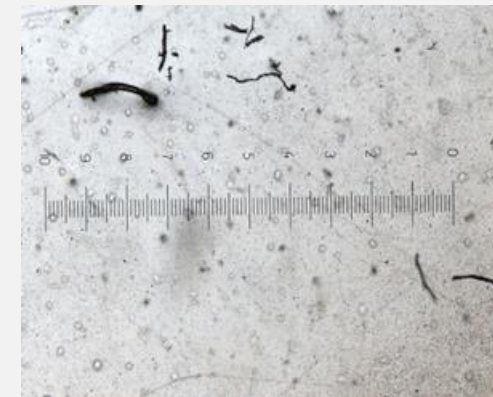
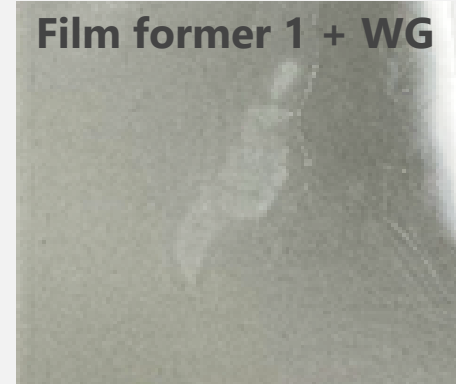
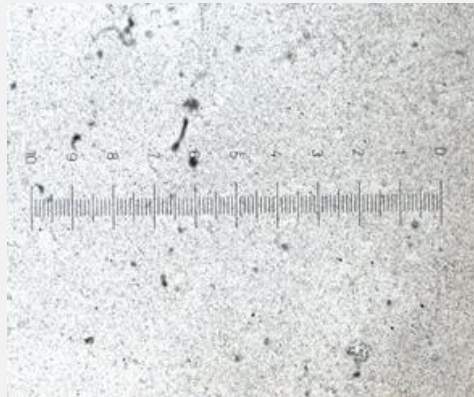


Development of sizing formulations

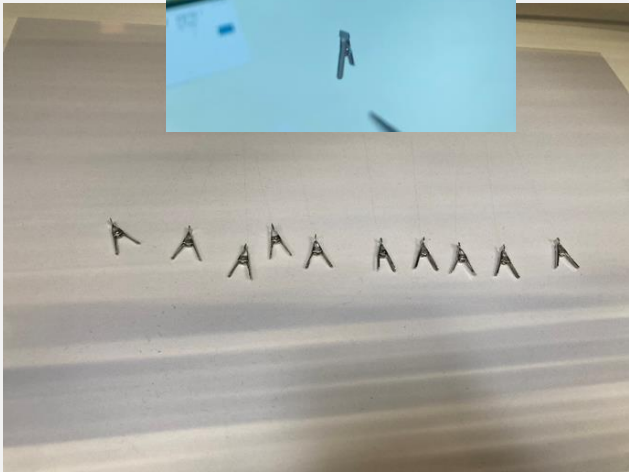
- Development of sizing formulation:
 - Ingredients: water + **film former** + **wetting agents** + coupling agents + lubricants + antifoaming agents
- Development of a process for applying sizing on individual fibres
- Characterisation of resized rCFs and comparison with unsized fibres

