Sizing Solutions for Upgrading of Carbon Fibres with Nanomaterials Synthesised from Solvolysis Wastes

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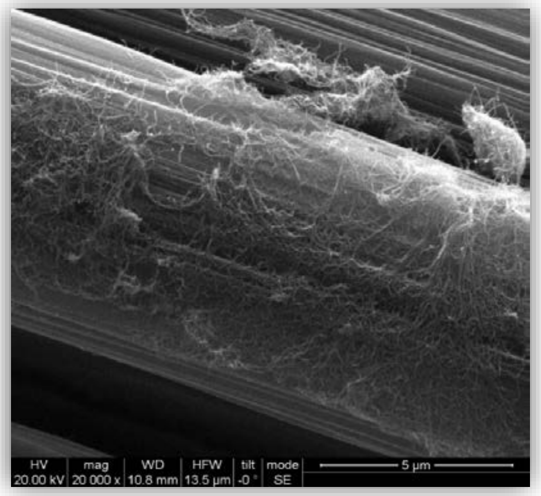
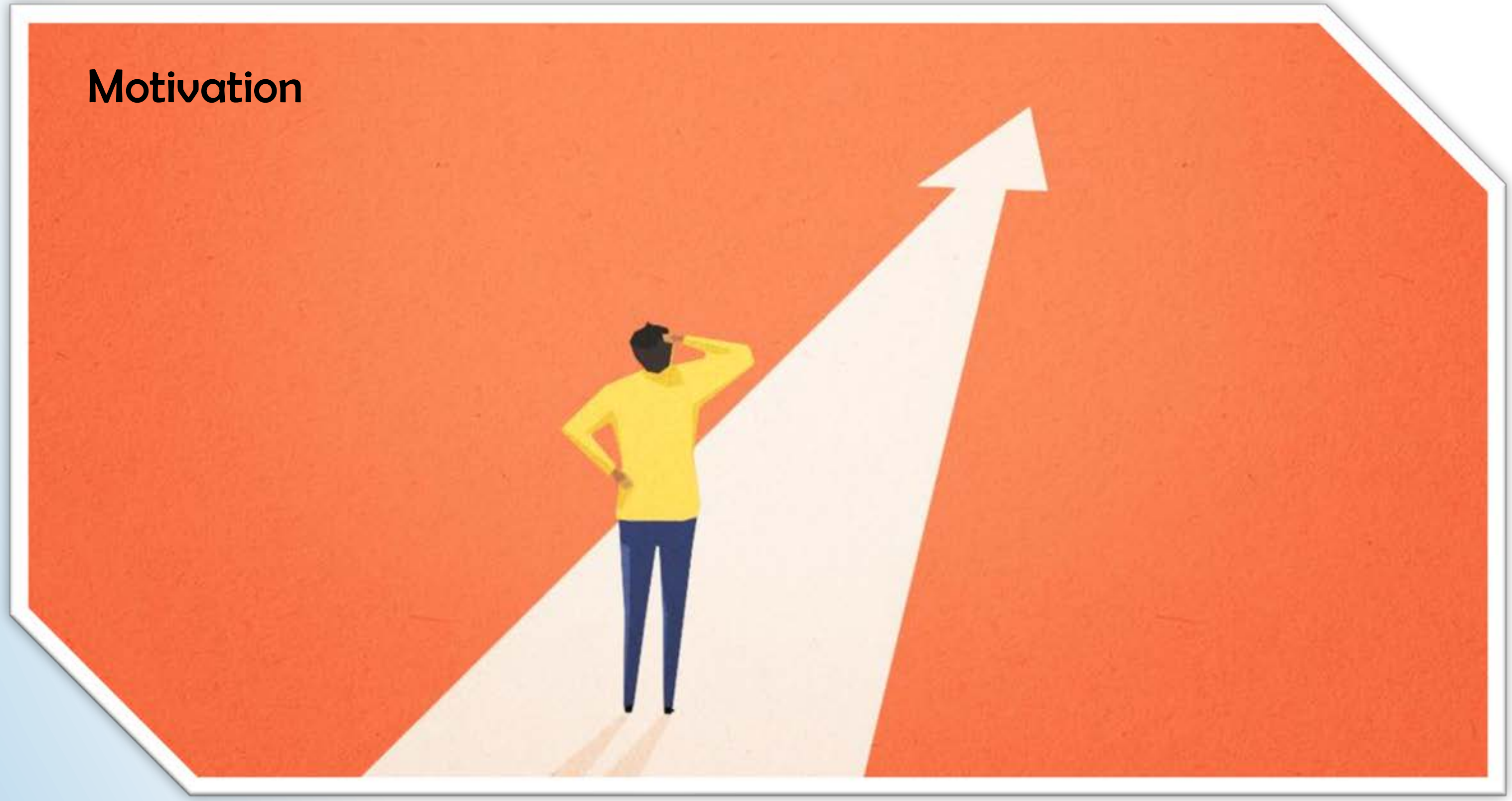


