Towards sustainable composites?

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Learning objectives

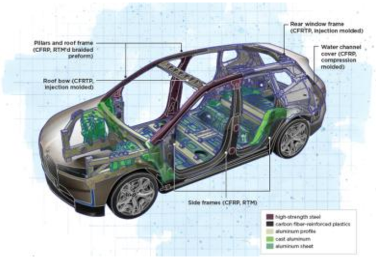
- Lower impact raw materials
- Give over-view of recycling
- Options for re-use
- Towards a circular economy for composites?

Composites will see rapid growth as enablers towards 2050 SSPs

Main sectors

Sub sectors

Aerospace	Wind	Automotive	Hydrogen	Infrastructure	Consumer
☐ Civil wide body	☐ Wind on-shore	☐ Super cars	☐ CNG tanks	☐ Concrete rebar	☐ Bicycles
☐ Civil narrow body	☐ Wind off-shore	☐ Premium	☐ Auto Hydrogen	☐ Buildings	☐ Marine
☐ Evtol/drones	☐ Tidal	☐ EVs	☐ Aero Hydrogen	☐ Train	☐ Consumer
☐ Military	☐ Fuel cells	☐ Other	☐ Ground Hydrogen	☐ Other	☐ Other
☐ Other	☐ Other		☐ Rail Hydrogen		
			☐ Other		

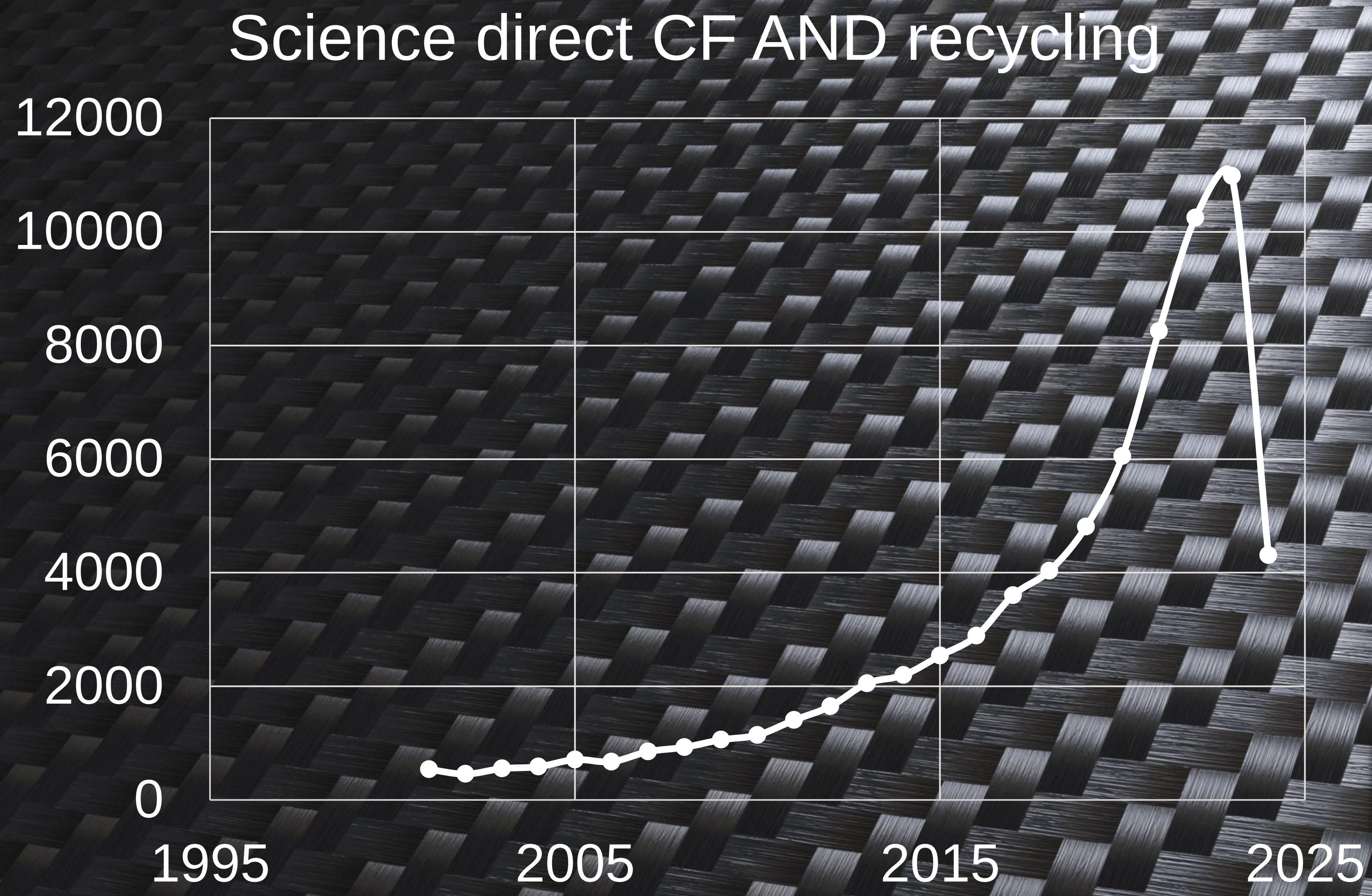


Needed industrial sector segmentation 1980-2050 for EPFL model

[Scenario modelling of the carbon fiber industry in the transition to NetZero 2050 – E4S]

[Re-wiring CAPEX and Finance in Circular Value Chains: A Case Study of Advanced Materials – E4S]

CF AND recycling: Science Direct



Question 1:
Does this correspond to an equivalent growth in CF recycling and recovery?

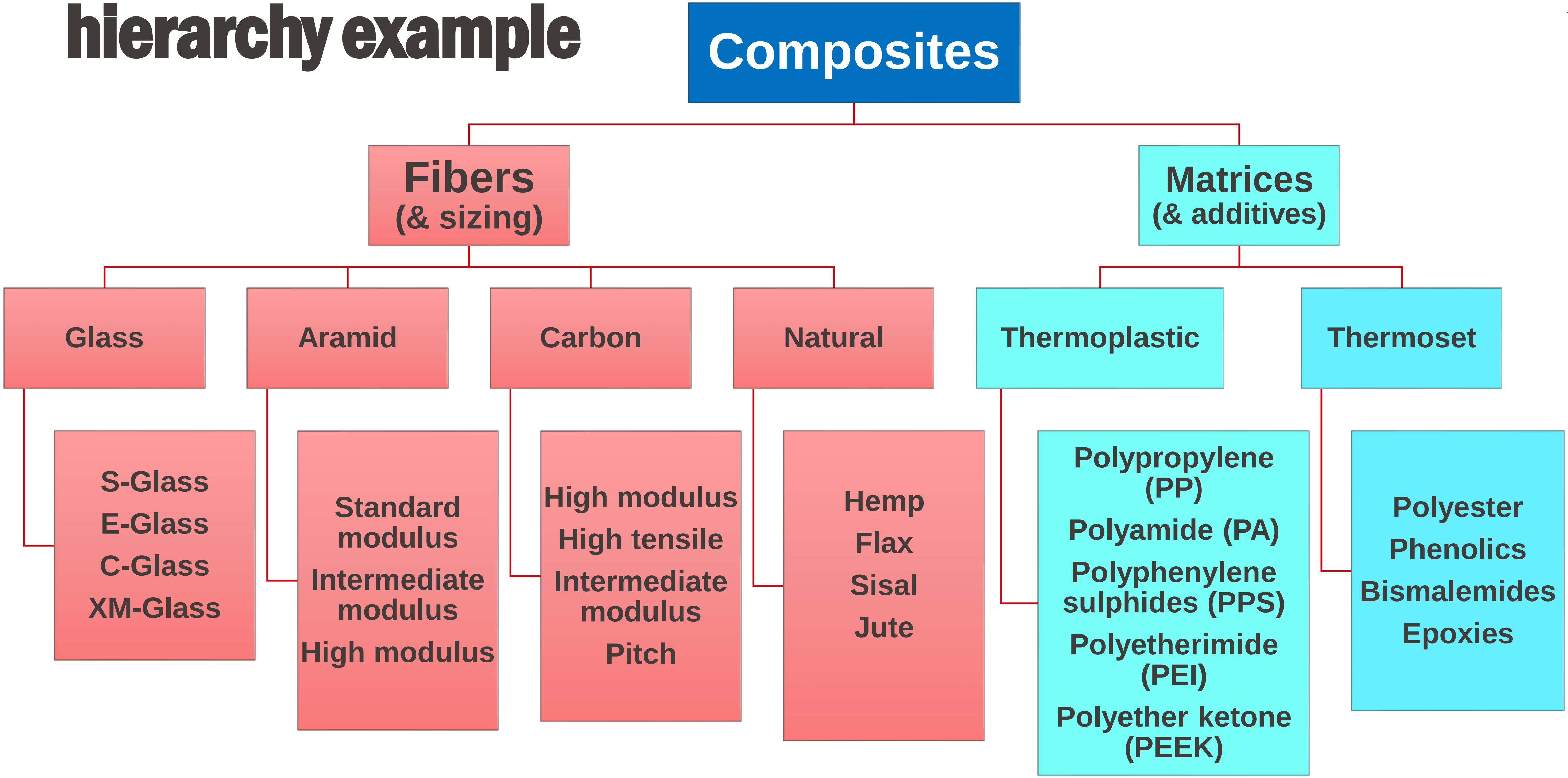
Question 2:
If NO, then why?

Question 3:
What is the degree of circularity of composites?

Question 4:
Can S&T solve this problem?