Sizing film characterization by optical microscopy

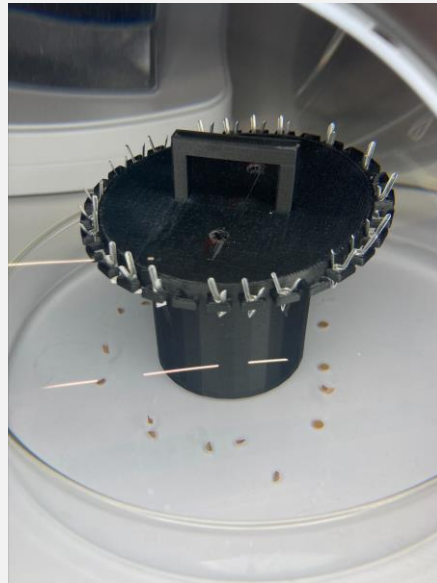
Dry sizing films on plastic foils



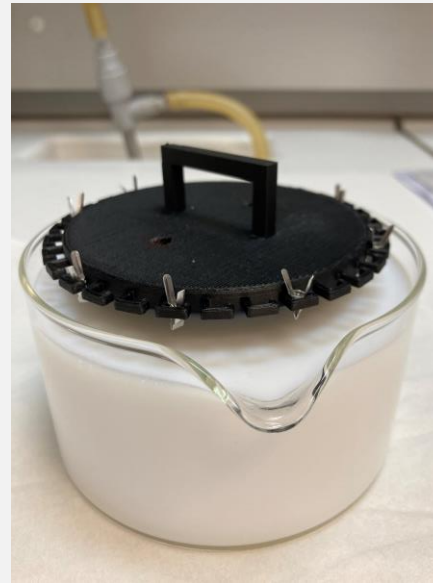
Resizing on individual fibers