30/07/2023

Presentation Outline

- Motivation
- Development of formulations
- From Lab to Pilot scale
- Conclusions


Motivation

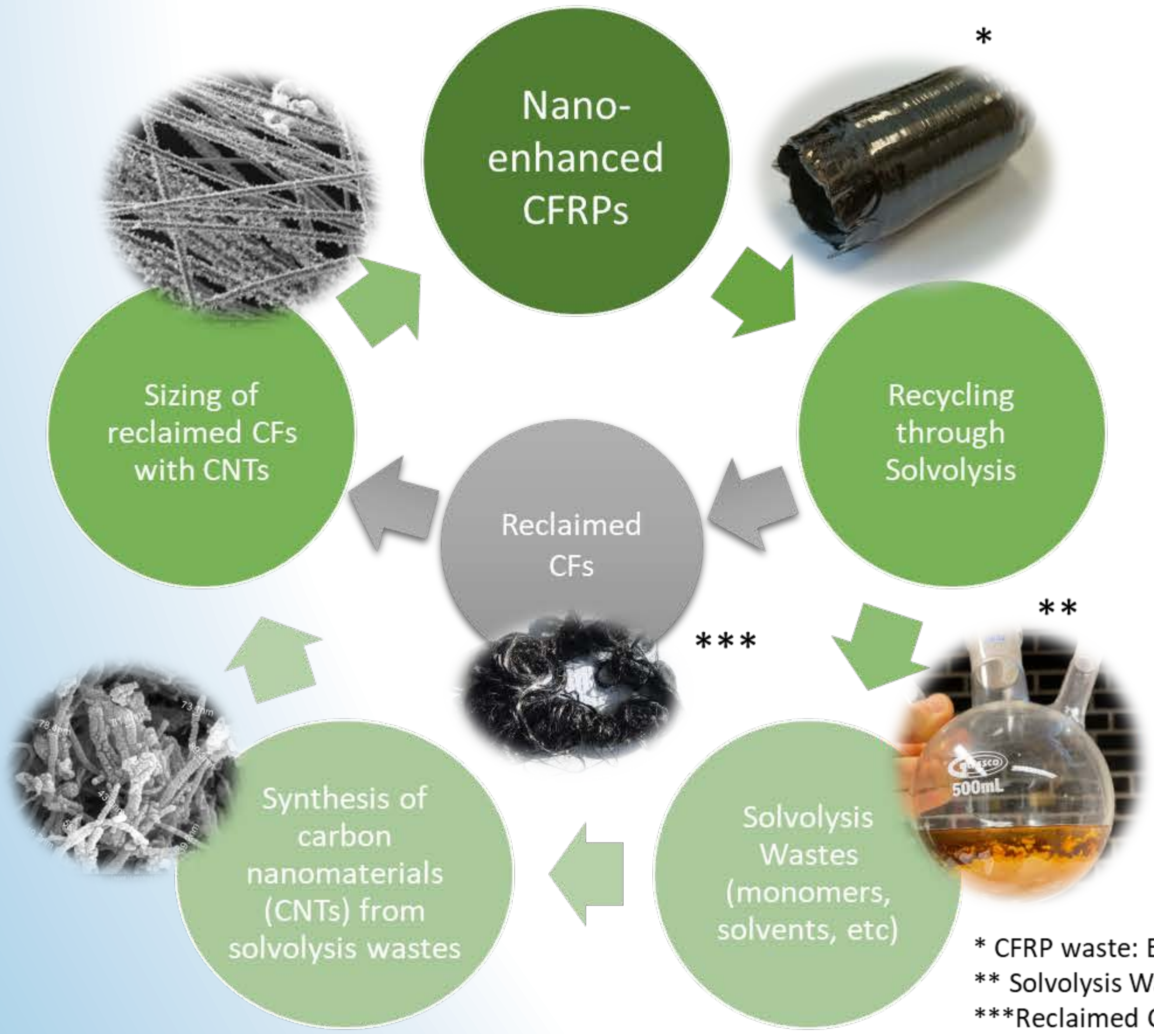
Motivation

Develop water based nano enhanced sizing solutions to improve compatibility of carbon fibres with resins and improve performance after recycling

Challenges:

Agglomeration of nanomaterials
Stable solutions
Uniform coatings
Upscaling

Circularity: From CFRPs to Nano-Enhanced CFRPs



Advantages:

- **Exploitation of solvolysis wastes** (major issue of the recycling process)
- Synthesis of **high-added value nanomaterials** (e.g. CNTs) from waste streams
- **Enhancement** of reclaimed Carbon Fibres and properties **improvement**
- New **nano-enhanced CFRPs** produced from recycled materials
- **Circularity** in the composites value chain

