Relicario : Up-scaling of the PHYre process

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RELICARIO AT A GLANCE

Project Content

RELICARIO - Recycling of End-of-LIfe CArbon fiber Reinforced plastics

April 2019 – March 2022

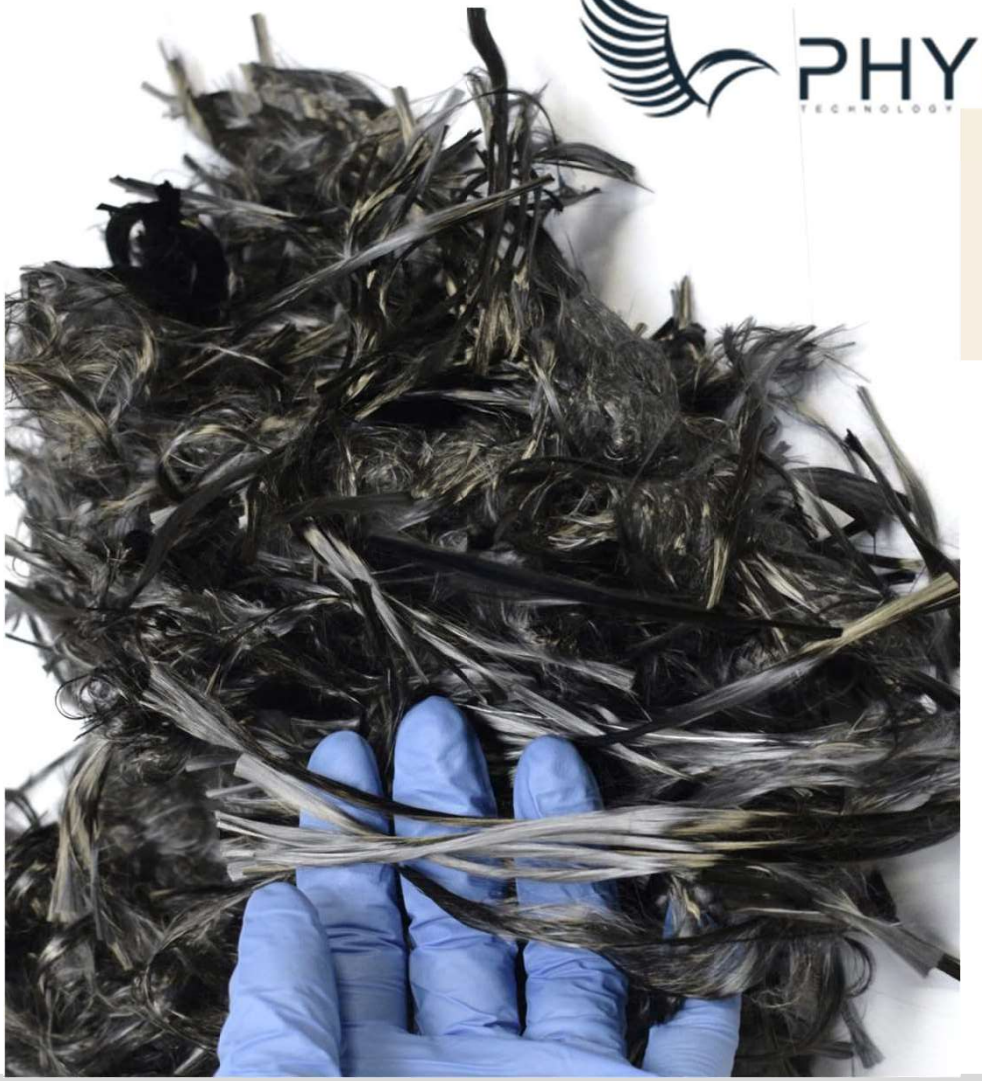
KIC Funding 2,6 M€

Total project budget: 4 M€

The project aims to:

- Offer a new viable solution to substitute steel containing CRM and primary carbon fibers in automotive industry
  - → Prove the competitiveness of the thermosolvolysis recycling process
- Scale-up the PHYre® technology – TRL 8
- Evaluate the limits of rCF
- Simulate the properties of new composites
- Prove oligomer valorization and solvent/catalyst recycling
- Stablish a complete Life-cycle Analysis and Cost
- Validate the use of rCF by end-users