Separate rCF using Favimat clamps and hang on the support



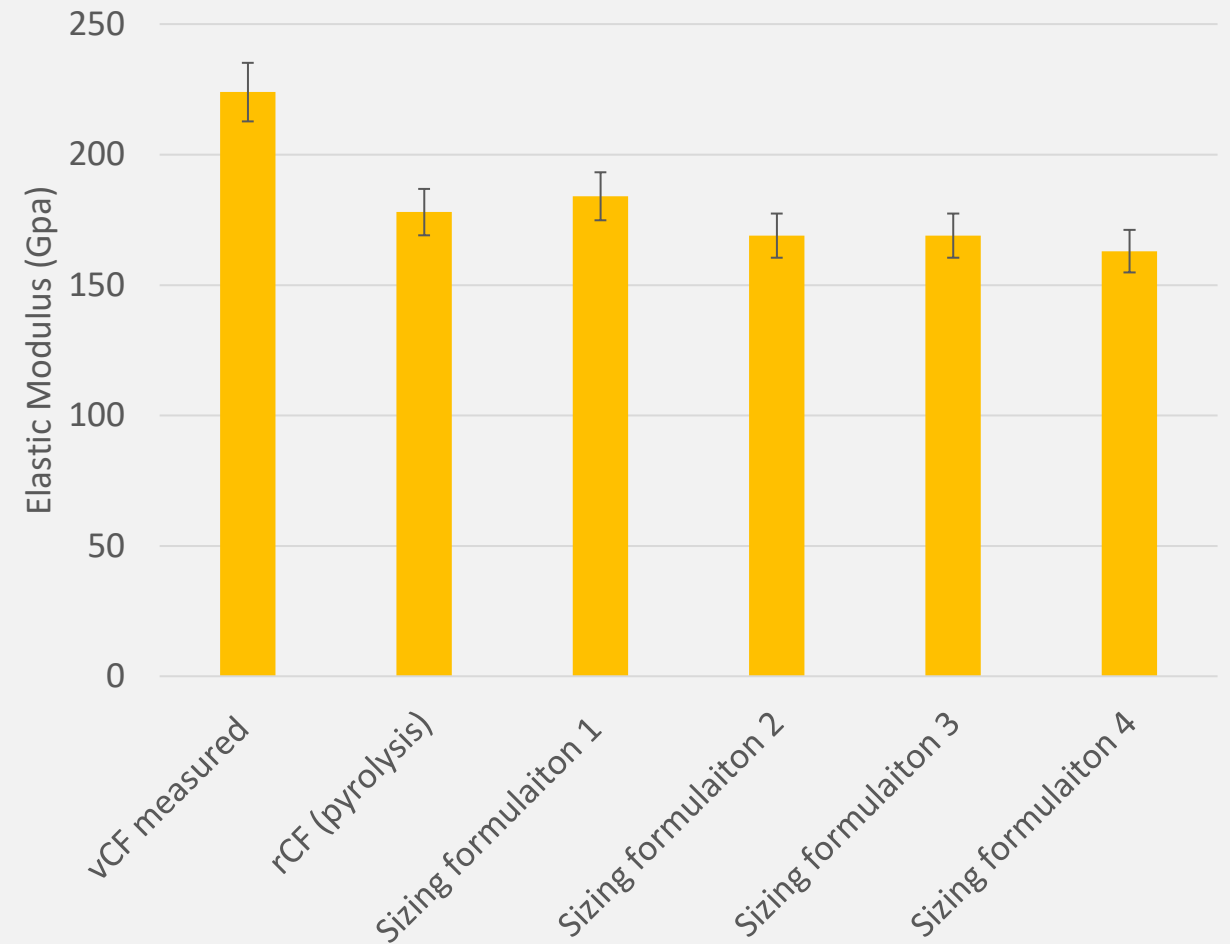
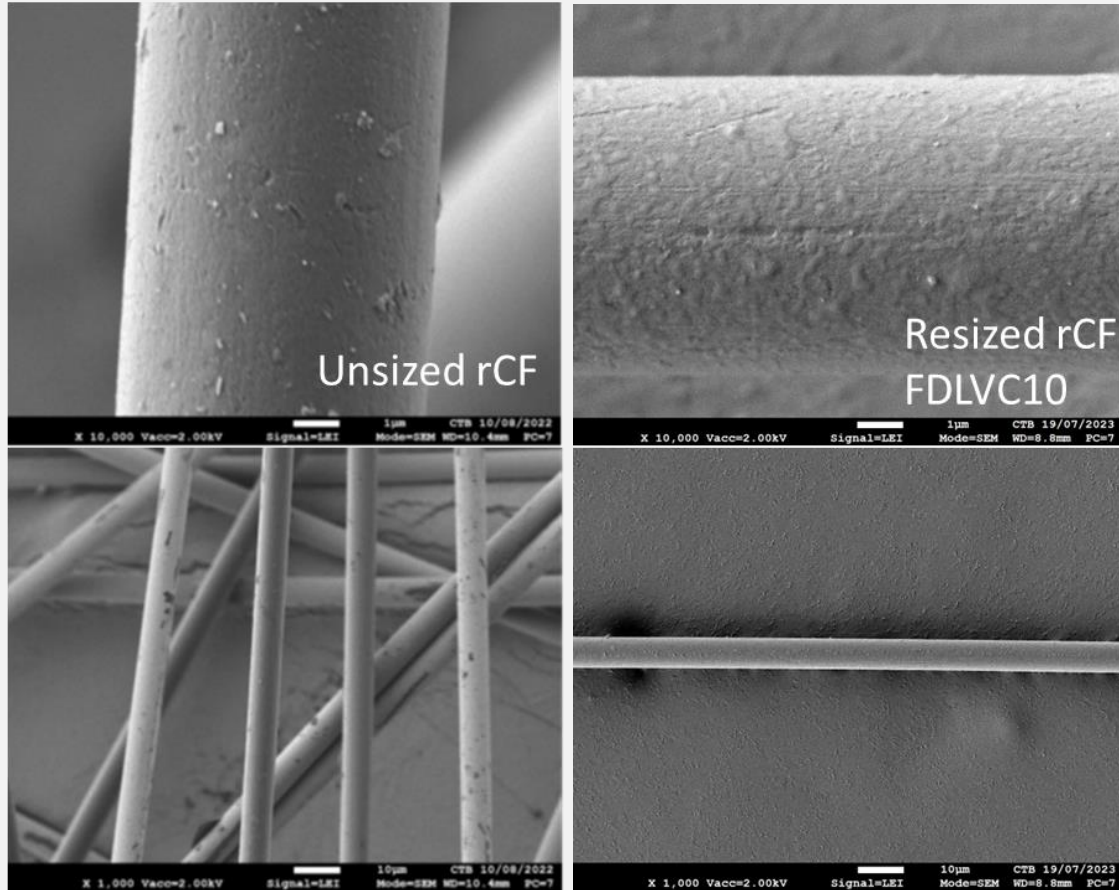
Immerse the rCF in the formulation solution with the support



Air dry overnight (conditioned room)

