NATIONAL COMPOSITES CENTRE

The case for solvolysis in composite recycling

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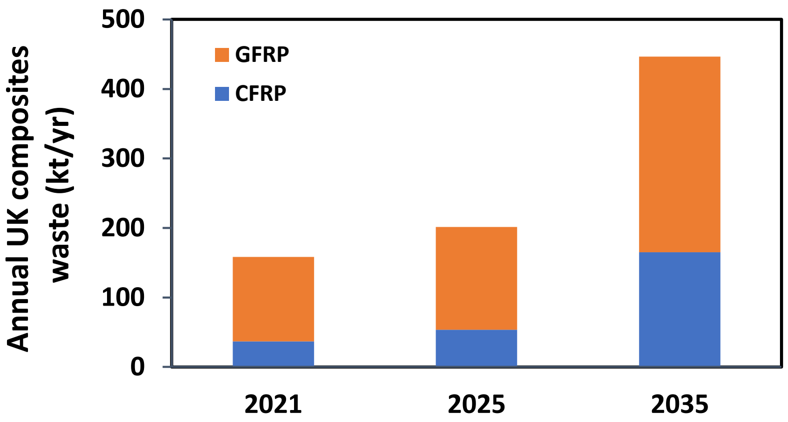
Composite recycling landscape

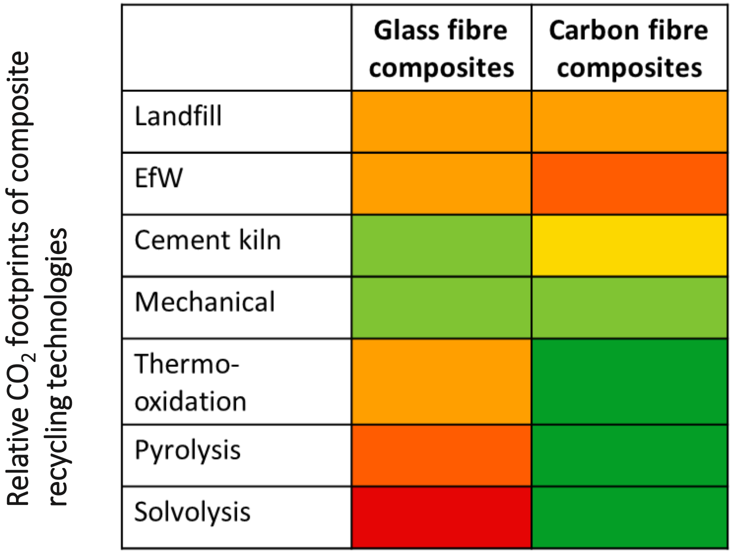
Around **110 000 tonnes** of FRP is produced in the **UK/year**

On average, **only 15% of this is recycled** at EOL

For composites to remain relevant in a **net zero future**, a lot **more** of it **needs to be recycled**

Due to value, lifecycle, and stability differences, **CFRP** and **GFRP** composites require **different treatment methods**





*information and figures kindly provided by the NCC Sustainability team, including the work of K. Pender, T. Young, and V. Summers