MSE-440

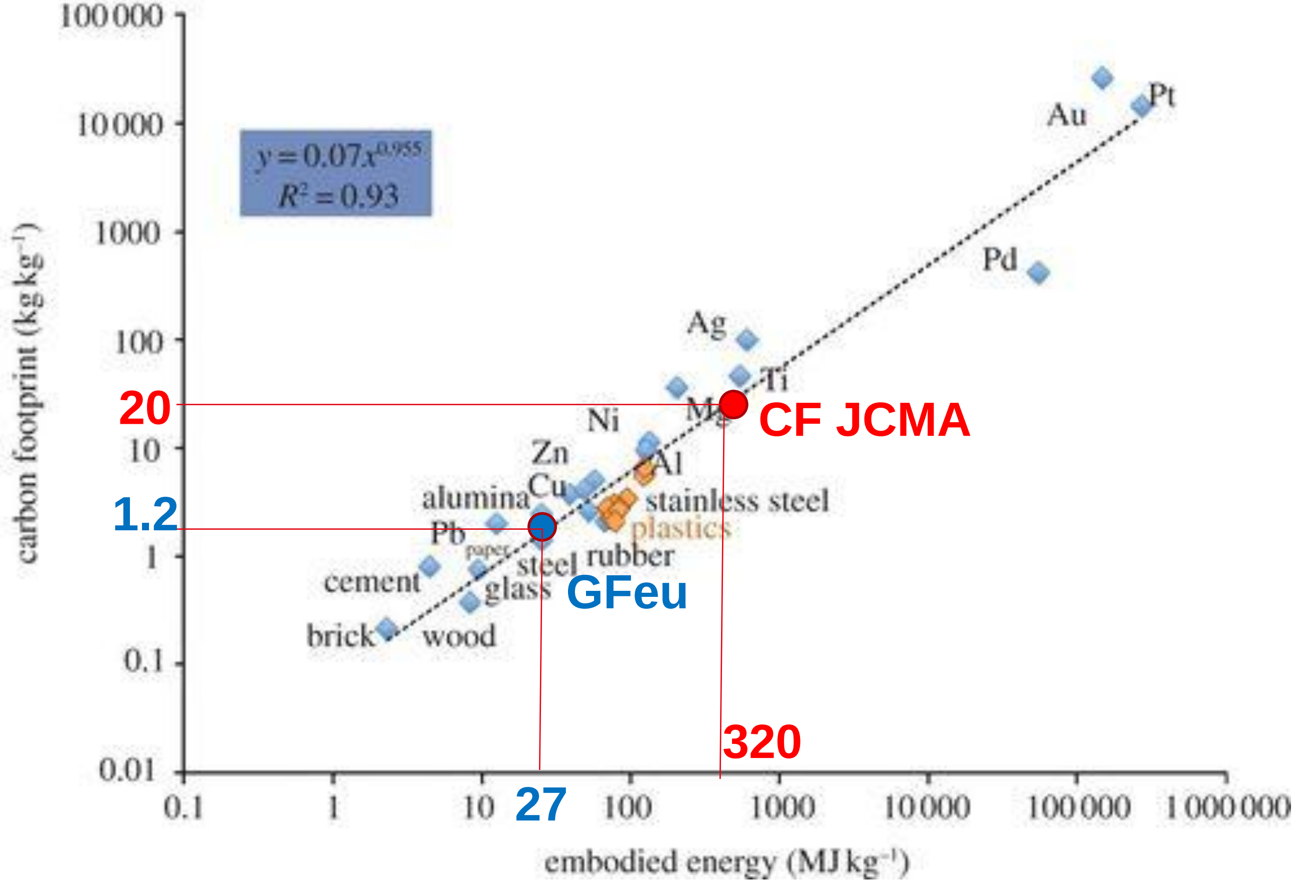
Polymer composite hierarchy example

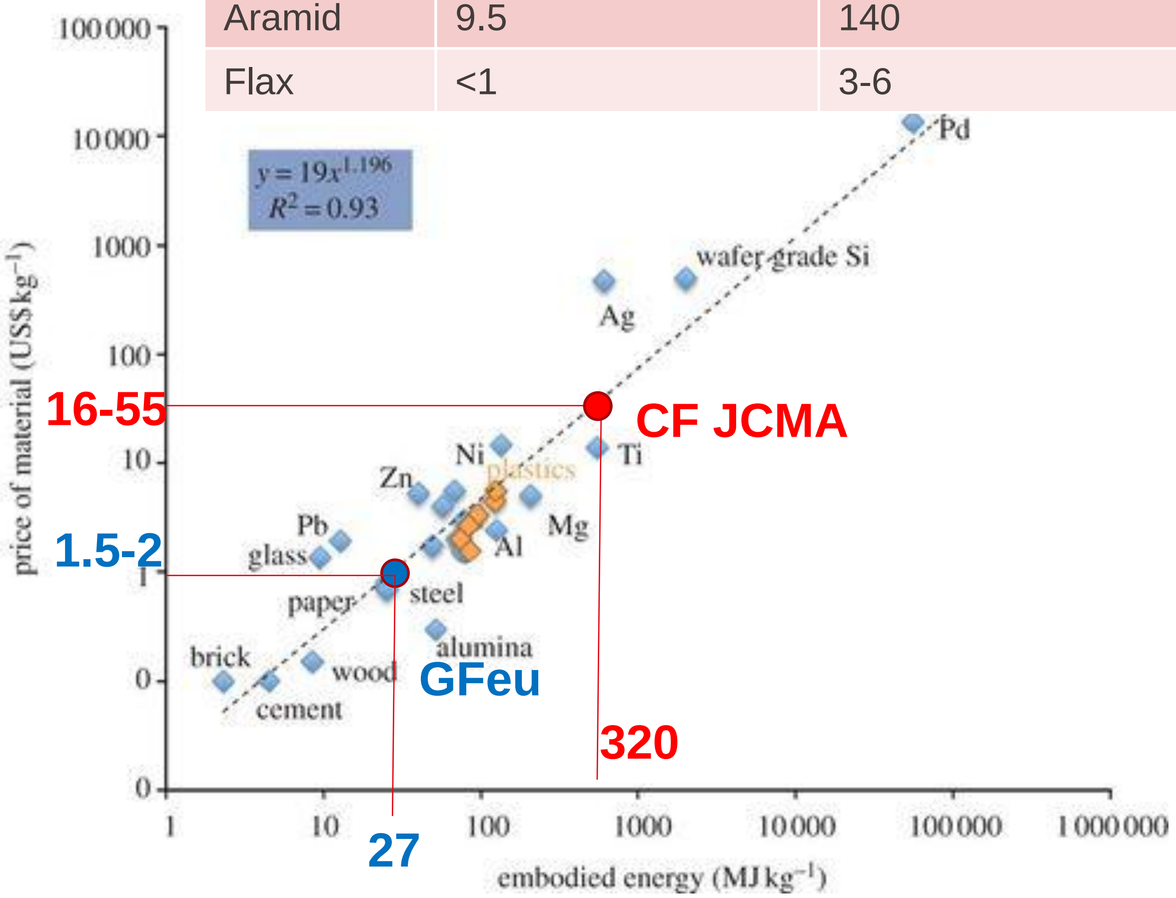


EPFL

CO2e vs. MJ/kg vs. $/kg

Fiber type	$kgCO_2e$/kg dry fiber	Energy Intensity MJ/kg
Glass	1.2	27-30
Carbon	19.8	318
Aramid	9.5	140
Flax	<1	3-6

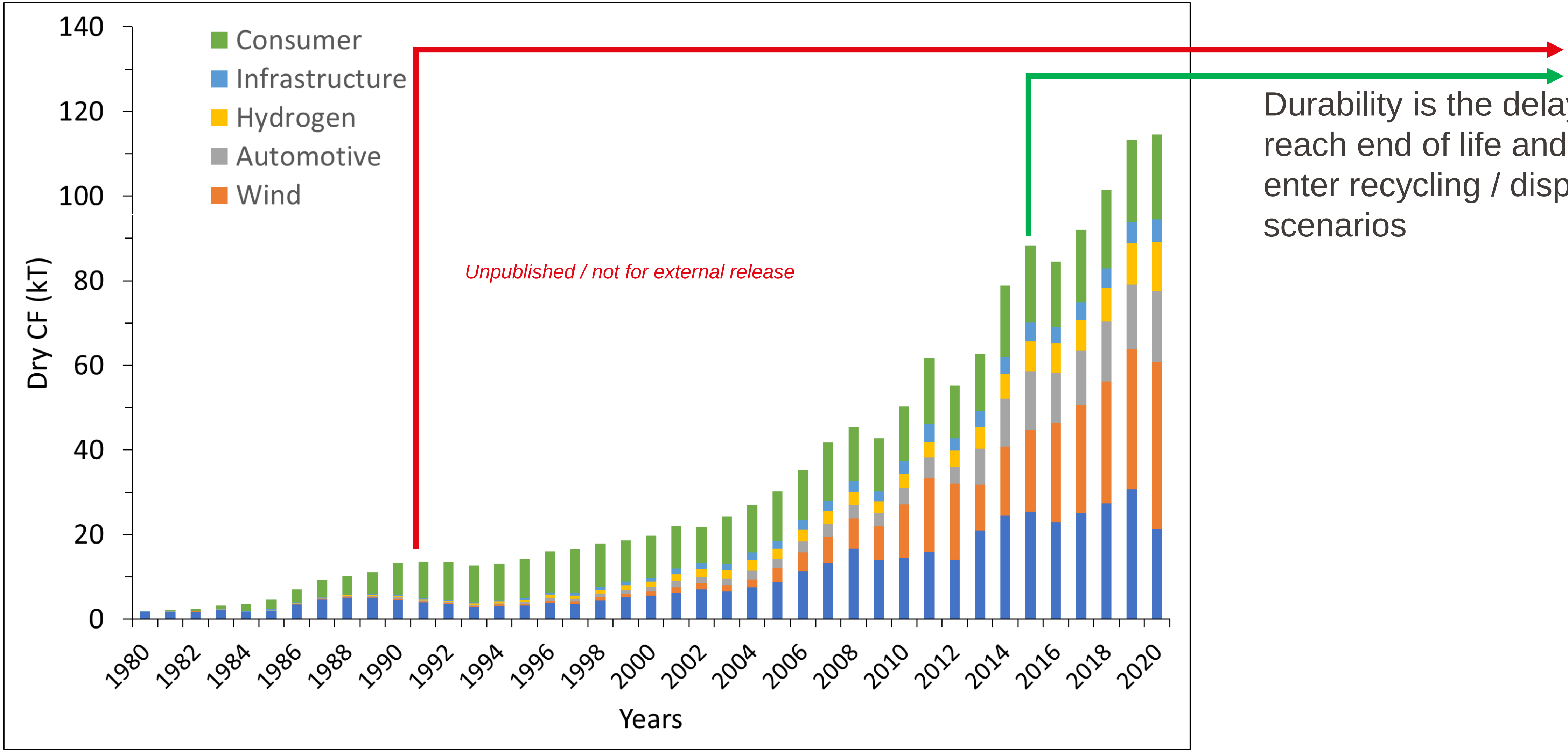




Energy intensity of derivatives and conversion drives environmental foot print and economics

The energy required to produce materials: constraints on energy-intensity improvements, parameters of demand (royalsocietypublishing.org)

Dry Carbon Fiber Historical Demand 1980-2020

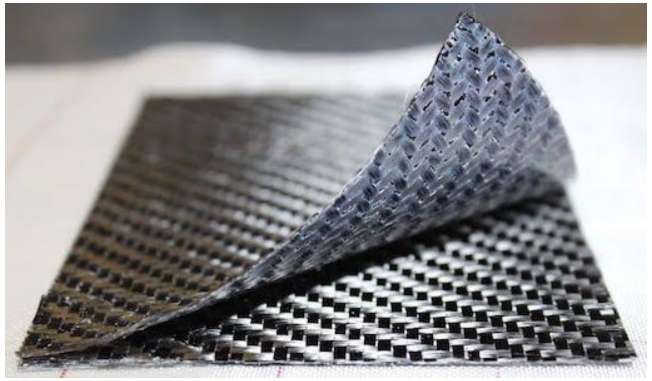


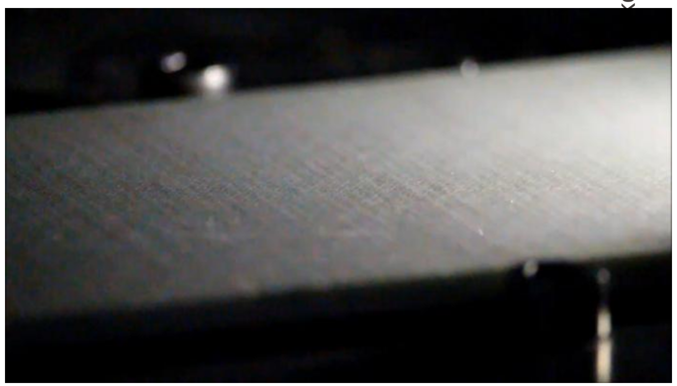
Durability is the delay to reach end of life and enter recycling / disposal scenarios

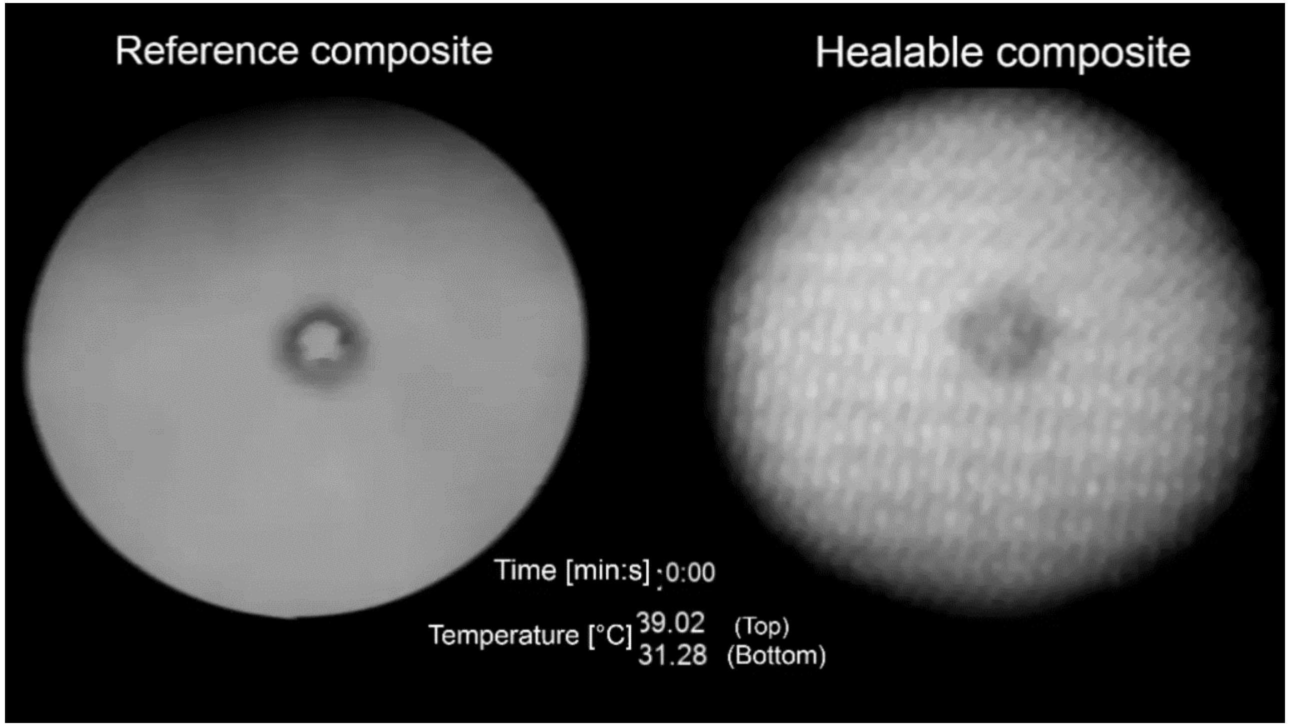
Durability: Self-healing composites

- Self-healing technology to
  - reduce maintenance cost,
  - reduce manufacturing defects
  - extend the lifetime of composites.
- 12 years of research at EPFL
- HealTech™
  - New type of resin giving composite materials the ability to heal resin micro cracks and de-laminations in a few minutes.
- Composite structures (resin damage) can be fully repaired on site as the parts stay structural during healing.

CompPair

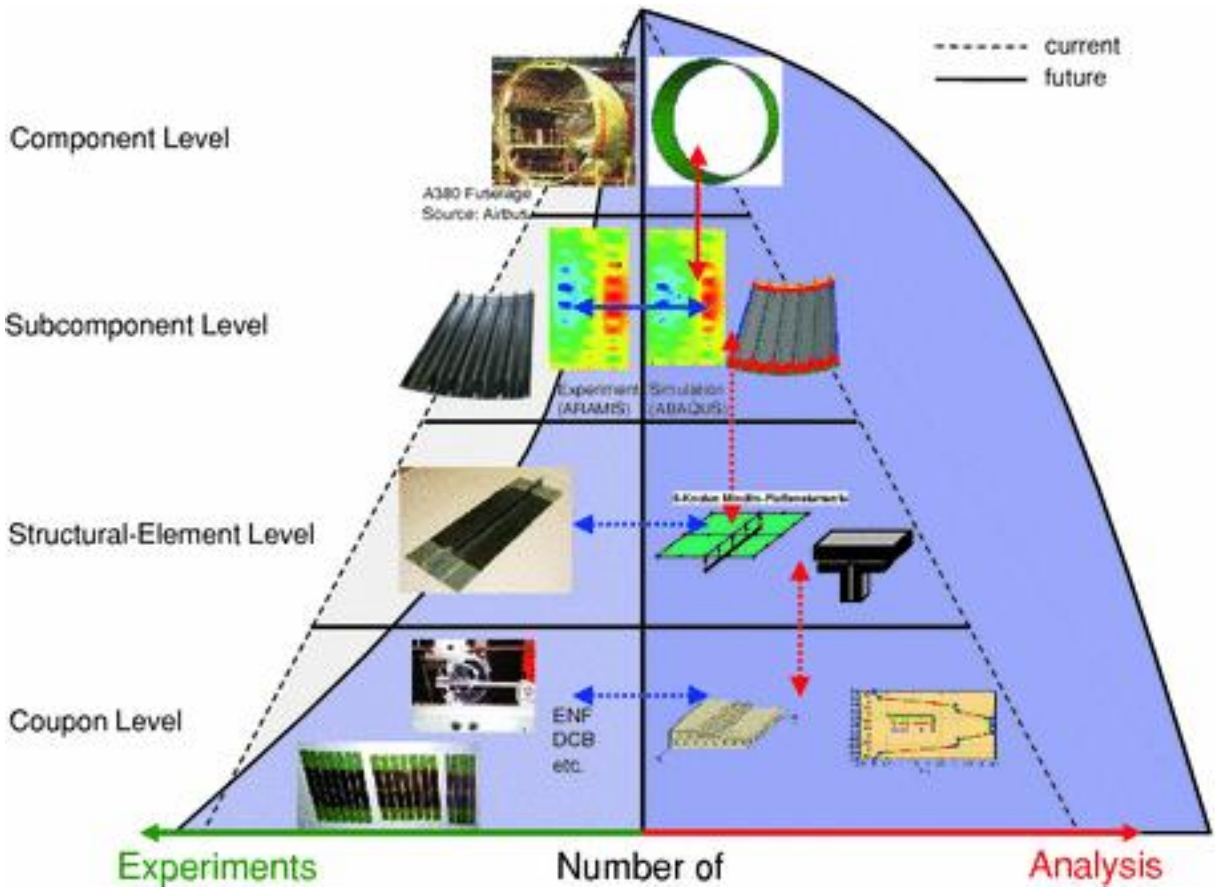


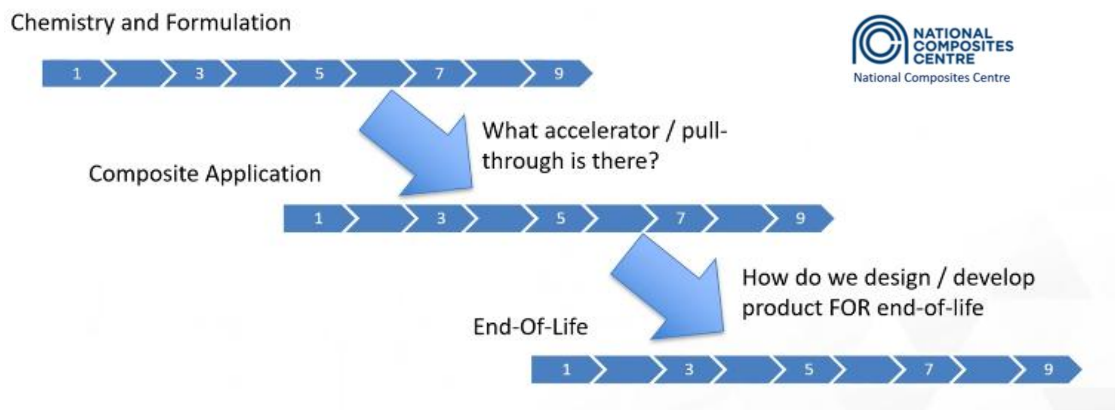




[Welcome - CompPair Technologies Ltd - Healable composite](#)

Virtualization to compress technology readiness





- Testing pyramid needed to approve CF materials
- Requalification through TRLs across supply chain (material, design, part making, ELV).
- Need concurrent TRL processes to reduce time to market and 1st revenue.