The PHYre Process



PHYre TECHNOLOGY

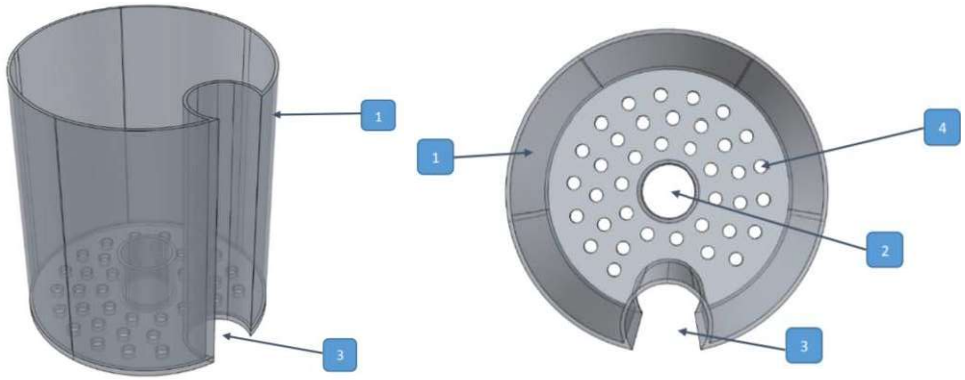
- Unique combination of catalyst and solvent
- Autoclave design that mitigates the damages on fibers and facilitate the cleaning process

PATENTED

- Valorization of the degraded resin is possible as well (oligomers) → full recyclability
- Recovery of long fibers compatible with yarning and carding
- Process adapted to different thermoplastic and thermoset resins
- Retention of the mechanical properties of the fibers
- No pre-grinding required
- Low carbon footprint

First results in a 10 L autoclave

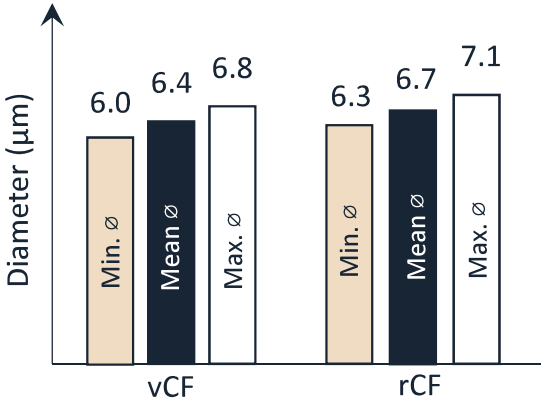
- Custom made pierced piece for support the CFRPs pieces and enable the treatment in vapor phase



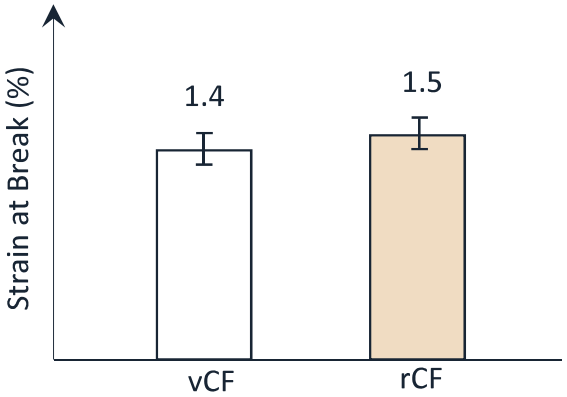
- Autoclave in Hastelloy C276 built by HP systems
- Total internal volume = 10 liters. Capacity = 1 kg of CFRPs per batch.

Mechanical properties of recycled CF

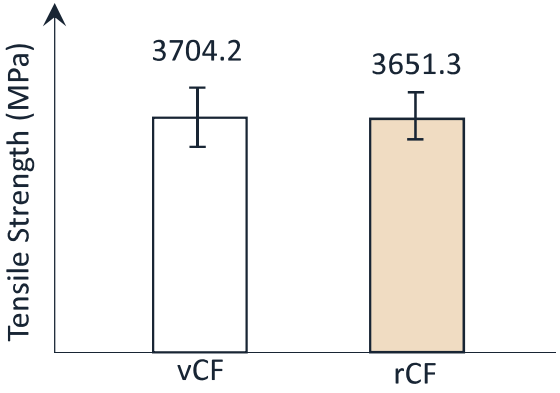
No significant variation



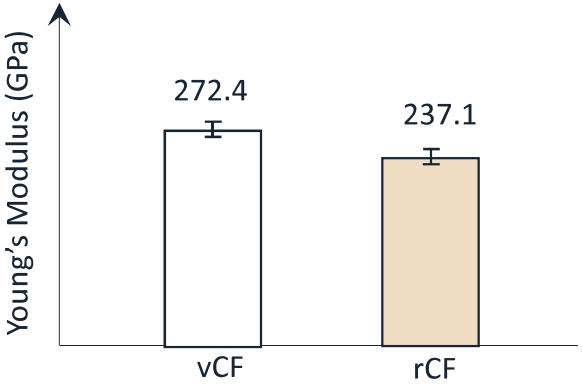
Increase of the fiber diameter. Cross section area of vCF: 32.2 µm²; rCF: 35.7 µm².