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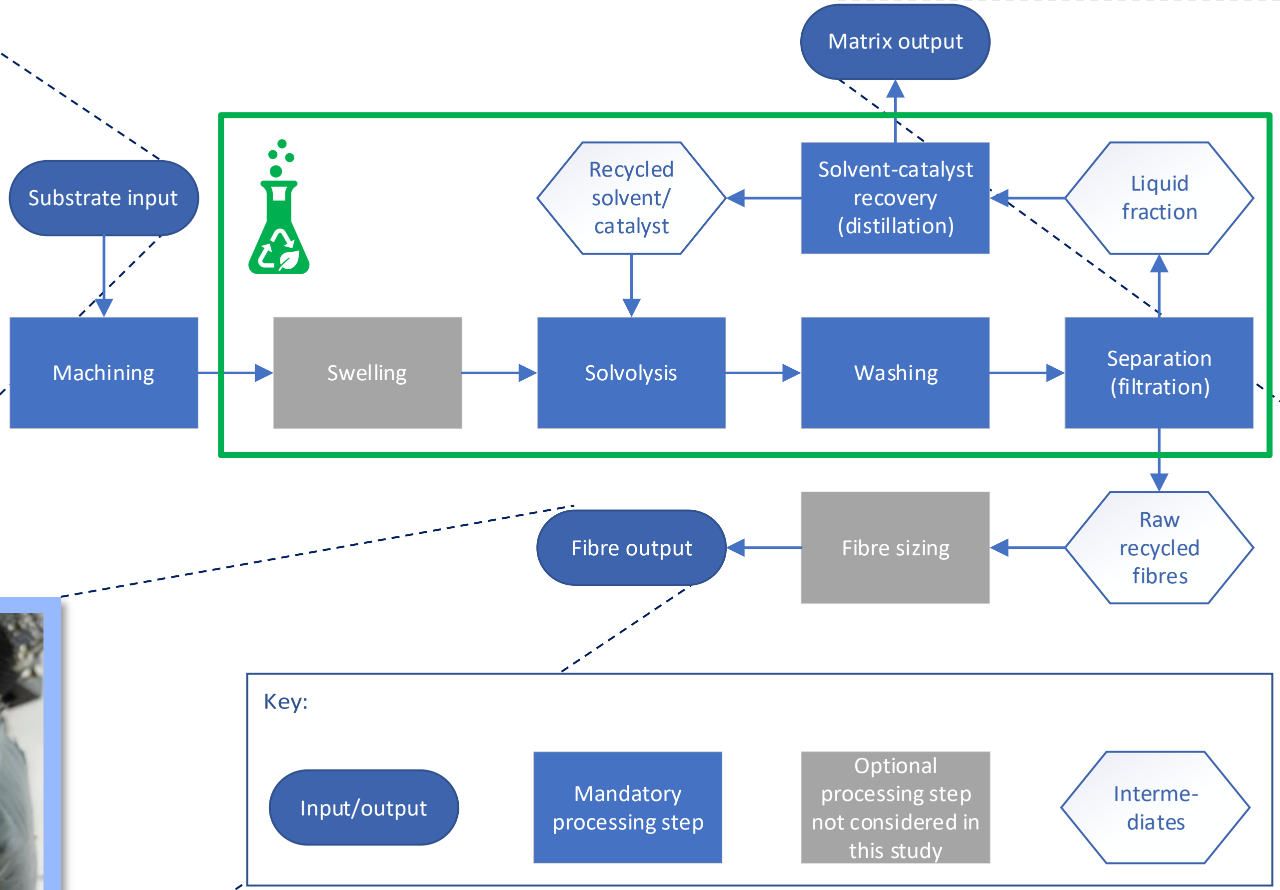
What is solvolysis?



- **Composite recycling technique:**
- Best in class recovered fibre performance
- Versatile but complex
- Thought to be more expensive than other techniques

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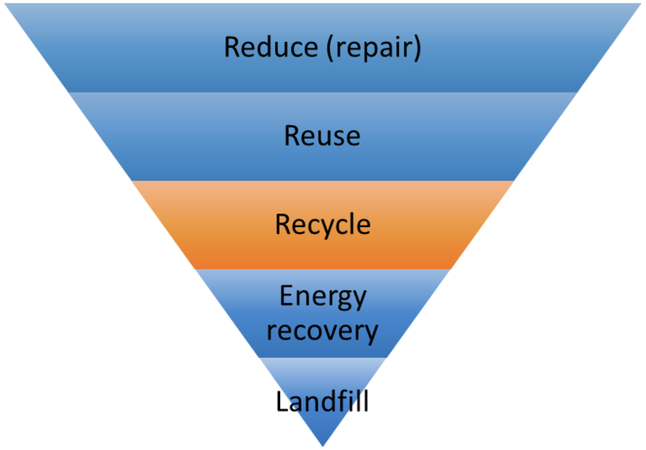
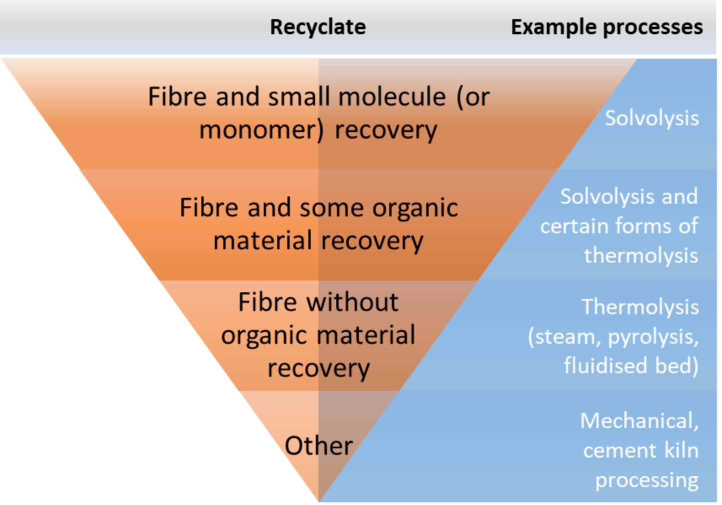
Where does solvolysis fit in?