Validation Approach for Robust Primary Carbon Fiber-Reinforced Plastic Structures | SpringerLink

Overview - Towards sustainable composite solutions

- Bio-based / lower impact materials
- End of life and recycling
- Additive manufacturing
- Unmet challenges

Natural fibers

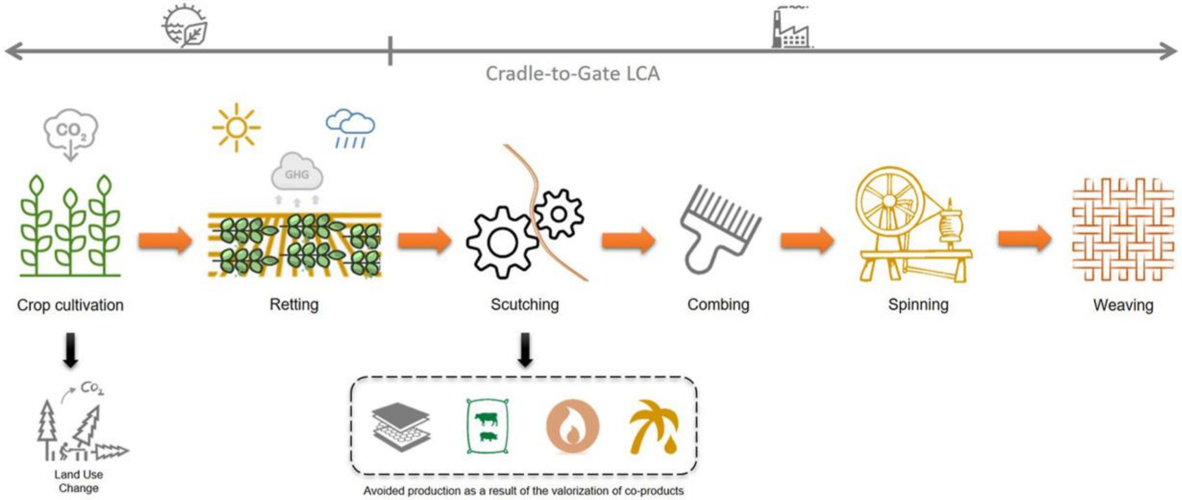
Wakeman

- Flax (42 GPa), hemp, sisal, bamboo, rice husk



Energy Intensity ~ 3-6 MJ/kg

- Bcomp, EPFL spinoff
  - European flax for TS and TP composites
  - Improved safety with non-catastrophic crash behaviour
  - High vibration damping, specific bending stiffness and fatigue resistance
  - Novel design options – colouring and natural translucency
  - Radio transparency



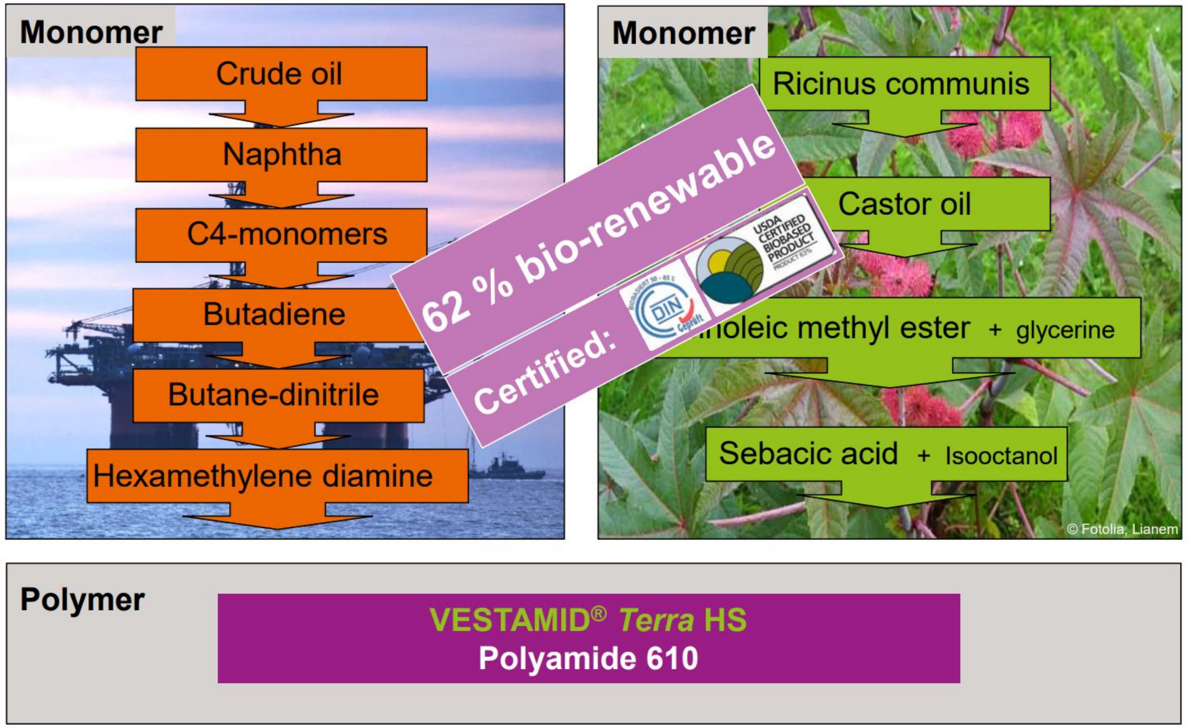
Recycling of natural fiber composites: Challenges and opportunities - ScienceDirect

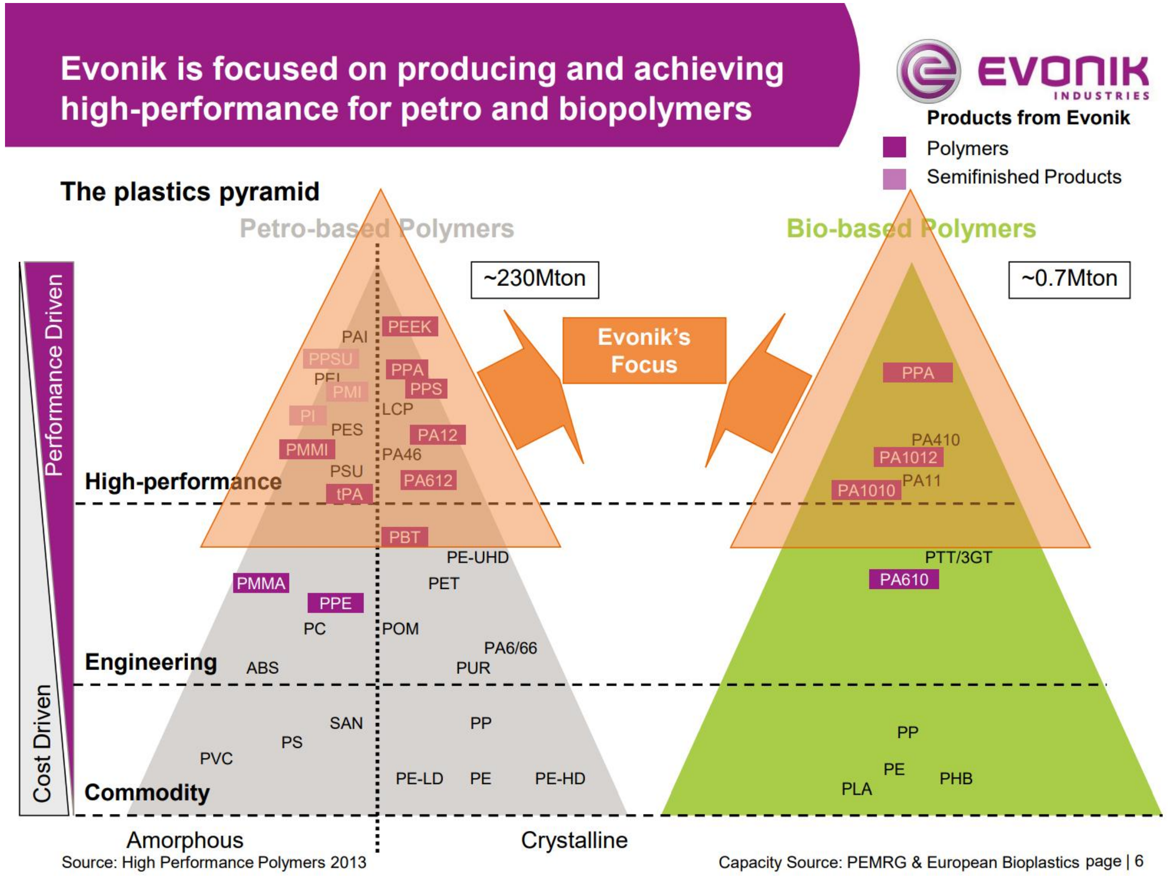
https://www.bcomp.ch

Flax fiber for technical textile: A life cycle inventory - ScienceDirect

Castor bean products for bio-based PAs

- Bio-based polymers from non-food stock sources polymerized via renewable energy
- Bio-based does not mean biodegradable
- Bio-polymers are not recycled polymers





Amodel® Bios | Solvay

Terra 2013 (bio.org)

Bio-based epoxy

- Bio-based epichlorohydrin (ECH) from vegetable glycerol derived from biodiesel and oleochemical production
- Bio diesel made from many types of plant oil
- Chemical intermediate for epoxy resin

Epicerol® is 100% bio-based epichlorohydrin (ECH) produced by an innovative technology from Solvay. Based on renewable glycerol, Epicerol® is the most sustainable ECH in terms of CO2 emis-sions and process environmental performance. It is a chemical intermediate for a wide range of industries, including epoxy resins for coatings and composites.

Epicerol® earns Sustainability Certification | Solvay
www.solvay.com/en/press-release/solvay-epicerol-earns-roundtable-sustainable...

Green resins: Closer to maturity | CompositesWorld

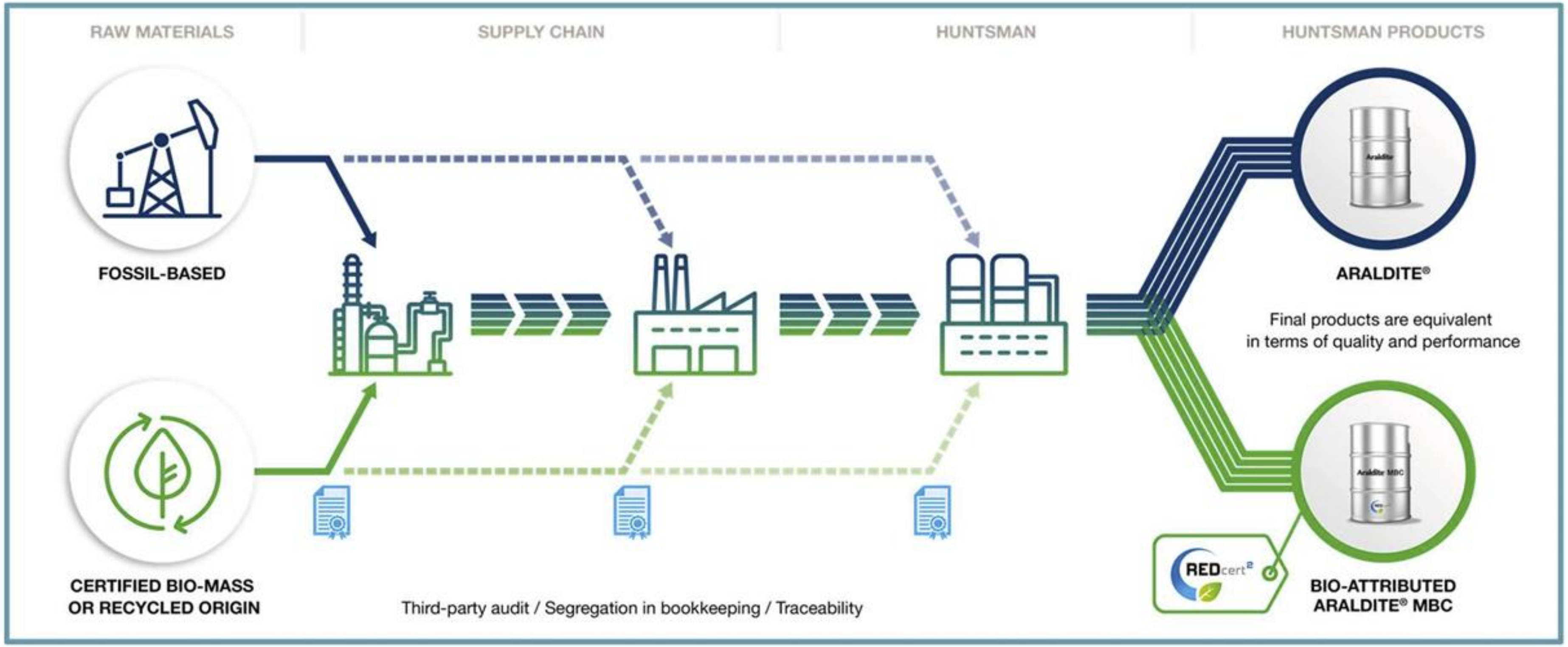
Epicerol® earns Sustainability Certification | Solvay