* CFRP waste: EoL part from B&T made by filament winding
** Solvolysis Wastes: By-product of Solvolysis process of SUT
***Reclaimed CFs: Achievement of UPATRAS through plasma enhanced solvolysis

Development of formulations

Solvolysis Wastes as Precursors for CNTs growth

Various organic precursors (hydrocarbons) in liquid form have been used to synthesize CNTs:

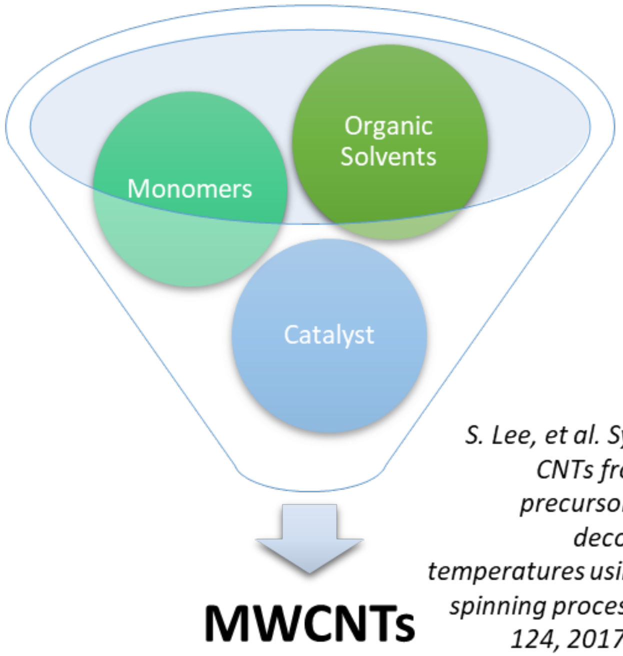
CNT precursors

- **Type I: High decomposition temperatures**
  - Toluene
  - Hexane
  - Benzene
  - Xylene
- **Type II: low decomposition temperatures**
  - Acetone
  - Ethanol
  - Ethylene Glycol
  - Butanol
- **Type III: low decomposition temperatures (gaseous precursors)**
  - Methane
  - Ethene
  - Acetylene

Sufficient time for the growth of catalyst particles to a critical size before the catalysts contact C atoms.

Oxygen atoms in their structure that prevent catalyst particles from deactivation by removing C atoms from the catalyst surfaces.

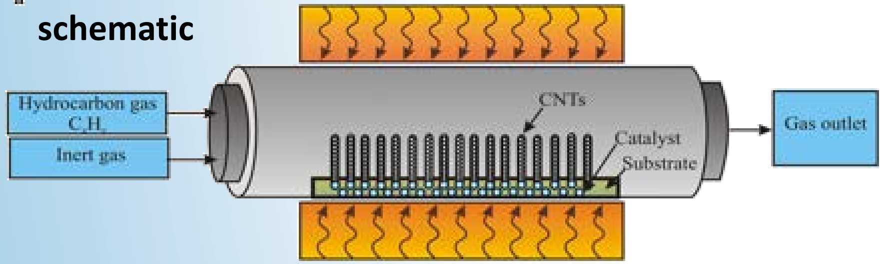
The resulting liquid waste byproduct from a CFRPs solvolysis process contain a mixture of water (in the case of supercritical water) and **organics** from the depolymerization of the thermoset resin (monomers and **excess reactive solvent**)



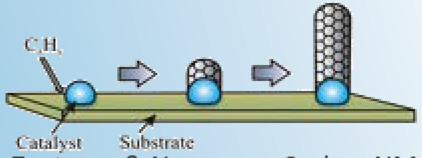
S. Lee, et al. Synthesis of CNTs from carbon precursors with low decomposition temperatures using a direct spinning process, Carbon, 124, 2017, 219-227,