Waste hierarchy
by *circularity*

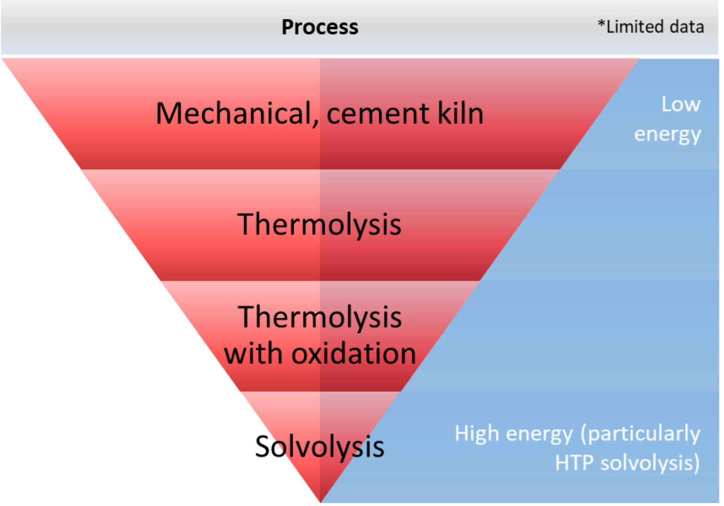


FRP recycling hierarchy
by *circularity*



The ideal world…

FRP recycling hierarchy
By *cost* and *energy intensity**



…reality?

- Solvolysis has three key **advantages**: **max. recyclate value** (near **pristine fibre** performance **+ matrix recyclates**), **applicability to all fibre** types (mild conditions), and **tunability**
- The major **disadvantages** are the **high cost** (energy + financial), so high environmental impact, **complexity**, **and low TRL**
- Solvolysis is most suitable for **three composite scenarios**:
- (1) **High performance fibres** (i.e. carbon and aramid) where maintaining this for 2nd life is critical, (2) composites with **valuable or specially degradable polymers**, and (3) any composites **mandated to be** recycled into **fibres *and* matrix**

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Commercially relevant solvolysis

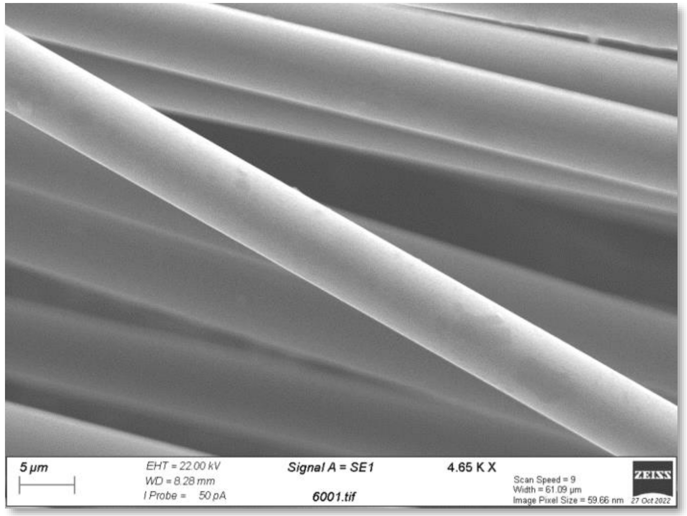
AFRP (Kevlar-49)/CFRP hybrid (infusion)
Racing kayak
Bisphenol A–derived **epoxy** with a *design for degradation* hardener, **Recyclamine** by Aditya Birla