Glycerol from biodiesel production: Technological paths for sustainability - ScienceDirect

HUNTSMAN
Enriching lives through innovation

Wakeman

Verified substitution of fossil raw materials by sustainably certified biomass



Mixing

Tracking

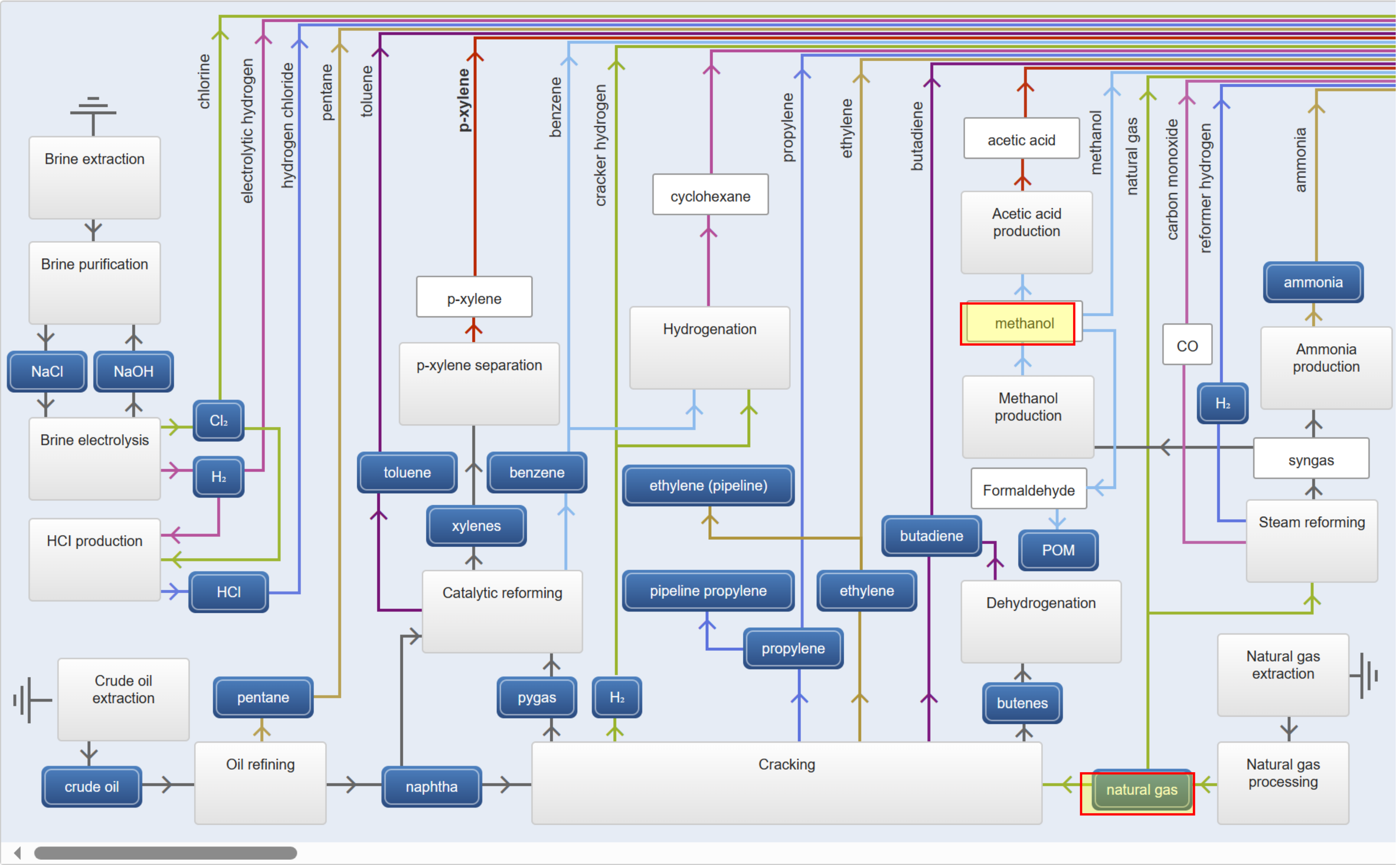
Verifiying

Mixing renewable and fossil raw materials,
Tracking the quantities in the books,
Verifiying through an independent certification

15/11

huntsman_mbc_presentation_0324.pdf (ost.ch)

Bio-methane and bio-methanol for bio-attributed polymeric products

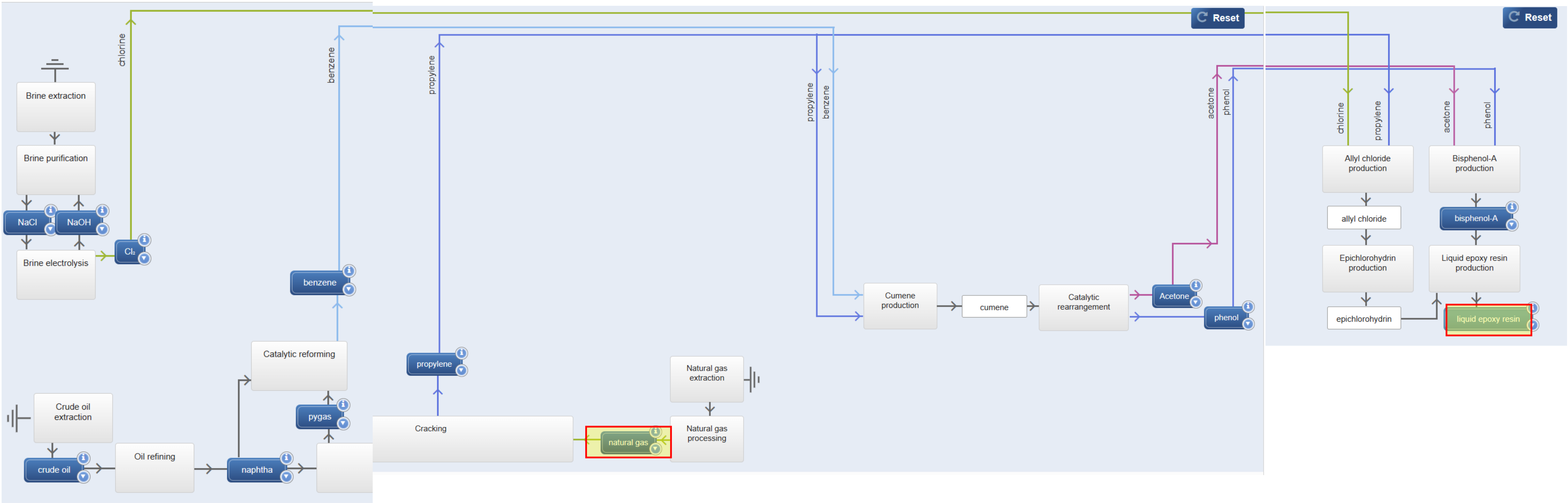


[Synthetic methane could smooth the path to net zero (nature.com)](https://www.nature.com)

“ The chemical industry uses a small set of raw materials or feedstocks to produce tens of thousands of products,…

[Eco-profiles (plasticseurope.org)](https://plasticseurope.org)

Bio-methane to propylene to phenol to bisphenol-A to mass-balanced epoxy resin

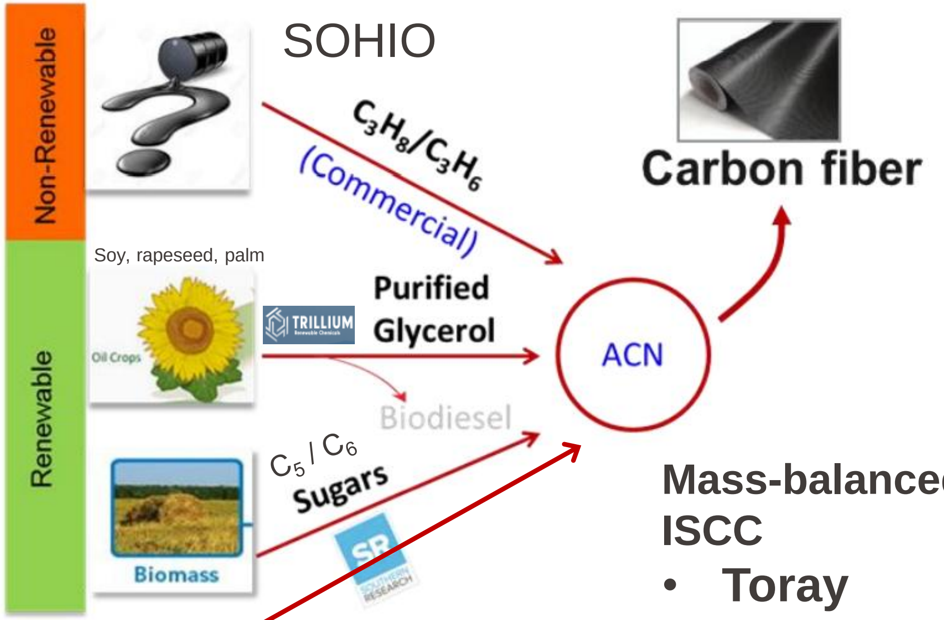


Carbon fiber manufacture

Block A
- Stage 1: AN precursor (propylene & ammonia)

Block B
- Stage 2: PAN polymerization
- Stage 3: Solution spinning

Block C
- Stage 4: Stabilization / oxidation
- Stage 5: Carbonization

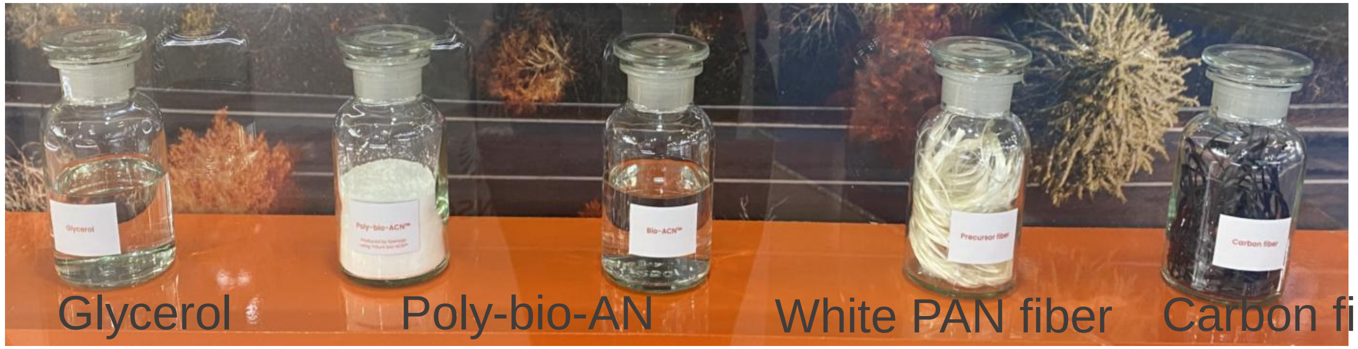


Bio-methane to bio-attributed propylene

Routes to ACN

Mass-balanced ISCC
- Toray
- Teijin
- Ineos

SYENSQO

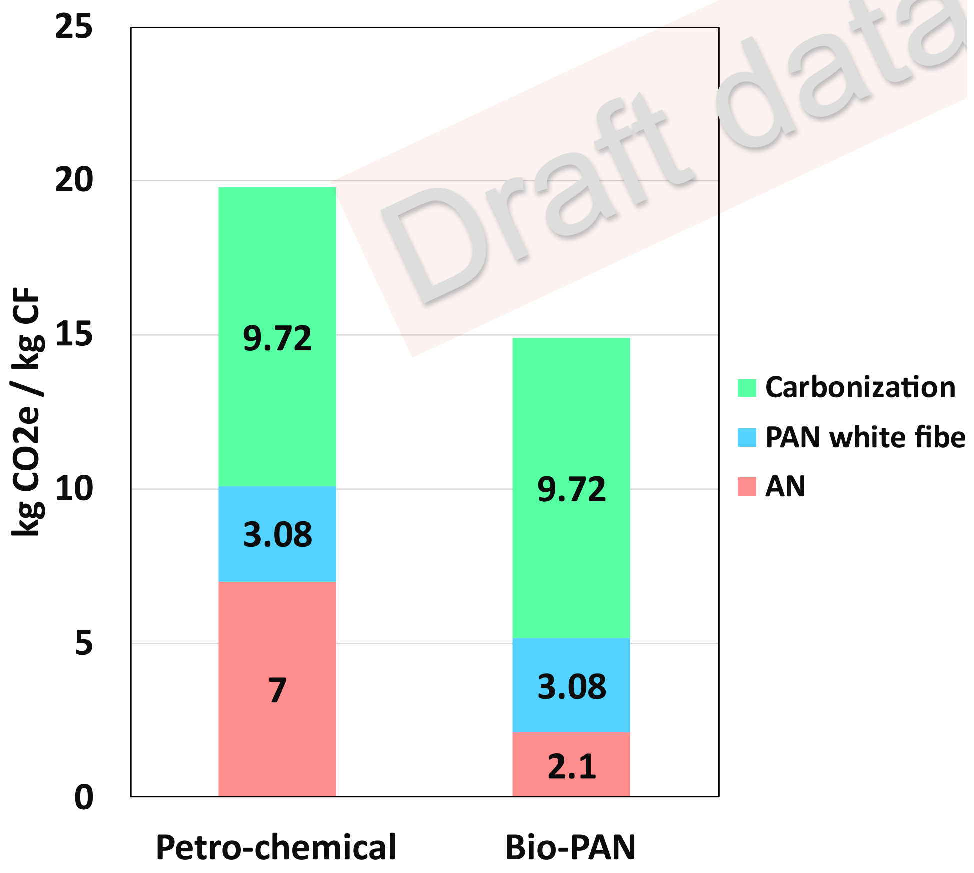


"Syensqo to Showcase Sustainable Mobility and Technology Collaborations at JEC 2024." Accessed: May 14, 2024. [Online]. Available: https://polymer-additives.specialchem.com/news/product-news/syensqo-jec-world-2024-carbon-fiber-applications-000233226