Chemical Vapour Deposition Set-Up

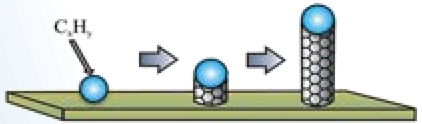
a **CVD reactor schematic**



b Base CNT growth

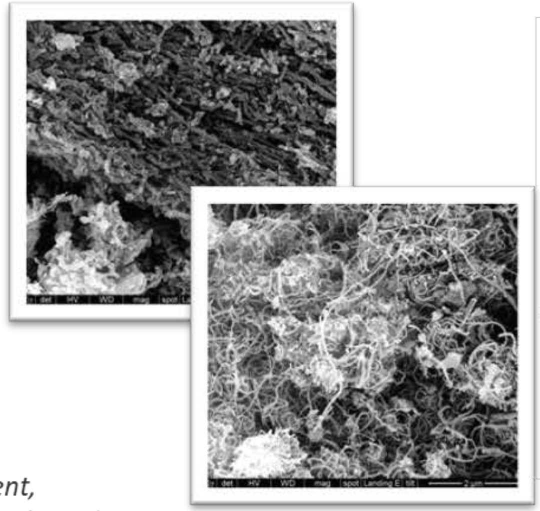


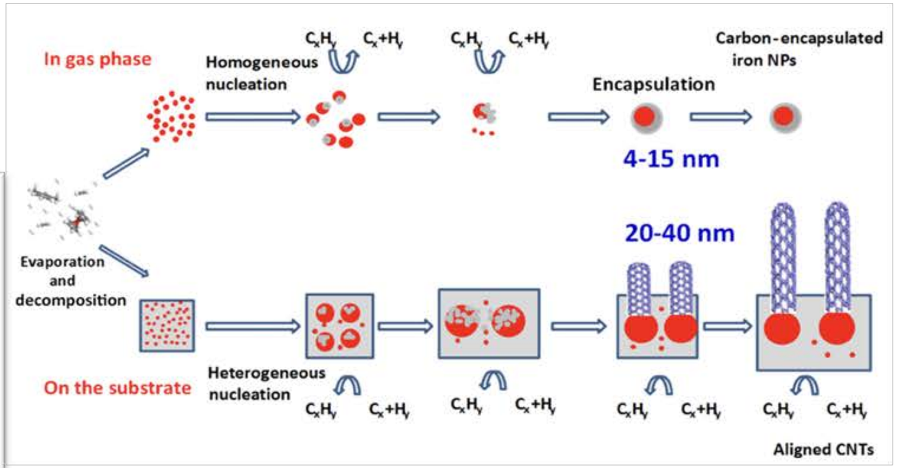
c Tip CNT growth



Zaytseva & Neumann, Carbon NMs: production, impact on plant development, agricultural and environmental applications. Chem. Biol. Technol. Agric. 3, 17 (2016).

MWCNTs in entangled or aligned form





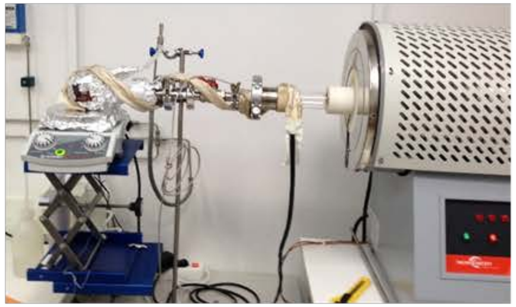
Y. Xu, et al. Evolution of Nanoparticles in the Gas Phase during the Floating CVD Synthesis of Carbon Nanotubes. J. Phys. Chem. C, 2018, 122 (11), 6437-46.

Different inlet alternatives to introduce the Solvolysis Liquid Waste in the CVD Reactor in R-NanoLab