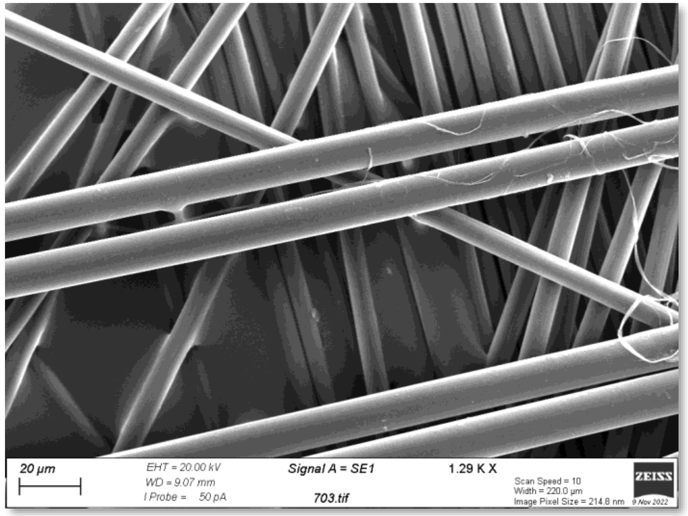
CFRP (filament wound)
Hydrogen tank (pressure vessel)
Bisphenol A–derived **epoxy** by Huntsman

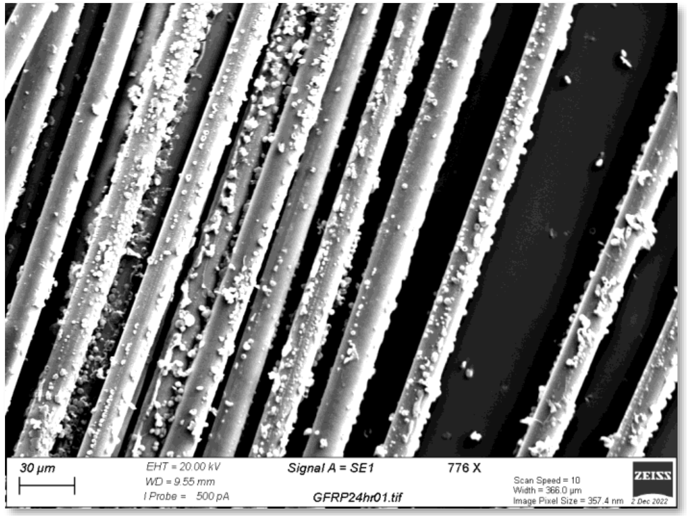
GFRP (EOL mixed wind blade, shredded)
Wind turbine blade
Bisphenol A–derived **vinyl ester**, *determined by FTIR spectroscopy*

Results: fibres and degradation%

- In most cases, TGA indicates >95 wt% of organics* are removed by solvolysis/washing
- EOL wind blade, best case ~85 wt% organic removal—affected by balsa and unidentified organic material?







*organics = mostly matrix polymer, also includes other materials that can thermally decompose on TGA: sizing and core materials (balsa)