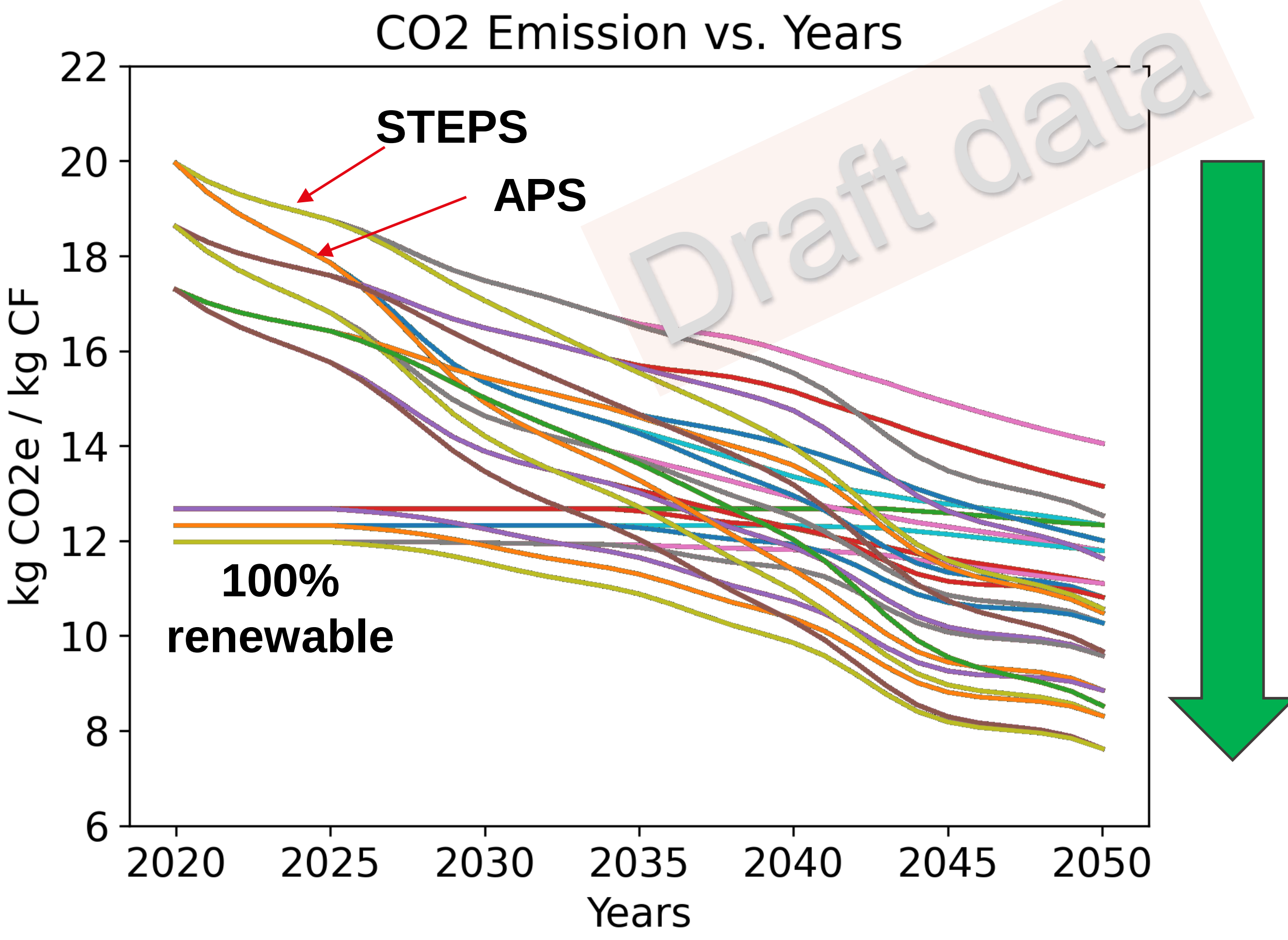
kg CO2e/kg CF

Wakeman

JCMA 19.8 $kgCO_2e/kg$ CF [4]



JCMA 19.8 $kgCO_2e/kg$ CF



Preliminary data subject to revision

Overview - Towards sustainable composite solutions

- Bio-based / lower impact materials
- End of life and recycling
- Additive manufacturing
- Unmet challenges

EU legislation

- EL vehicle for automotive
- None for aerospace
- Landfill legislation
  - Current: Waste Framework Directive 2008/98/EC
  - Different EU state implementation; legal gaps
    - The need of harmonized EU provisions is highlighted by different approaches taken up by some EU Member States.
    - Belgium, Denmark and Austria allow landfilling in general
    - Germany & Netherlands established a ban on composite materials waste landfill
  - Future: Directive (EU) 2018/850: stronger limits

show.cgi (uvt.nl)

Policy - European ELV legislation

- “Polluter-pays”
  - Vehicle producers meet all or significant part of ELV treatments, collection & recycling network
- Component and material coding standards
  - Vehicle manufacturers must provide dismantling info for each new vehicle on the market
- Special mean values and prohibitions
  - Heavy metals - lead, cadmium, hexavalent chrome and mercury
- Design for recycling & recovery (conception phase)

Vehicles made (not designed) before 1 Jan 1990, recycling target of 75% (including max 5% to energy recovery)

From Jan 2015:

reuse & <u>recovery</u>: min. 95% wt./vehicle/yr
reuse & <u>recycling</u>: min. 85% wt./vehicle/yr

Directive 2000/53/CE on the End of Life Vehicles

TOMORROW (2015):

Landfill: 5%
Re-use, materials recycling: 85%
Energy recovery: 10%

- Improve circular design of vehicles to facilitate removal of materials, parts and components for reuse and recycling
- **Ensure that at least 25% of plastic used to build a vehicle comes from recycling (of which 25% from recycled ELVs)**
- **“25 + 25”**

How much will these rules cost?

Less than 70€ per vehicle

Big issue for composite material systems (e.g. epoxy resin)

End-of-life vehicles Regulation - European Commission (europa.eu)

Aerospace end of life

- No equivalent EU legislation

In storage	2017-2030
• over 2’000 aircraft in storage world-wide • number of military aircraft in storage is considerably greater	• 6’000 to 8’000 commercial airlines reach end of life, 3kT CF/year • over next 20 years, ~5’000 commercial airliners are expected to be withdrawn or retired from service • ~250/year

1 (ox.ac.uk)

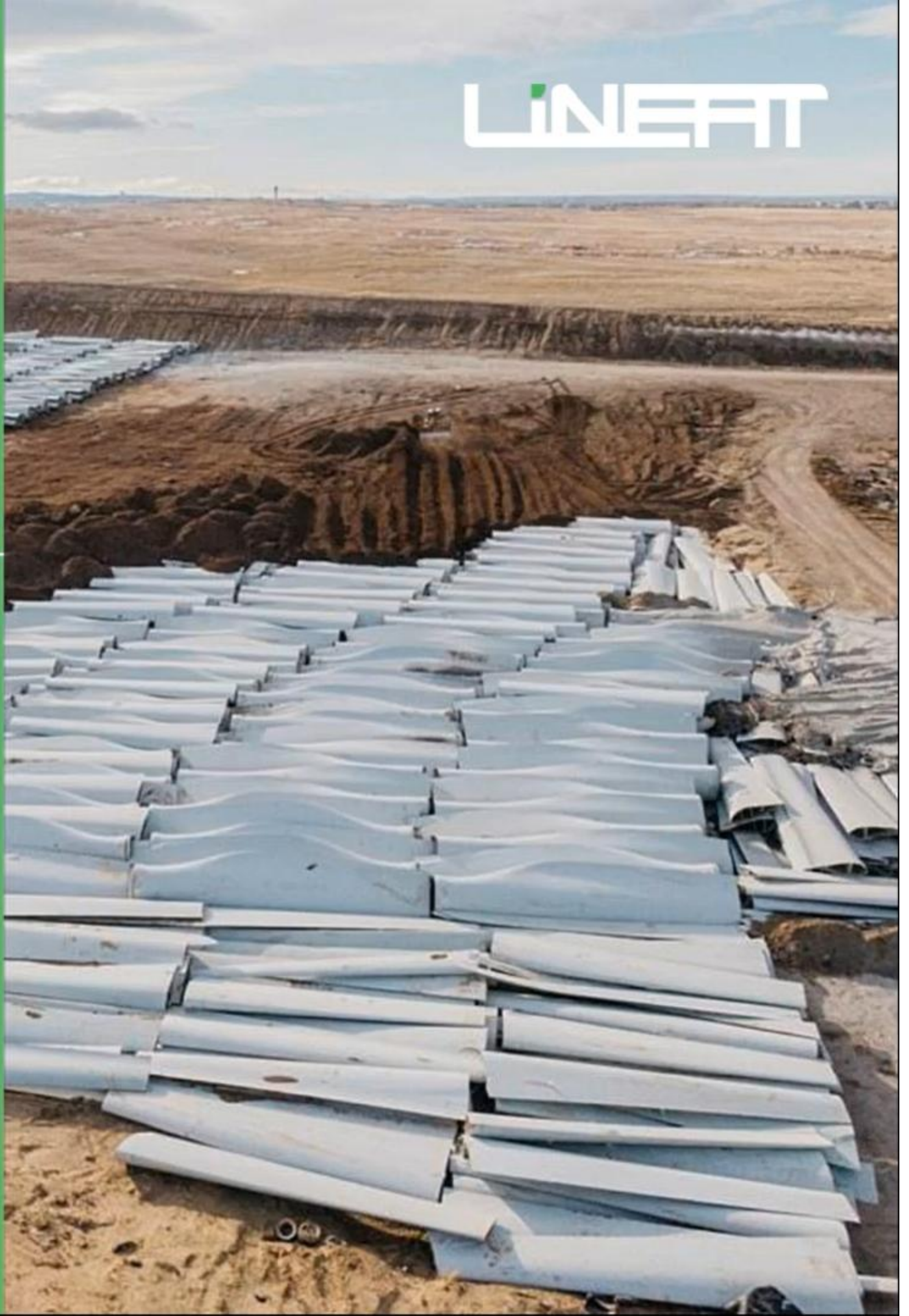
End-of-life aircraft recycling offers high grade materials | Eco-innovation Action Plan (europa.eu)

LIFE 3.0 - LIFE Project Public Page (europa.eu)

THE WORLD'S MOST ADVANCED MATERIAL HAS A DIRTY WASTE PROBLEM

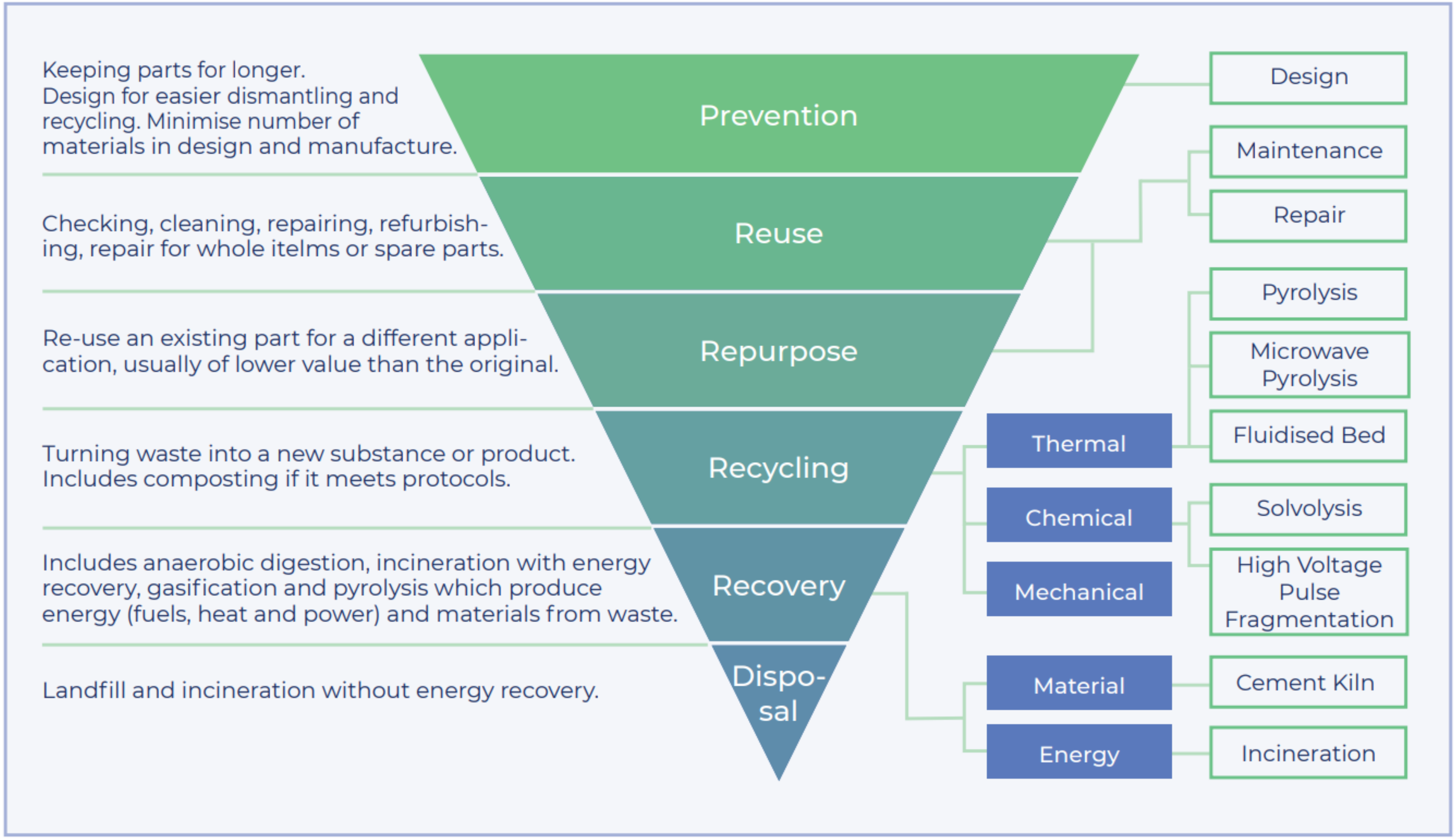
- Over **90%** of carbon fibre ends up in landfill
- **20x** more CO_2 emitted than steel
- Expected **500,000,000 kg** of carbon fibre waste by 2035

15.5 Mt CO_2e



Composite waste management hierarchy

- Desirability is highest at the top of the diagram and decreases going down
  - Preventing a composite part from reaching its EoL is more desirable than recycling it
    - Recycling more desirable than disposing of it



For each step, the processes identified as being most promising and desirable are given.

[Suschem_composites_circularity_pages.pdf (baxcompany.com)]

What is in our waste and EL streams?