No influence on the strain at break.

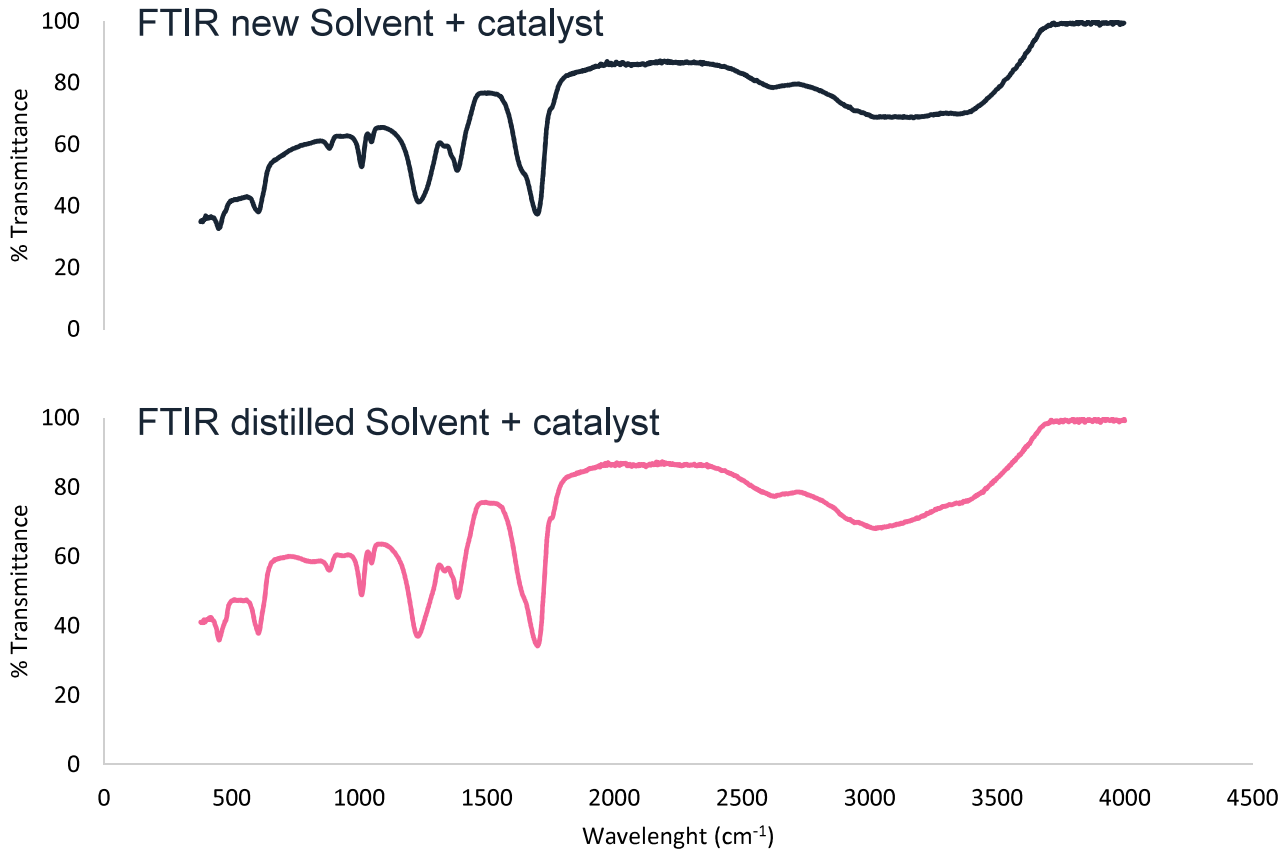


Very similar Tensile Strenght. Measured loss of ~ 2%.



Loss of 13% on the Young's Modulus.