Introduction through separation funnel

Boiling in conical flask

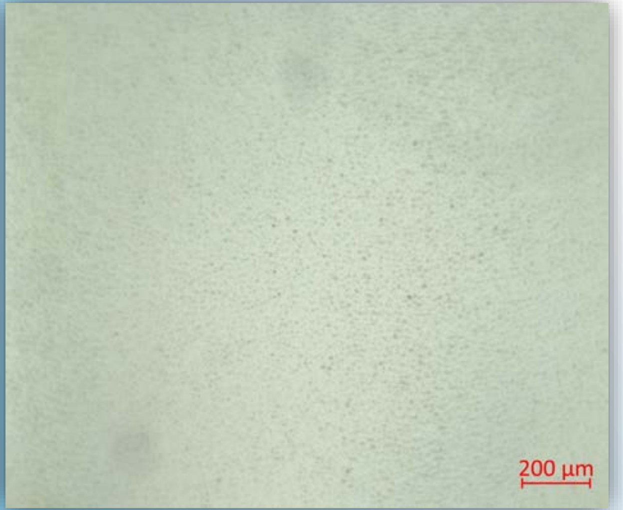
Boiling in spherical flask



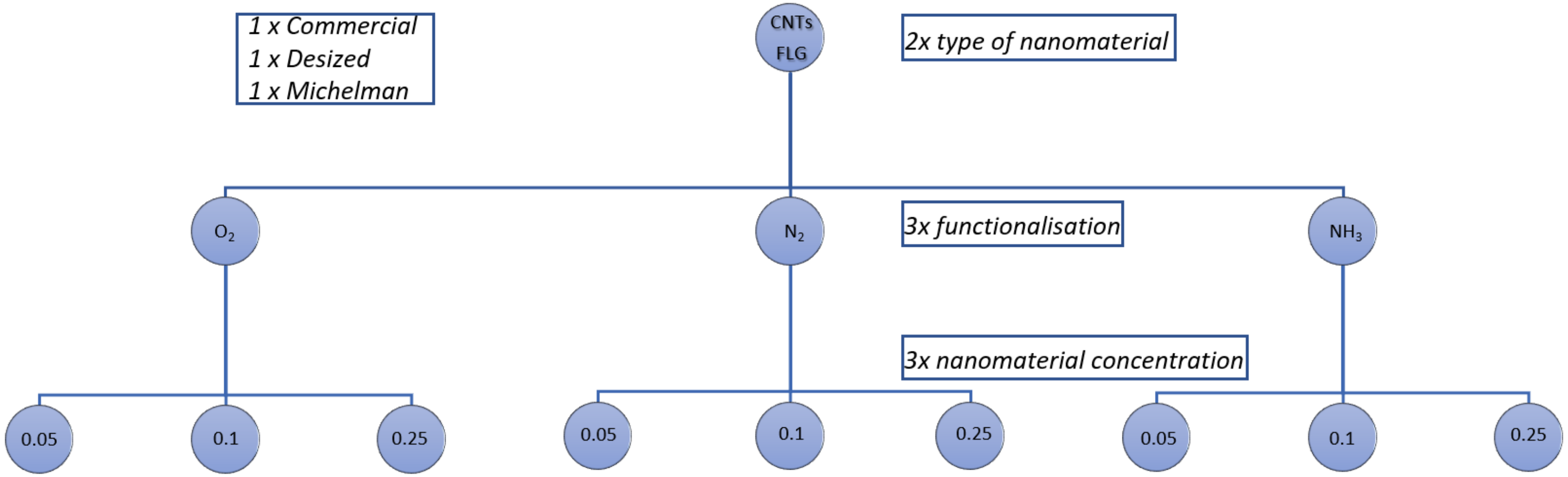
Boiling in two-necked spherical flask

Development of formulations

Michelman HP2-06 as commercial water base sizing (1-2.5-5%) + water dispersed nanomaterials (0.05-0.1-0.25%)



Hielschler Sonication Probe 45m + Triton X

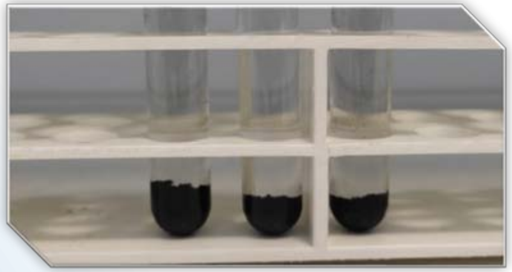


FLG dispersions

- 2 %wt FLG-O_2 water dispersion
- 2 %wt FLG-N_2 water dispersion
- 2 %wt FLG-NH_3 water dispersion

CNT dispersions

- 2 %wt CNT-O_2 water dispersion
- 2 %wt CNT-N_2 water dispersion
- 2 %wt CNT-NH_3 water dispersion

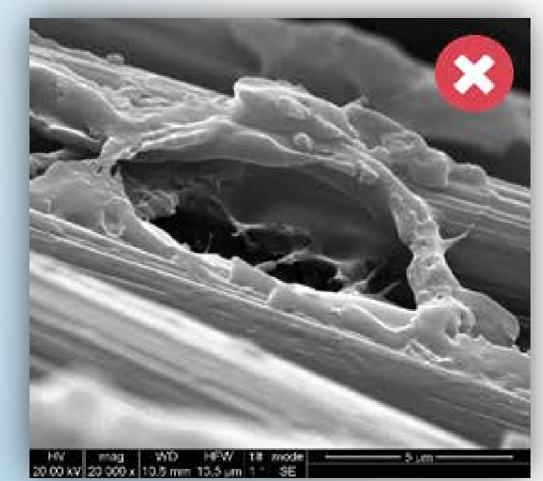


Use of surfactant for CNT dispersions

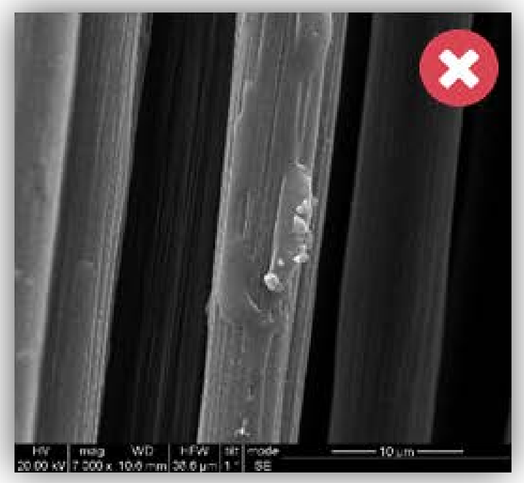
- Sodium Dodecyl Sulfate (SDS)
- Cetrimonium bromide (CTAB)
- Polyvinylpyrrolidone (PVP)
- TRITON X 100