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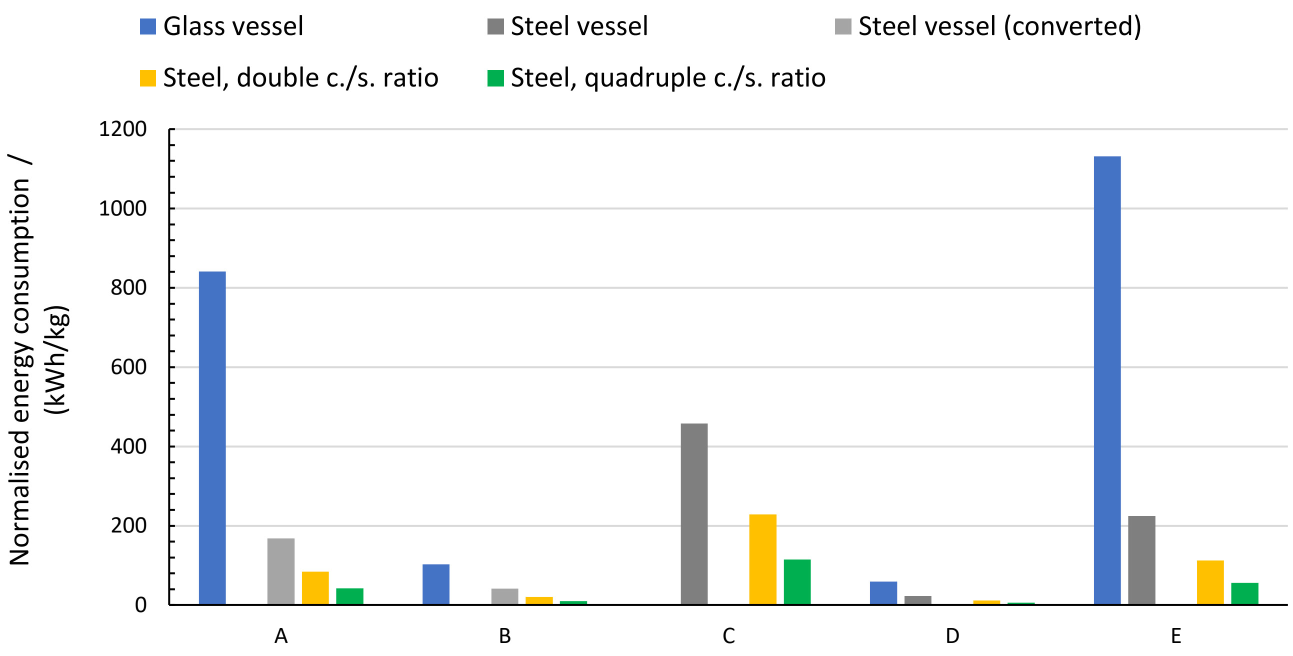
Results: energy measurement



A: BnOH-K_3PO_4, **200 C, 1 bar, 20 h**

B: MEA-KOH, **150 C, 1 bar, 1.5 h**

C: Ac/MeOH-KOH, **280 C, 80 bar, 1.5 h**

D: H_2O-AcOH, **80/90 C, 1 bar, 1.5 h**

E: MEA-NaOH, **200 C, 1 bar, 20 h**

- Composite/solvent (c./s.) **ratio not optimised**
- Previous results indicate **higher ratios** can give **better degradation yield**
- **Yellow and green bars** indicate **estimates** with optimised c./s.
- **Bigger reactors** can be much **more energy efficient** (e.g. 6-fold…)

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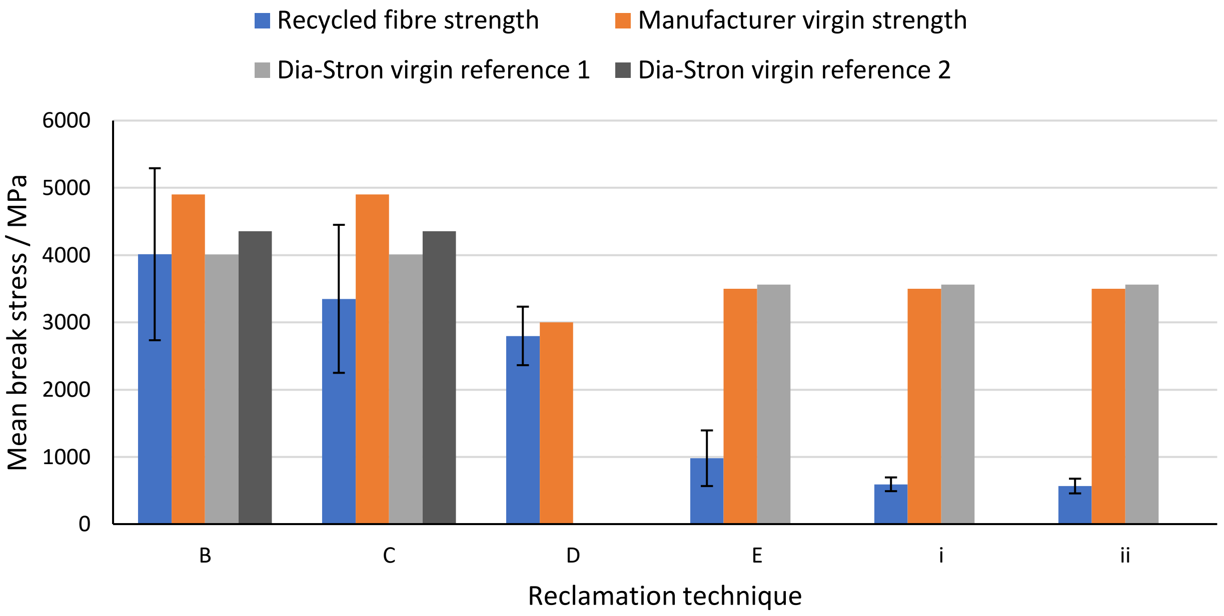
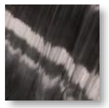
Results: single fibre tensile testing