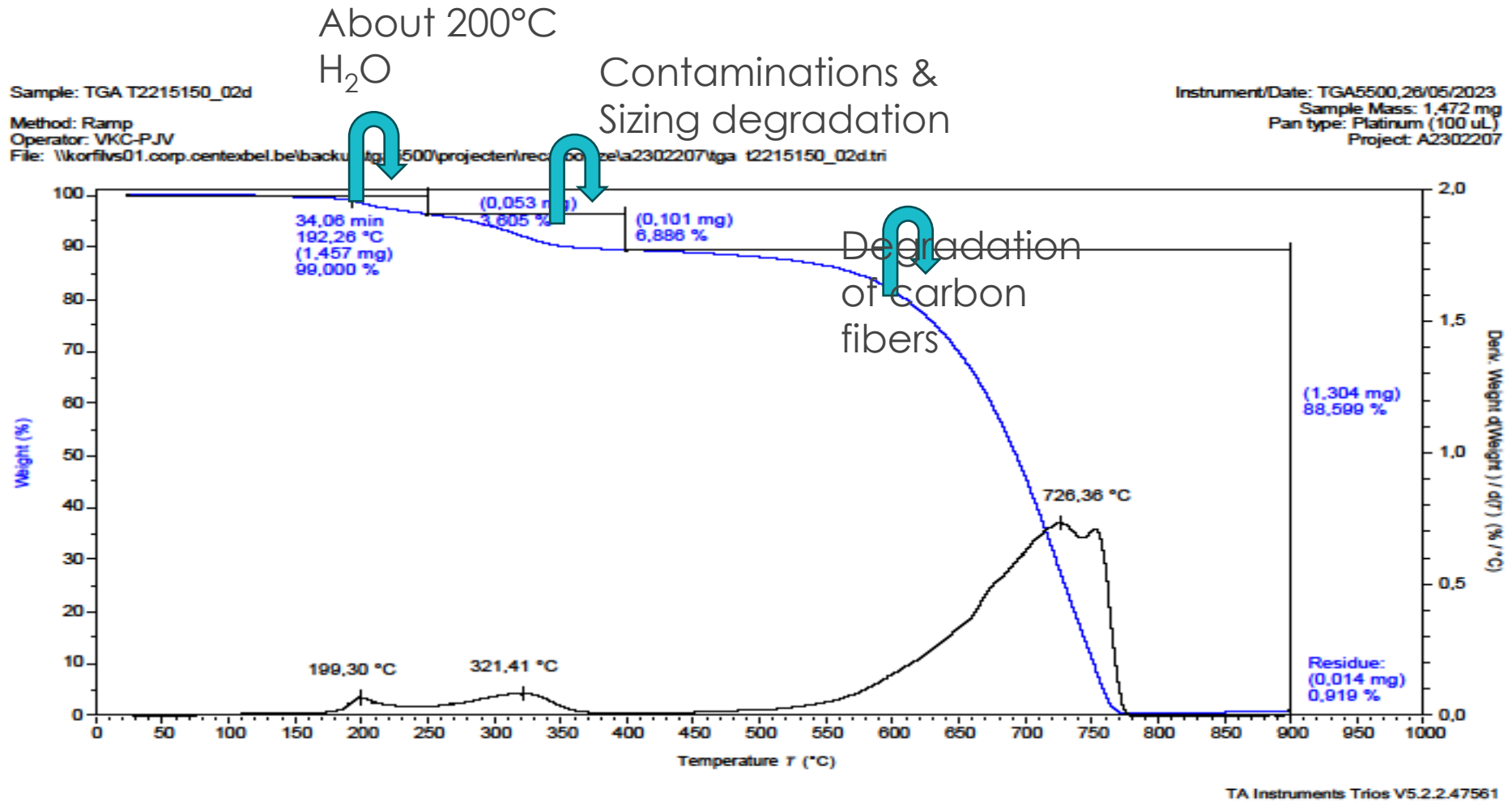
➔ Characterizations

Characterization of resized rCFs



➔ Not evident impact on the mechanical properties
Adhesion force with epoxy resin ?