Solvent / Catalyst recovery



No difference between the FTIR spectra of new and distilled CH_3COOH + HCl

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Chemicals can be recovered

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Up to 3 re-uses validated in lab-scale

First up-scaling objectives

The **PHYre** recycling process operates with HCl and Acetic acid a 180° C under autogenous pressure (~ 11 bars)

Currently the process is operated in a 10 l reactor

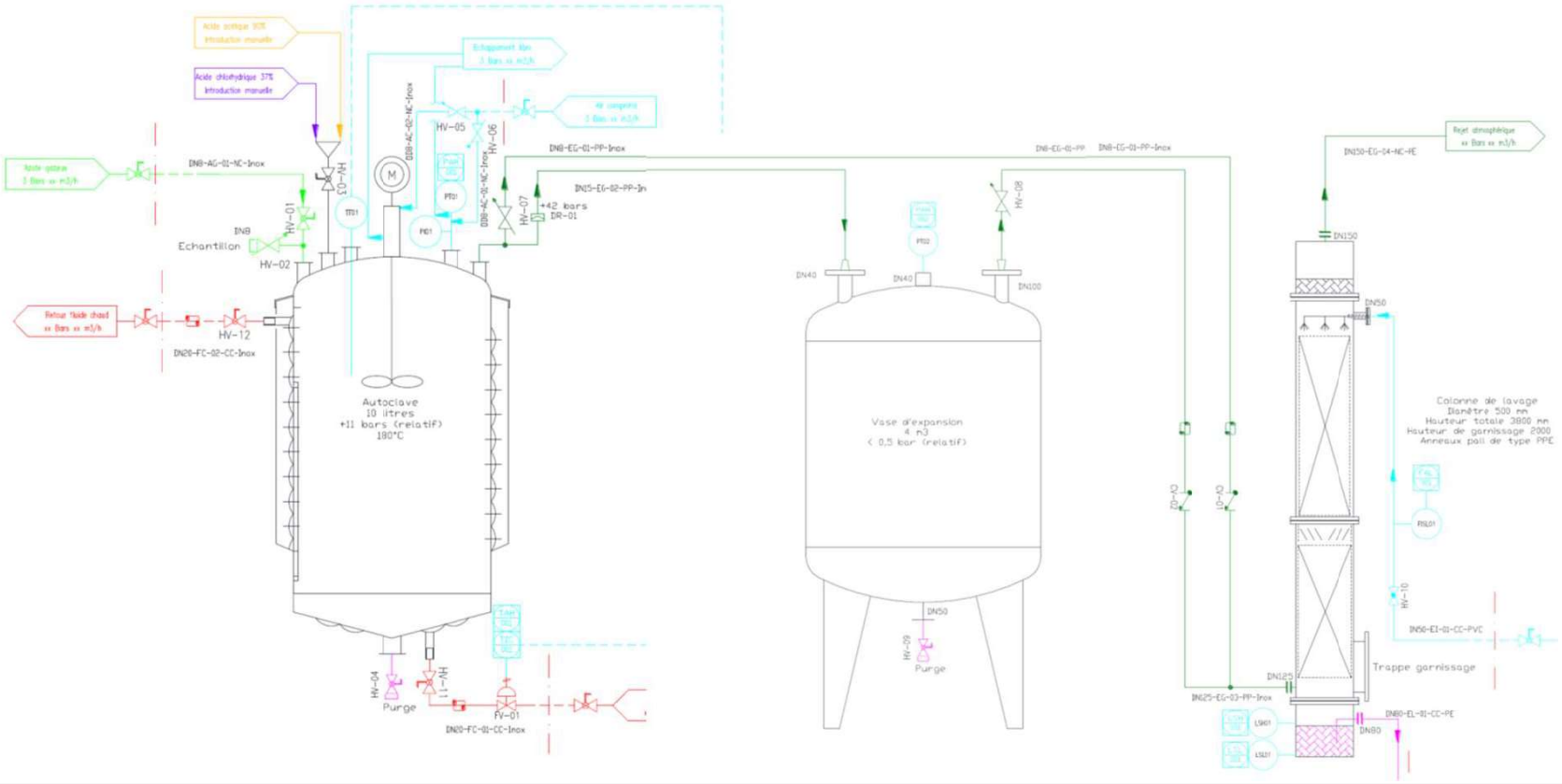
Up-scaling focusses on two aspects :

- **Safety**
  - Pressure management
  - Corrosion resistance
- Operability
  - Handling of the fibers
  - Limitation of the inter campaign time
  - Limitation of the heating-cooling time