Key
- B–E correspond to the previous solvolysis conditions
- i = steam pressolysis on the same EOL blade substrate as E
- ii = pyrolysis on the same material

Fibre diameter
E = 18.7 µm, i = 21.0 µm, ii = 18.7 µm

Key points
- Comparing **B/C** with the average of the two reference values for virgin T700:
- **B** (low temp solv.) = **96% of virgin strength**
- **C** rCF strength is **20% poorer**, therefore lower temp. preferred?
- All **rGF** was very **low performance**
- Consistent stiffness and strain data





There is more to fibre quality than strength (and other mechanical properties)…

…fibre format
- Continuous filaments
- Woven sections
- Short fibres
- Currently little interest in ‘long’ but discontinuous

And surface properties
- Surface energy
- Sizing?

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Summary

- **Validated four previously reported solvolyses** and one new process on one of **three different commercially relevant FRP substrates—**clean fibres that could be re-purposed into 2nd life composites were isolated
- The **real EOL wind blade waste was most challenging**—unknown materials make things difficult
  - Crucial to match the chemistry to the material
- **Single fibre tensile testing** data revealed that **mean rCF strength was ~96% of vCF**
  - **rAF (K-49)** was similarly high performing*
  - **rGF strength was very poor** but the solvolysed rGF's mean strength of 28% (of virgin) was better than those fibres reclaimed from steam pressolysis (17%) and pyrolysis (16%)
  - Much of this rGF damage is attributed to service life and shredding
- **Reactor** design and **size** has a **big influence on energy efficiency data—it's difficult to** extrapolate up to a pilot scale to **compare with other reclamation techniques**
- Like-for-like comparison of different conditions indicates that **newer, low temperature solvolyses can be 10x more energy efficient** than supercritical solvolyses that have been used in LCA comparisons
- Initial testing of the matrix recyclates has been preformed but much more needs to be done…
- **Solvolysis can be fairly straightforward** *when you know a lot about your substrate composite*

*92% of vAF strength according to manufacturer. No comparable baseline SFTT data was available.

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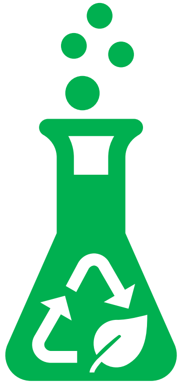
Final remarks

Where does solvolysis fit?

- Where high performance rCF or rAF is required
- Where the substrate composite is well identified
- Any next generation composite with design for degradation polymers
- As one part of the future composite recycling landscape



What next?

- **Scale:** with a 50 L pilot reactor, energy measurements should support **meaningful LCA and TEA** data
- **Tough composites:** aerospace dominates the market for high performance carbon, can we validate w/ tougheners?
- **Matrix recyclates**: there is value in matrix recyclates, but how much? Much more work to be done…
- **2nd life** composite **demonstrators**: prove the value in the circularity to drive investment to bigger scales…

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