Thermosets

Cured / B-staged

- Polyester
- Polyurethane
- Vinyl-esters
- Epoxy
- Phenolic
- Cyanate Ester
- Bismaleimide
- Benzoxazine
- Phthalonitriles
- …

Thermoplastics

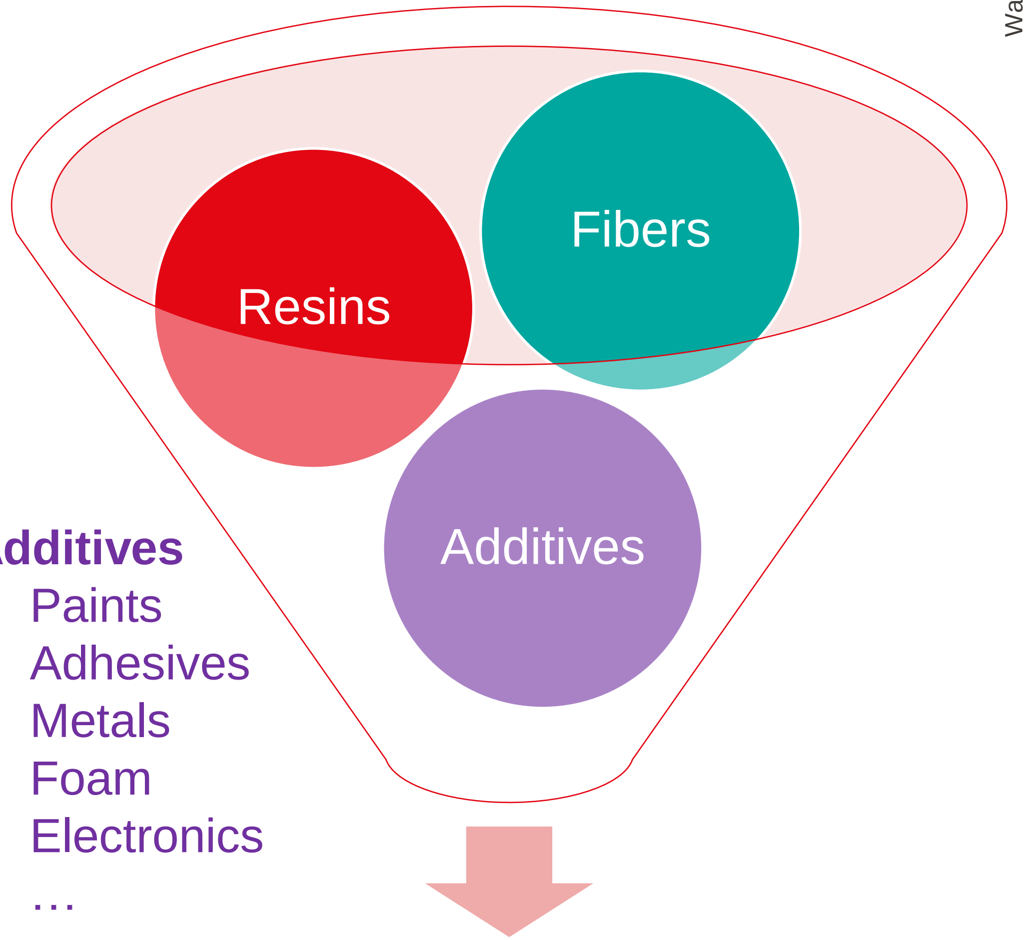
- PE
- PP
- PET
- PA
- PPA
- PC
- PEI
- PPS
- PEAK
- PEEK
- …

Fibers

- Different lengths
- Different architectures
- Dry / impregnated
- Glass
  - S-Glass
  - E-Glass
  - C-Glass
  - XM-Glass
- CF PAN
  - Many different grades
- CF Pitch
- Kevlar (different grades)

Additives

- Paints
- Adhesives
- Metals
- Foam
- Electronics
- …

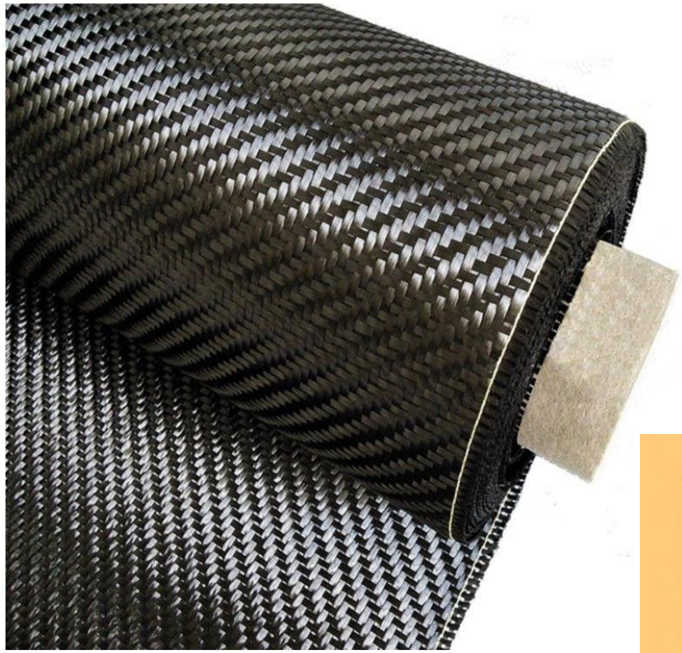


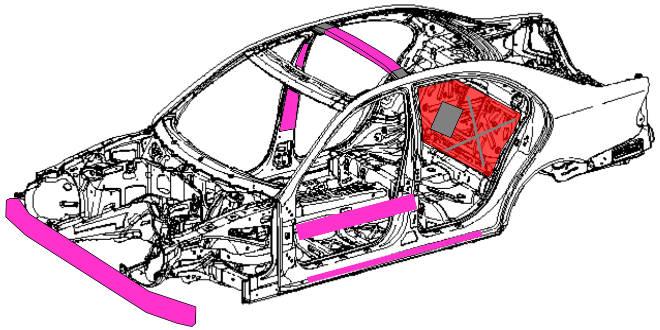
Our waste flow

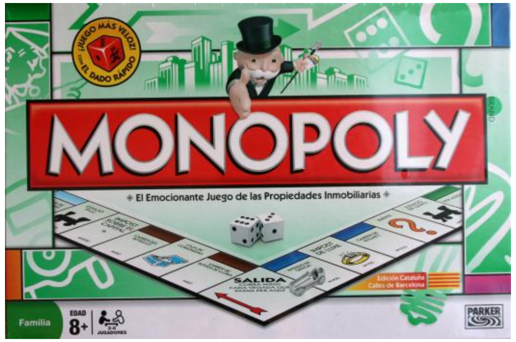
- cm to 10’s meters

Post industrial CF waste = post industrial $ money

Wakeman







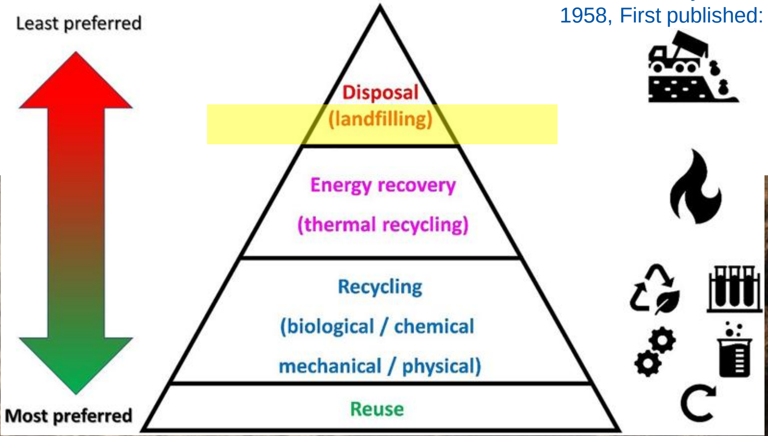
Composites waste management?

Issues with landfill

Journal of Polymer Science, Volume: 61, Issue: 17, Pages: 1937-1958, First published: 19 May 2023, DOI: (10.1002/pol.20230154)

Wyoming's Wind Turbine Graveyard
CASPER, WYOMING, USA - January 9, 2020: Pieces of wind turbine blades are buried in the Casper Regional Landfill in Casper, Wyoming. Around 8,000 wind turbine blades will need to be removed and disposed of a year in the United States alone. Because of the conditions they are built to withstand, the blades cannot be easily recycled. This leads many of them to be buried in landfills like Casper's, where 870 blades stacked into holes 30 feet deep. (Photo by Benjamin Rasmussen/Getty Images)

[Pieces of wind turbine blades are buried in the Casper Regional... News Photo - Getty Images]



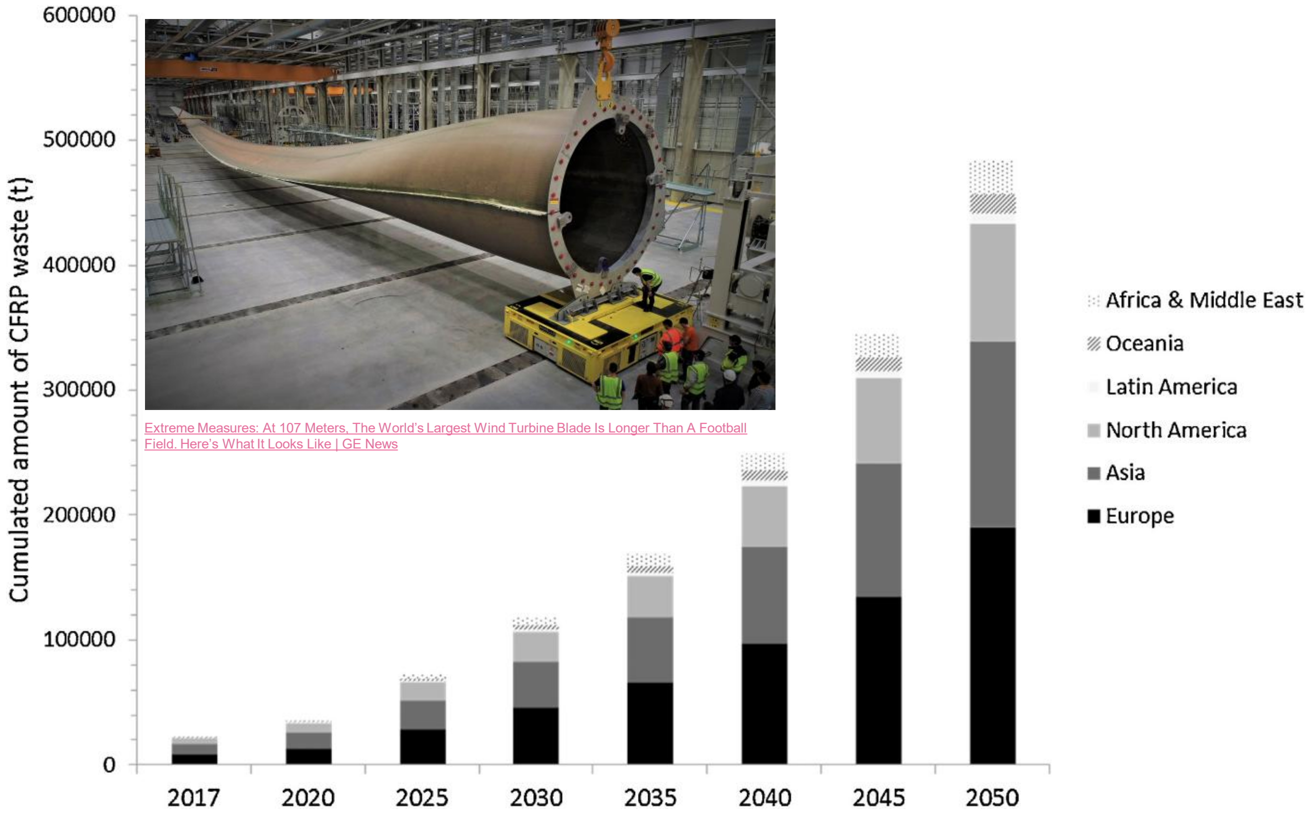
[Bisphenol-A-Pollution-Wind-Turbines.pdf (wind-watch.org)]
[Abandoned Wells Can Provide Use for Pulverized Wind-Turbine Blades | Journal of Petroleum Technology | OnePetro]
[ACP_MicroplasticsFactSheet_March-2023.pdf (cleanpower.org)]

Start by not throwing it

Anticipating in-use stocks of carbon fibre reinforced polymers and related waste generated by the wind power sector until 2050

Anaële Lefeuvre[a,*], Sébastien Garnier[a], Leslie Jacquemin[a], Baptiste Pillain[a], Guido Sonnemann[b,c]

600kT

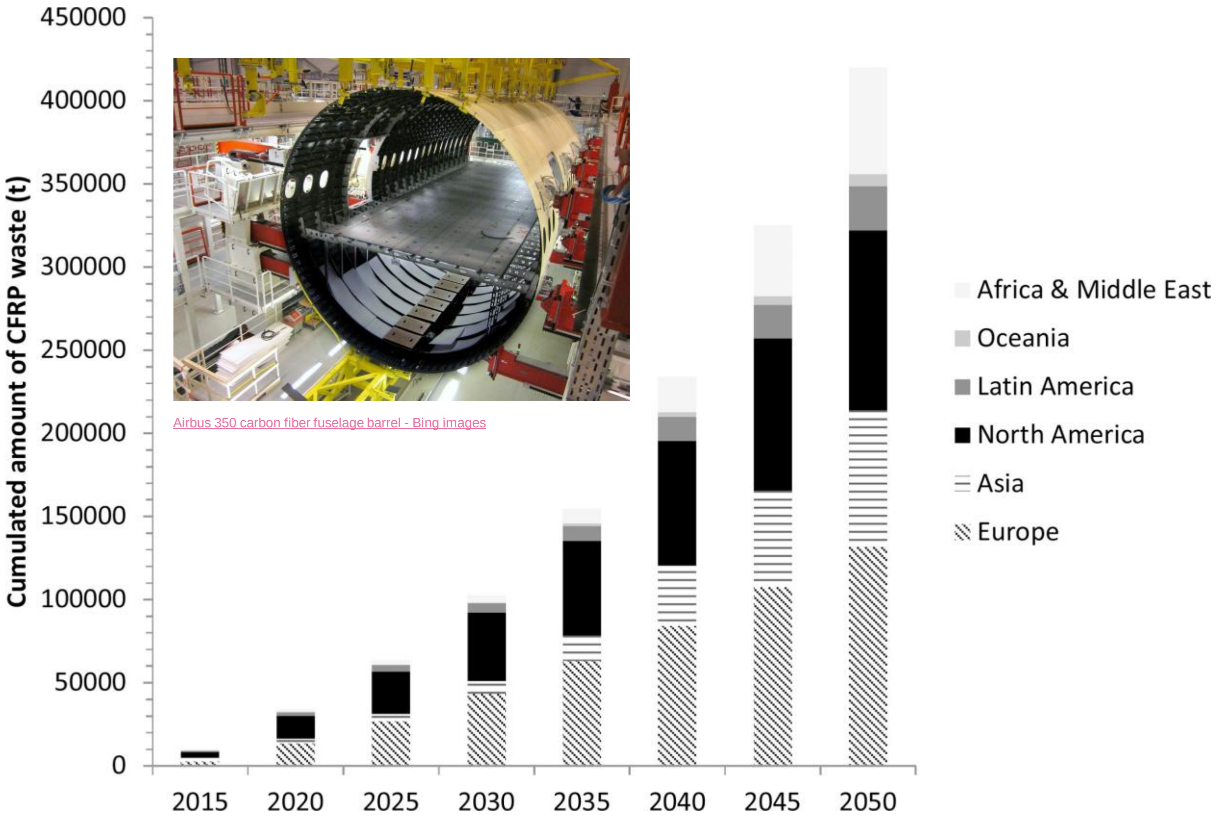


[Extreme Measures: At 107 Meters, The World's Largest Wind Turbine Blade Is Longer Than A Football Field. Here's What It Looks Like | GE News]