Safety considerations : part 1

Management of vapors : expansion tank + (small) stripping column

-> **Not an option** for the 800 l demo unit

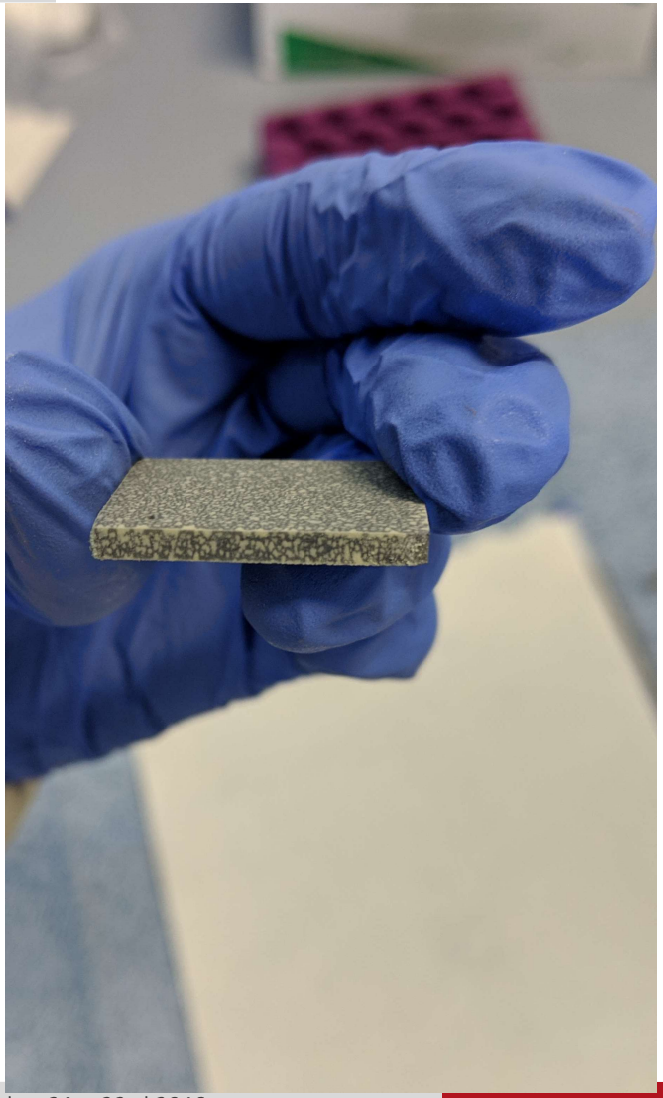


Safety considerations : part 2

Corrosion resistance : ongoing corrosion tests

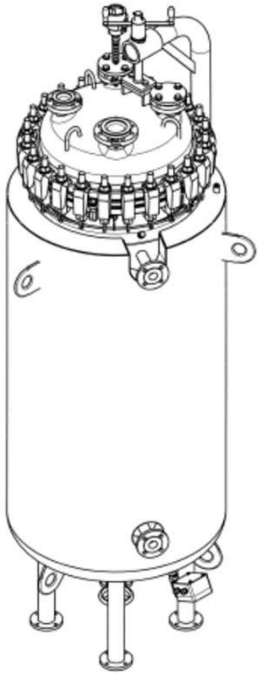
Conditions : about 50 g of metal, 15 ml EtOOH, 4 ml HCl, in a 50 ml digestion bomb, 180 ° C, 10 days

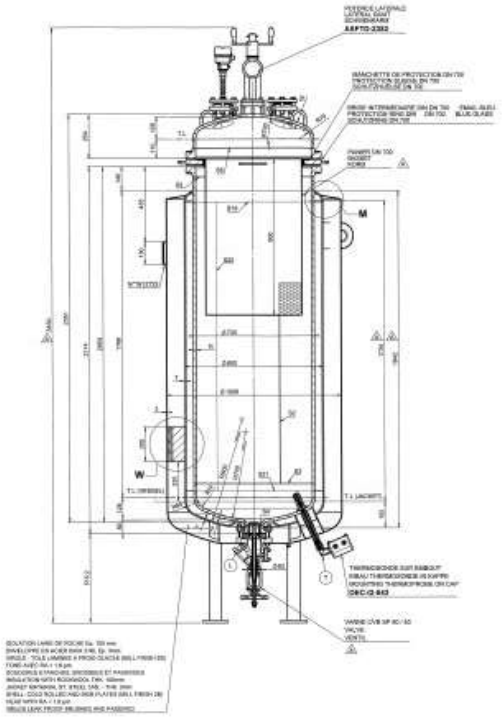
Alloy	Weight loss (%)
Hastelloy® C22	4,0
Uranus® B6 940L	4,0
Titanium 7	50



Safety considerations : impact at demo scale

800 l reactor: safety first, at the cost of operability
- Enameled steel
- Direct discharge of the vapor in a large stripping column





Impact on the process:
- Long heating and cooling phase (6 h)
- Opening and closing the reactor takes ~1 hour
- Not an option for mobile units