Characterization of thermal stability of sizing by TGA



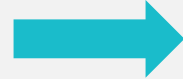
rCF + Sizing formulation

Reuse of resized rCFs in composites manufacturing

Nonwoven with 100% rCFs
or rCFs mixed with PA6
fibres



100% rCF sized



Application of selected sizing



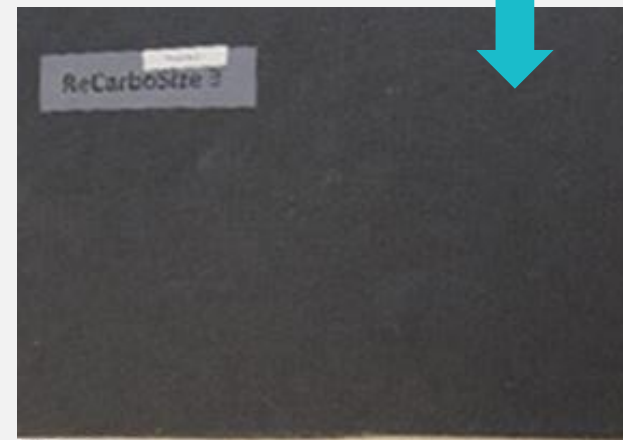
CTB



Composites by infusion

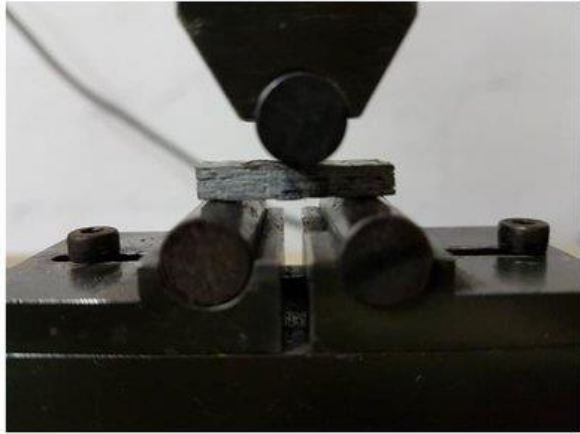


CTB

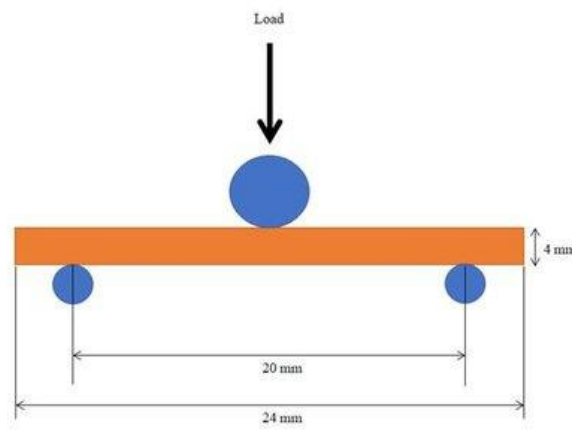


STFI

Interlaminar shear strength (ILSS) test



a



b

To measure the resistance of composite to delamination under shear forces parallel to the layers of the laminate, and so to the adhesion of interface.

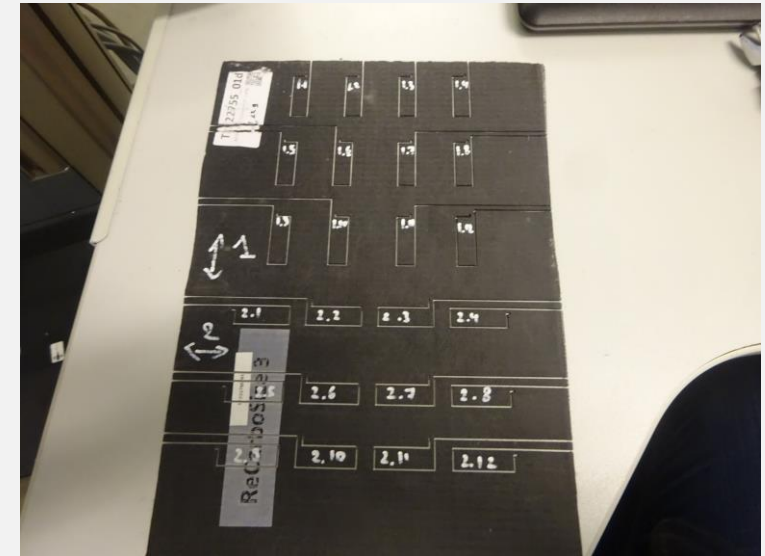
To calculate the interlaminar shear strength (ILSS), we use the equation you see on the right, where