Anticipating in-use stocks of carbon fiber reinforced polymers and related waste flows generated by the commercial aeronautical sector until 2050

Anaële Lefeuvre[a], Sébastien Garnier[a], Leslie Jacquemin[a], Baptiste Pillain[b,c], Guido Sonnemann[b,c,*]

450kT



[Airbus 350 carbon fiber fuselage barrel - Bing images]

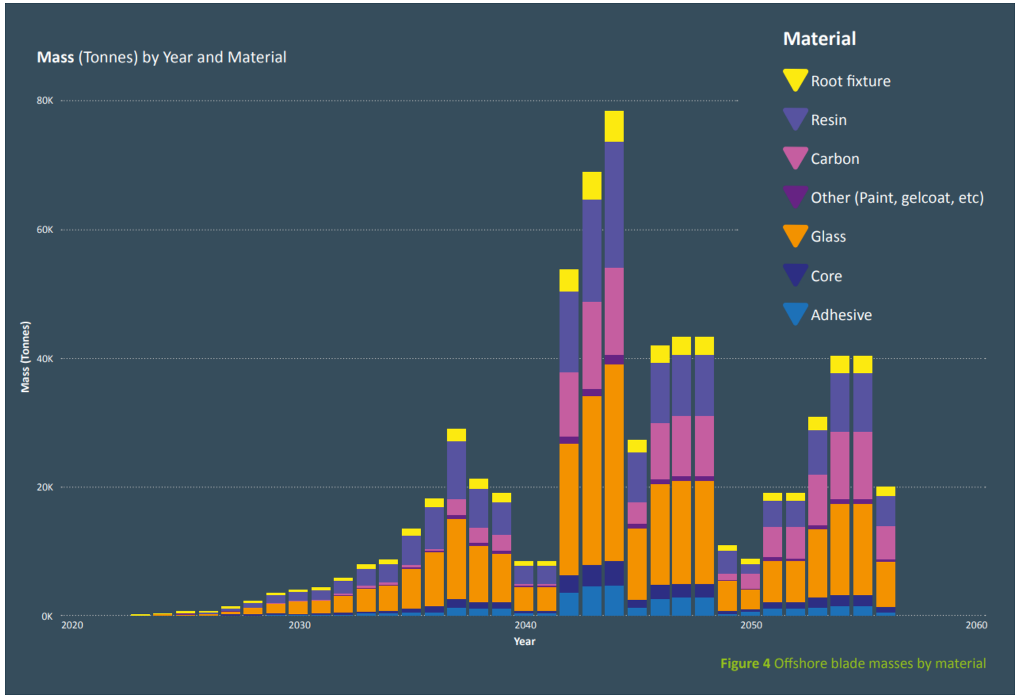
[Anticipating in-use stocks of carbon fibre reinforced polymers and related waste generated by the wind power sector until 2050 - ScienceDirect]

[Anticipating in-use stocks of carbon fiber reinforced polymers and related waste flows generated by the commercial aeronautical sector until 2050 - ScienceDirect]

EPFL

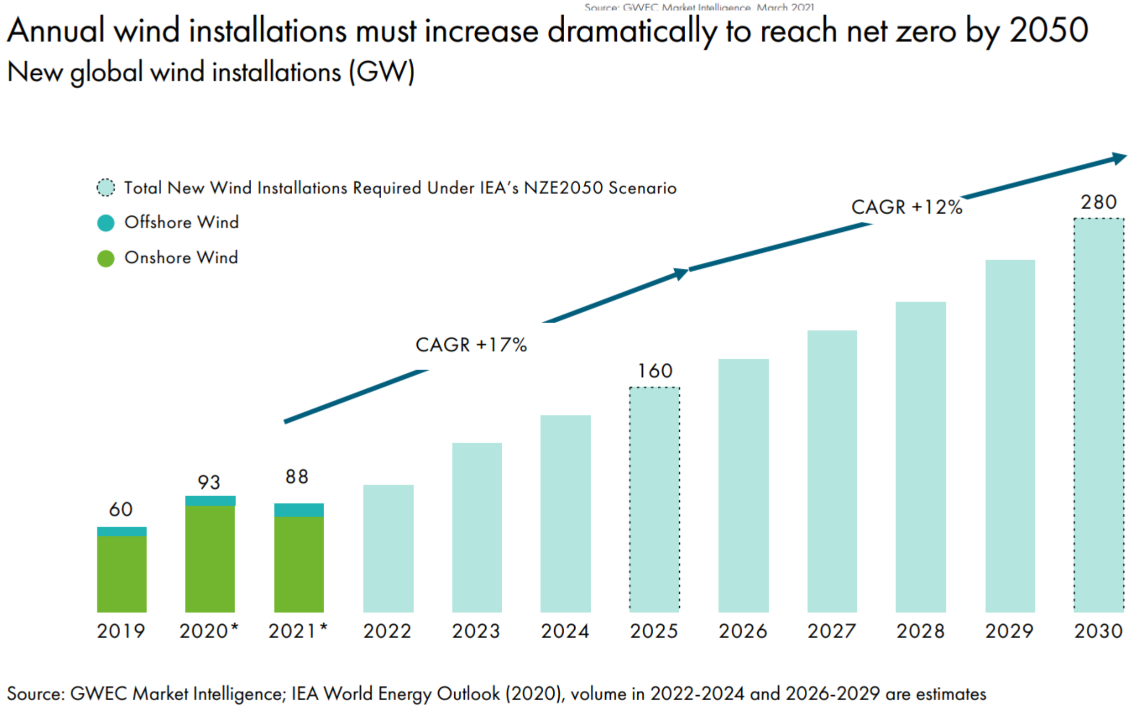
Wind energy needs a circular approach

- by 2050, 43 million tonnes of decommissioned turbine blades will reach their end of life and need to be managed through the global waste stream
- Current practice in US and EU is landfill



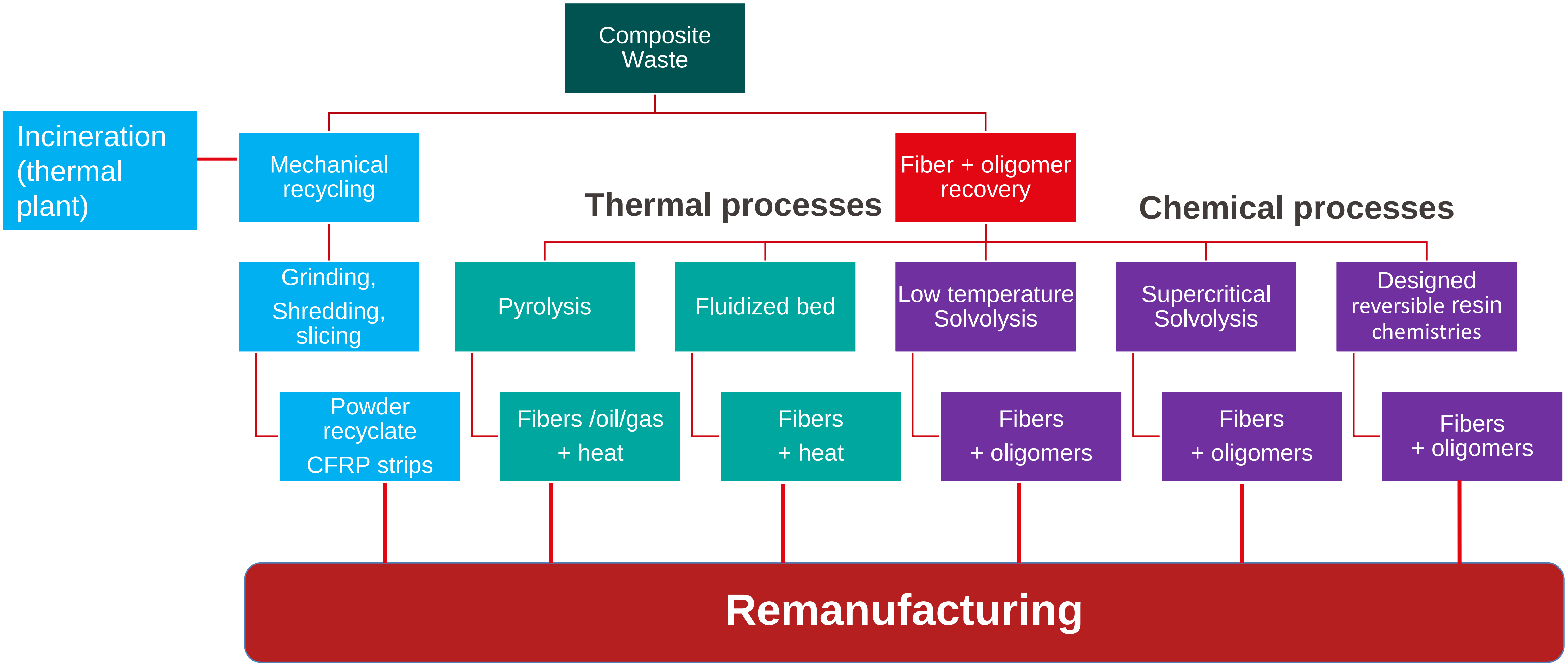
Offshore UK wind waste stream by material to 2055

[suswind-annual-review-2022-final.pdf (nccuk.com)]



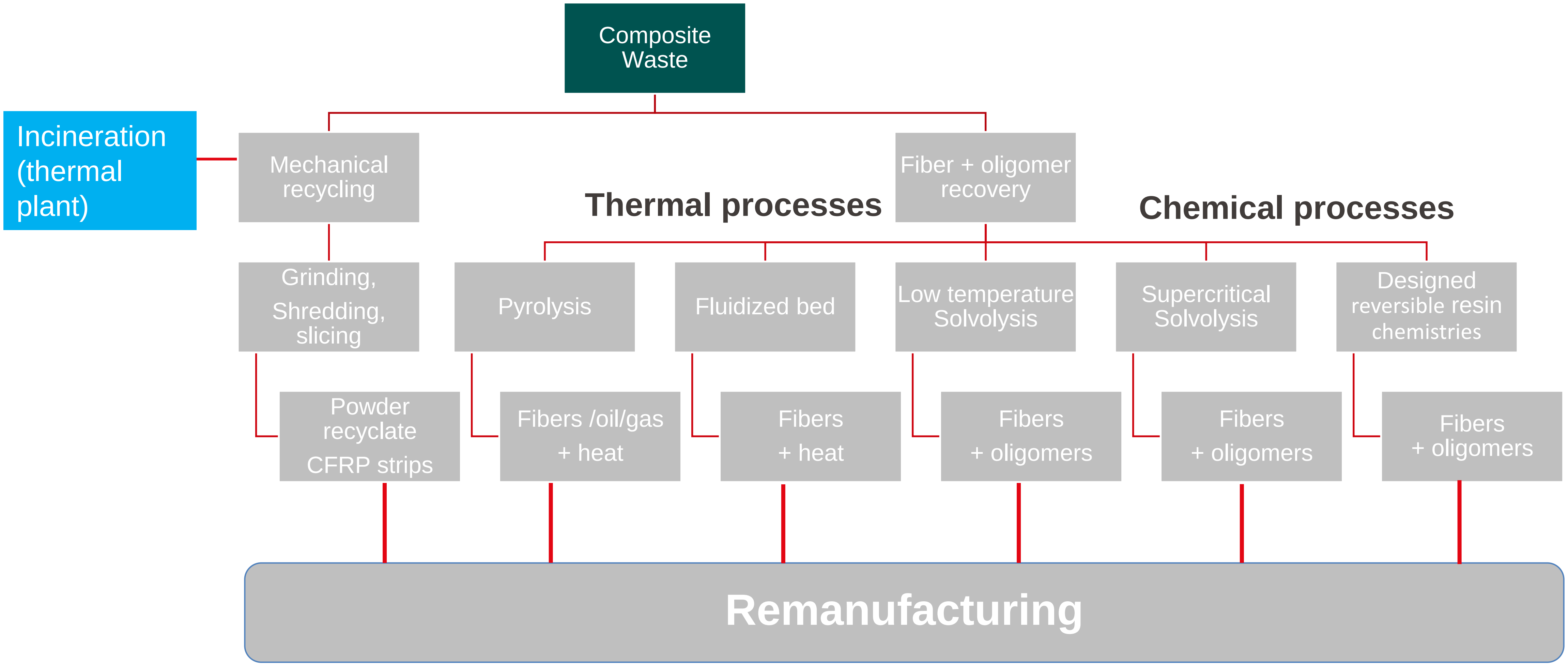
[Energies | Free Full-Text | How to Repair the Next Generation of Wind Turbine Blades (mdpi.com)]

Recycling Routes



Adapted from Pickering, Composites Part A (2006)

Recycling Routes



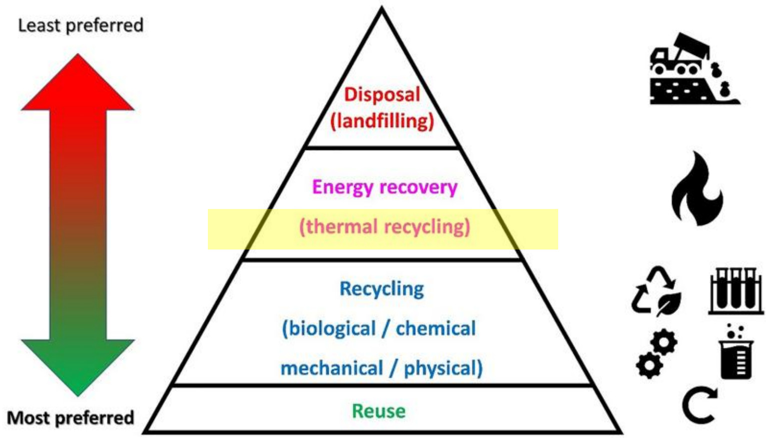
Adapted from Pickering, Composites Part A (2006)