Development of formulations

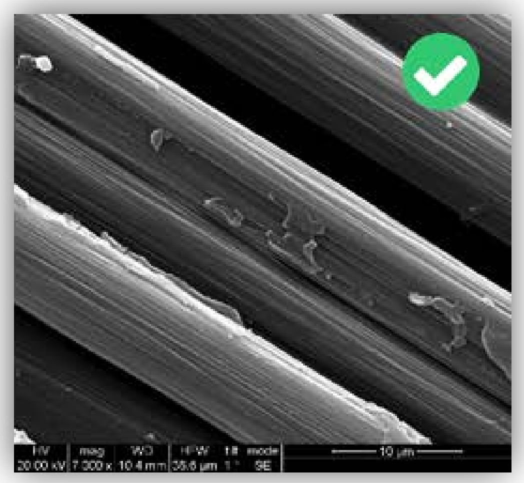
- Solid Content Concentration investigation:



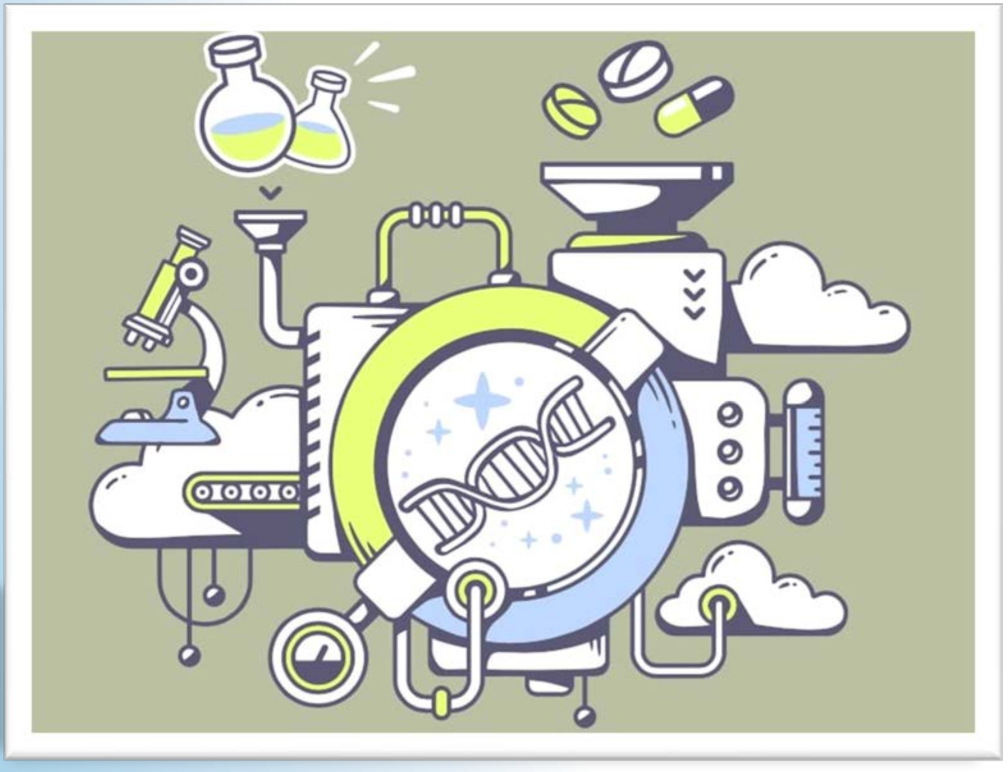
5% solids



2.5% solids



1% solids



From lab to pilot scale

From Lab to Pilot scale

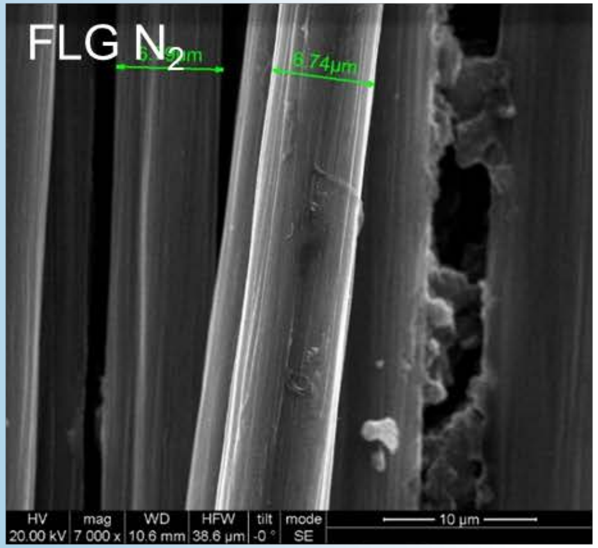
100ml of the 18 sizing formulations have been prepared and each one has been used to coat 10 CF bundles of 30cm length at the lab scale apparatus.