$$\tau_{12} = 0,75 \times \frac{F_m}{B * d}$$

F_m = maximum compressive force in Newton

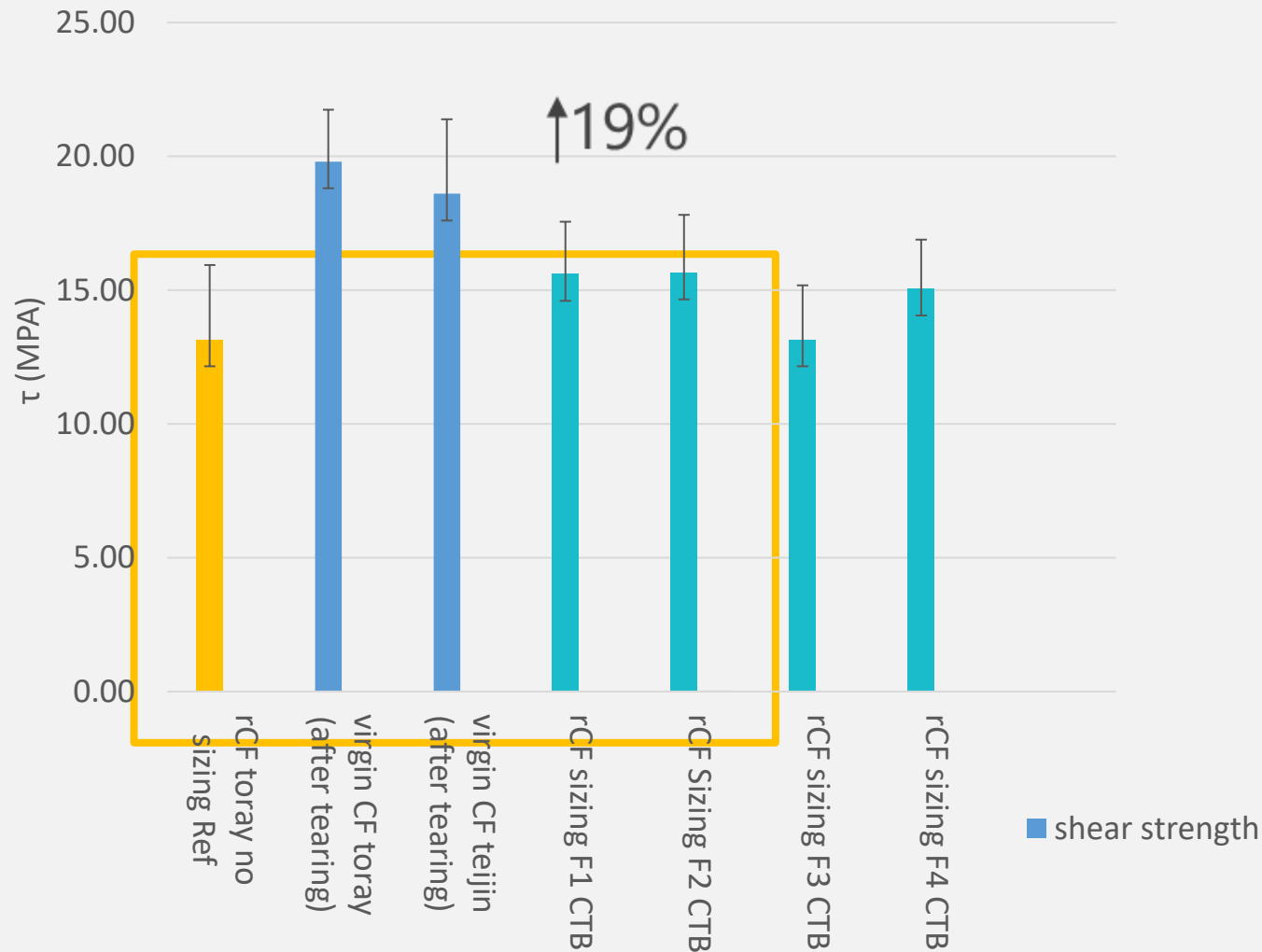
B = measured specimen width

d = measured specimen thickness



10 samples / direction

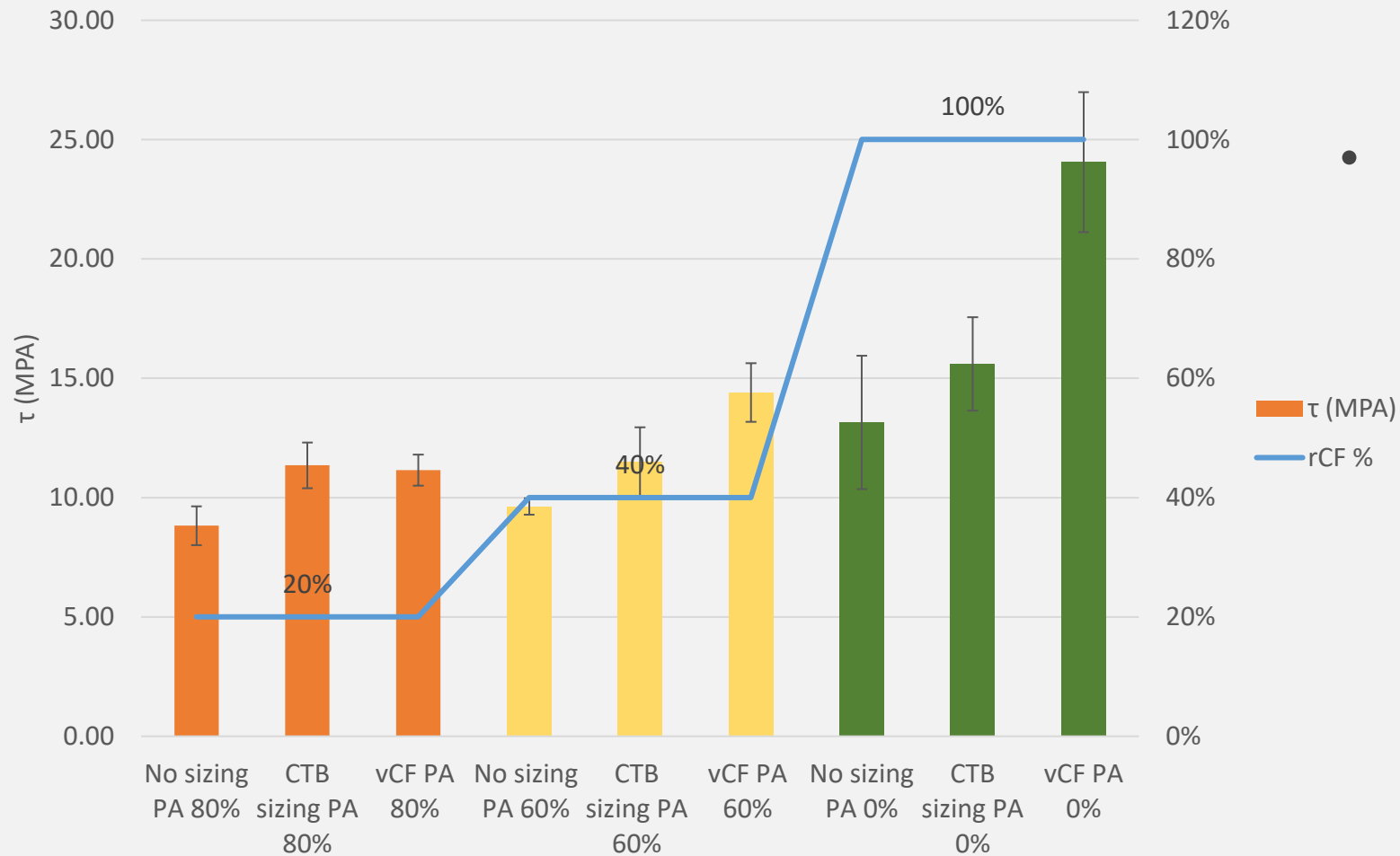
Comparison of shear strength VS sizing formulation



- Resized rCF composites > unsized rCF composites: an increase of 19% of shear strength
- Adhesion is improved by resizing
- vCF composites (with commercial sizing) > rCF composites (with resizing)
- Still need to improve

Comparison of shear strength VS rCF percentage

- Resized rCF composites > unsized rCF composites
- Increase of rCF % => increase of shear strength



Conclusion



Resizing recycled carbon fibers (rCF) can enhance the shear strength of the final composites.



There is no significant impact on the mechanical properties of the single fiber; however, resizing enhances the adhesion force between the resized fiber and epoxy resin.



Reusing resized rCFs in composites with various nonwoven structures is feasible. As the rCF% increases, the shear strength of the composites also improves.

Questions & remerciements



Contact:

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Wallonie