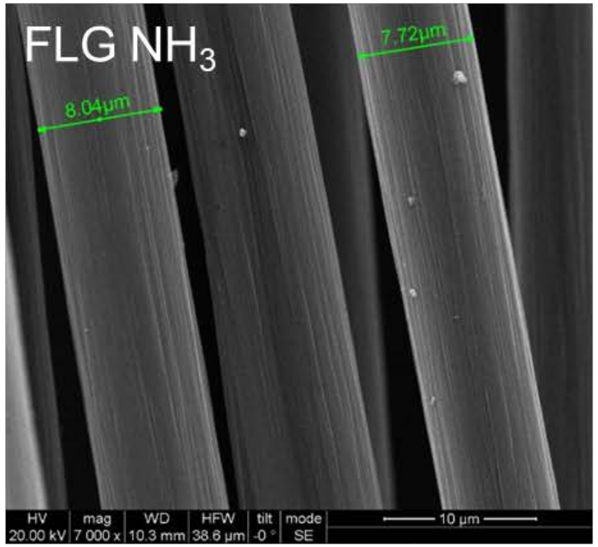
Subsequently the fibres have been assessed for their morphological characterization (SEM), affinity with resin (CAG) and mechanical properties (Tensile - 3 p. bend).

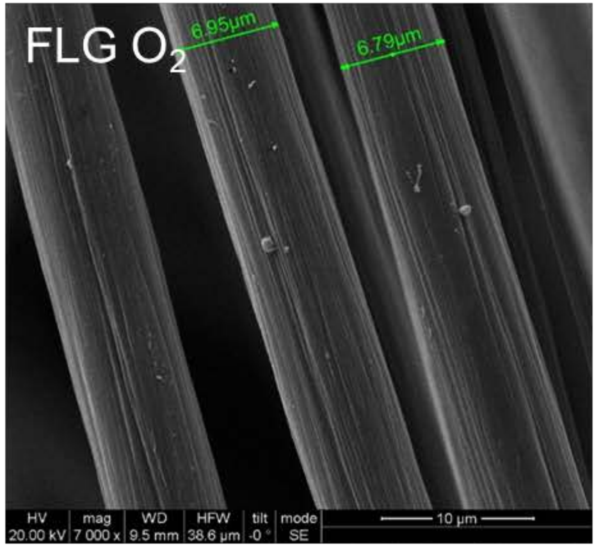
For the Mechanical tests the fibres have been impregnated in epoxy resin and left to cure in a 3D printed tool under tension.

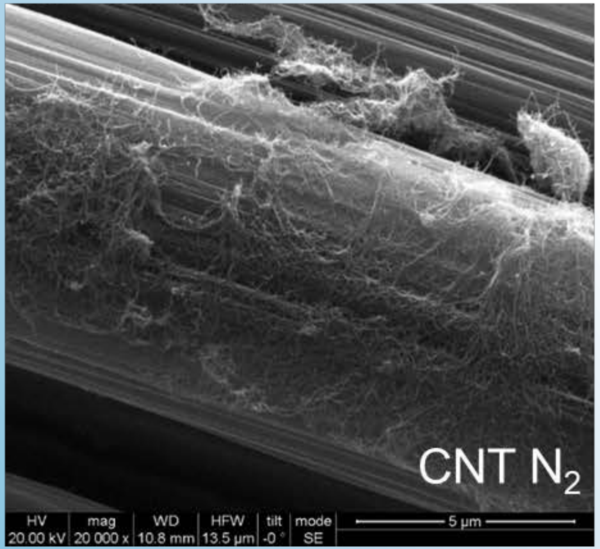
More than 200 samples in total were tested under tensile loading and 3 point.

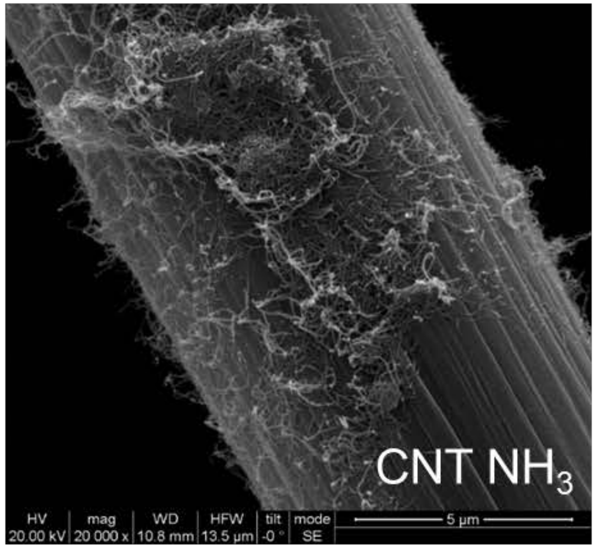
From Lab to Pilot scale

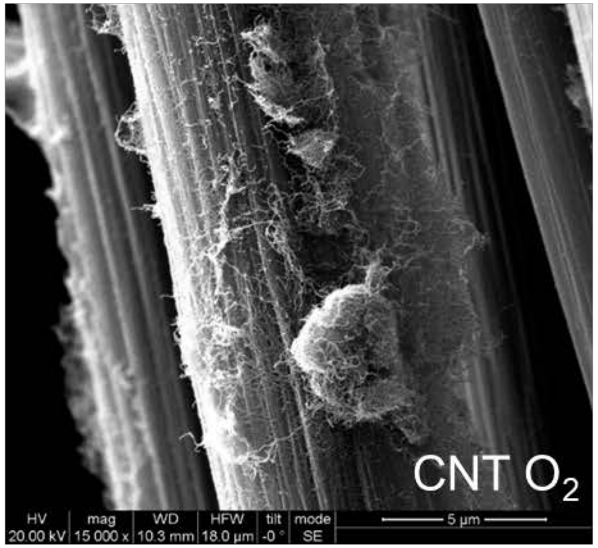








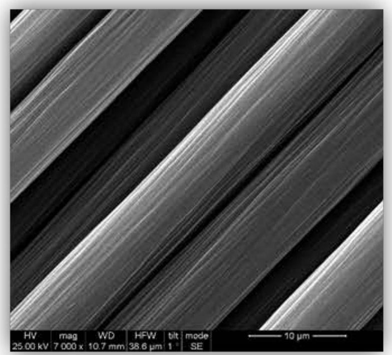




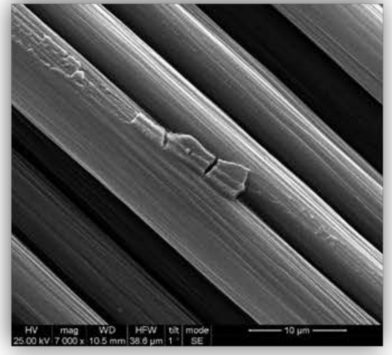
From Lab to Pilot scale

			CNTs									FLG								
			O2			N2			NH3			O2			N2			NH3		
	Desized	**Michelman**	**0.05**	**0.1**	**0.25**	**0.05**	**0.1**	**0.25**	**0.05**	**0.1**	**0.25**	**0.05**	**0.1**	**0.25**	**0.05**	**0.1**	**0.25**	**0.05**	**0.1**	**0.25**
3 point	31.3 ±3	37.1 ±4.3	35.6 ± 4.5	36.1 ± 5	37.9 ±3.8	41.8 ± 3.7	**44.5 ± 3**	34.2 ± 3.5	36.6 ± 2.4	39.1 ± 3.3	37.9 ± 2	41.3 ± 2.7	43.1 ± 6.6	41.9 ± 3.6	43 ± 4.1	**45.1 ± 2.8**	41.3 ± 3.1	33.6 ± 3	42.2 ± 3.9	34.2 ± 2.6
Tensile	262 ± 31	391 ± 49	317 ± 34	271 ± 24	334 ± 33	430 ± 27	**434 ± 12**	376 ± 44	333 ± 27	380 ± 32	388 ± 47	343 ± 27	391 ± 49	421 ± 61	391 ± 34	**444 ± 39**	354 ± 40	441 ± 156	336 ± 41	351 ± 15

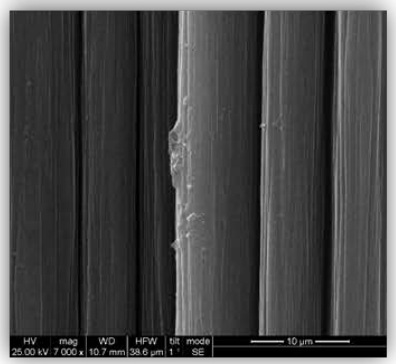
From Lab to Pilot scale



1% solids



2.5% solids



5% solids

Conclusions

Conclusions

Challenges:

- Agglomeration of nanomaterials
- Stable solutions
- Effective desizing
- Uniform coatings
- Upscaling

Solutions:

- Hielschler Sonication Probe
- Triton X surfactant
- Few seconds at 600°C
- Control of Solid Content in the sizing formulation
- Addition of nanomaterial can improve mechanical properties

Further Investigation:

- Uniform distribution of nanomaterials onto the CF surface during sizing process

Acknowledgments

EuReComp

CARBO4POWER

Thank you for your attention

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