How the EU sees composites recycling

EuPC EUROPEAN PLASTICS CONVERTERS

EuCIA

European Composite Recycling Services Company



Glass fibre reinforced thermosets: recyclable and compliant with the EU legislation

June 2011

The European Plastics Converters (EuPC), the European Composites Industry Association (EuCIA) and the European Composite Recycling Service Company (ECRC) welcome the End-of-Life of Vehicles Directive (2000/53/EC) and the Waste Framework Directive (2008/98/EC): **glass fibre reinforced thermosets are both material and energy recyclable through the cement kiln route and compliant with the EU legislation.**

[EuCIA-position-paper-52816.pdf (csmres.co.uk)]

GF composites: Thermal plant feedstock

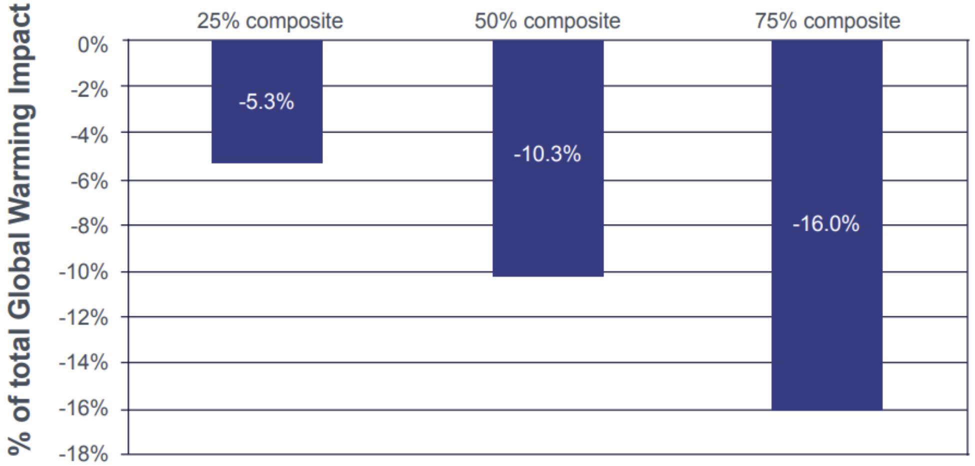
- Co-processing of End of Life/ composites used in cement manufacturing
  - highly effective source of energy
  - calorific values of~30,000 kJ/kg
- Glass fiber in oxide form, replacing hydrated or carbonated minerals in Portland cement
- Replacement of fossil fuels by non-recyclable & biomass waste, and use of alternative raw materials
  - Gives 15% CO_2 emissions reduction in the cement industry by 2050
- Co-processing plants (such as cement or lime kilns, steel plants, etc.) that produce material products, waste used as a fuel and/or raw material that otherwise would be subject to disposal.


Fig.1 : Unloading of windmill blades at processing site
Courtesy: Zajons, Melbeck


Fig.2 : Part storage prior to mechanical treatment
Courtesy: Zajons, Melbeck

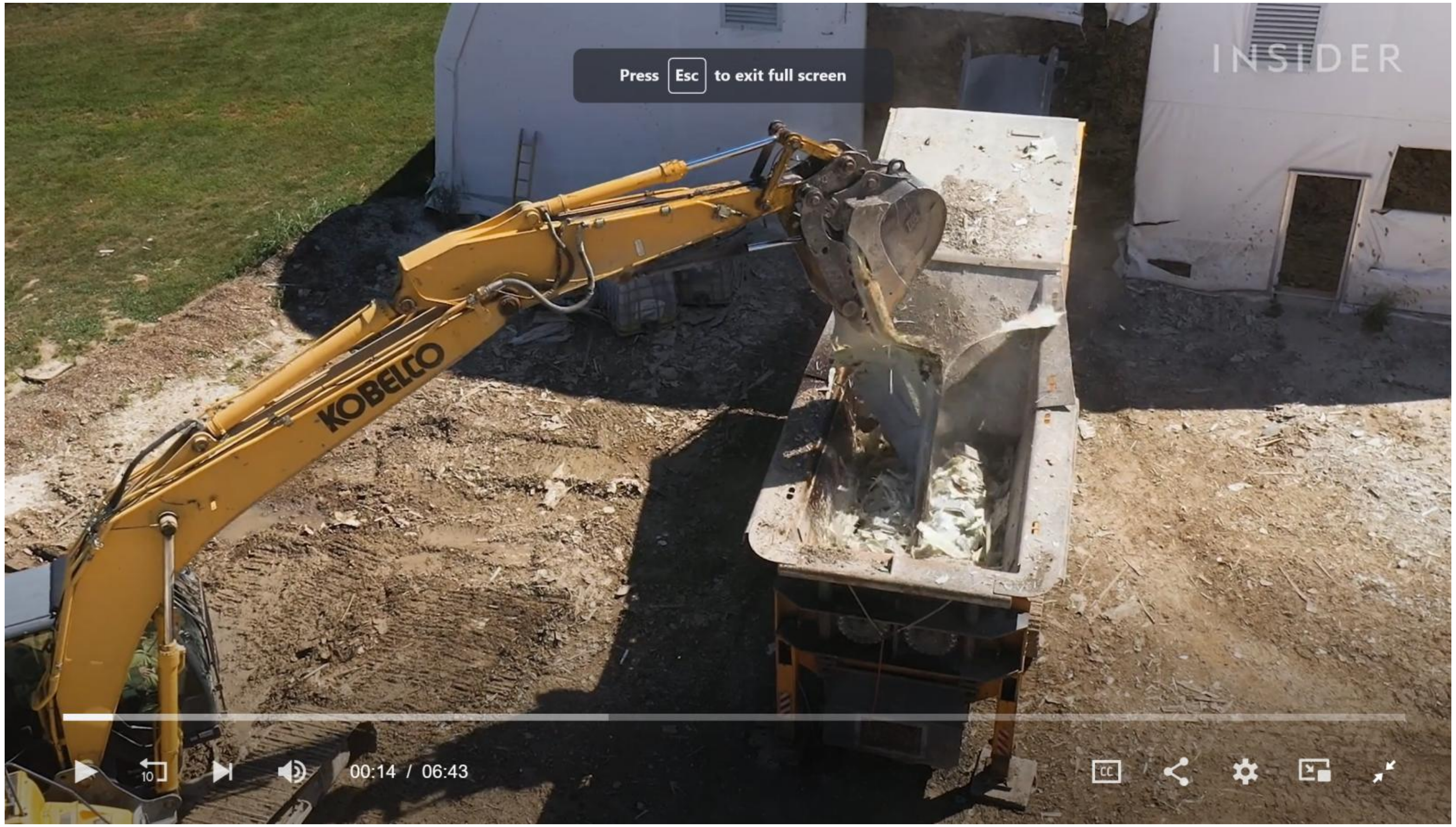

Fig.3 : Mobile sawing equipment to reduce transport costs
Courtesy: Zajons, Melbeck


Fig.4 : Typical composite regrind, made from ground windmill blade
Courtesy: Zajons, Melbeck

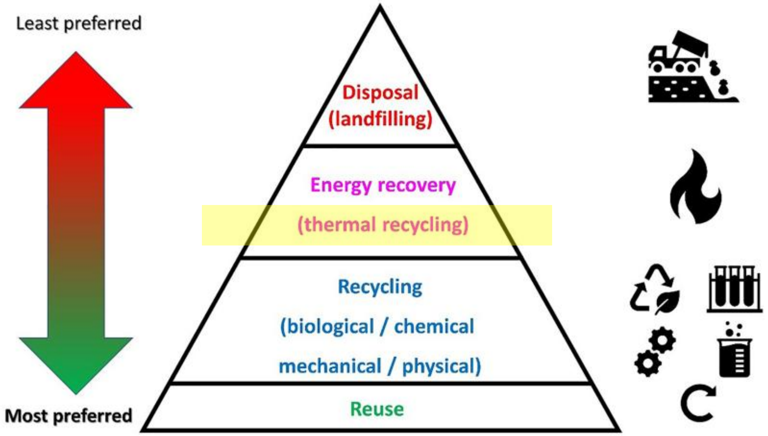


20130207_eucia_brochure_recycling.pdf

Shredding blades for cement feedstock



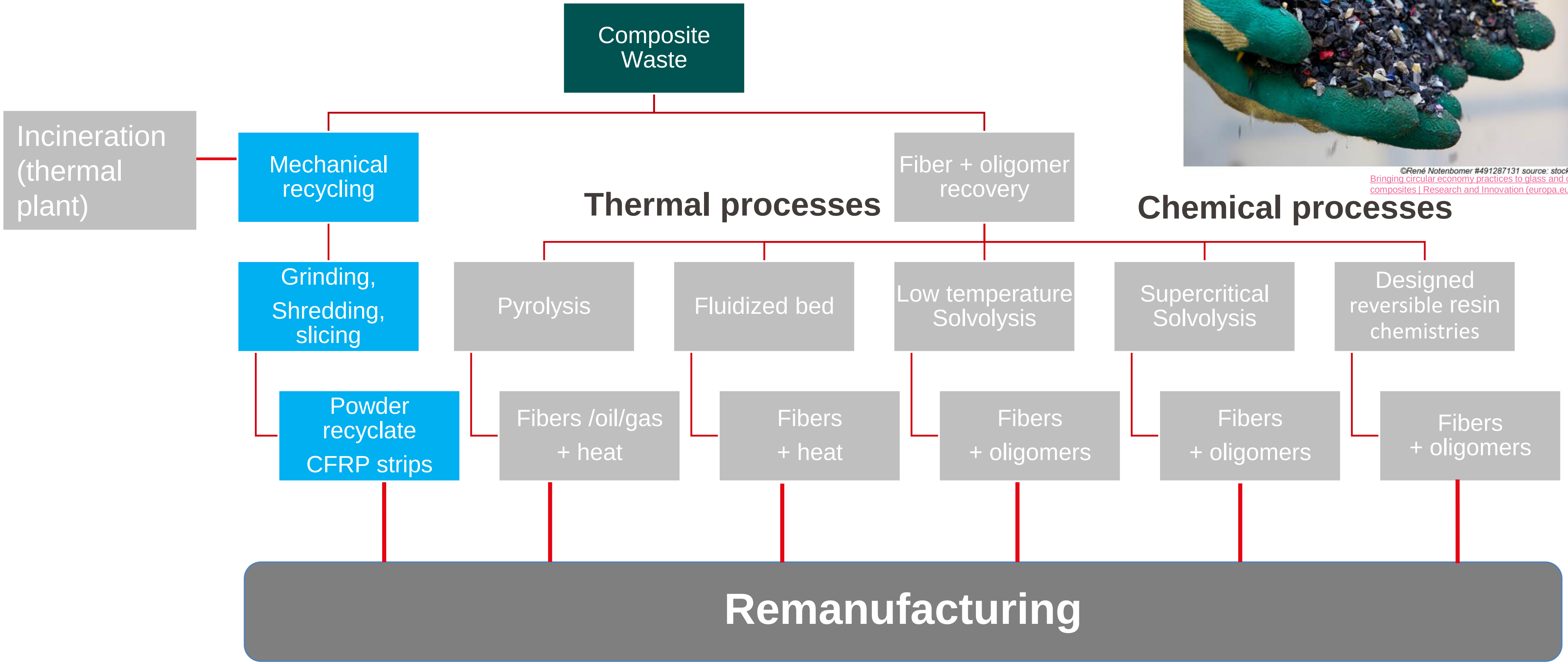
How One Company Keeps Wind Turbine Blades to Keep Out of Landfills (businessinsider.com)



Journal of Polymer Science, Volume: 61, Issue: 17, Pages: 1937-1958, First published: 19 May 2023, DOI: (10.1002/pol.20230154)

Recycling Routes

©René Notenbomer #491287131 source: stock.adobe.com 2023
Bringing circular economy practices to glass and carbon fibre composites | Research and Innovation (europa.eu)

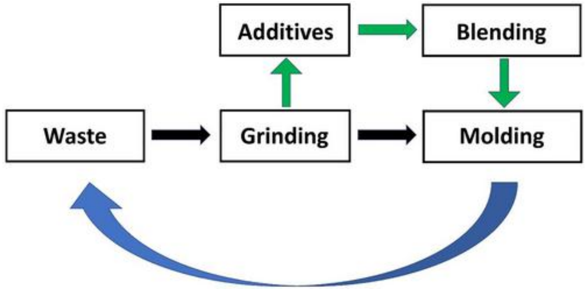


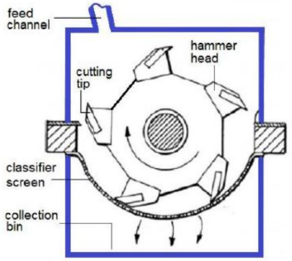
Adapted from Pickering, Composites Part A (2006)

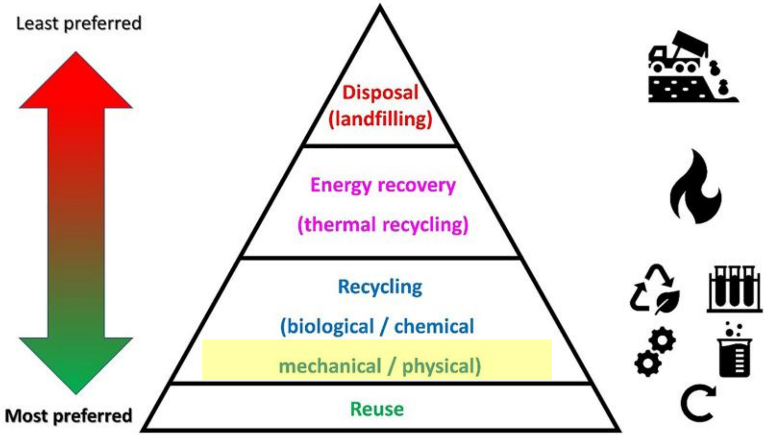
Mechanical recycling (thermoplastic composites)

Wakeman

- Post industrial waste
- Shredded
- Used for injection molding
- Higher fiber lengths vs. short glass injection







Composites Part A 139 (2020) 106110

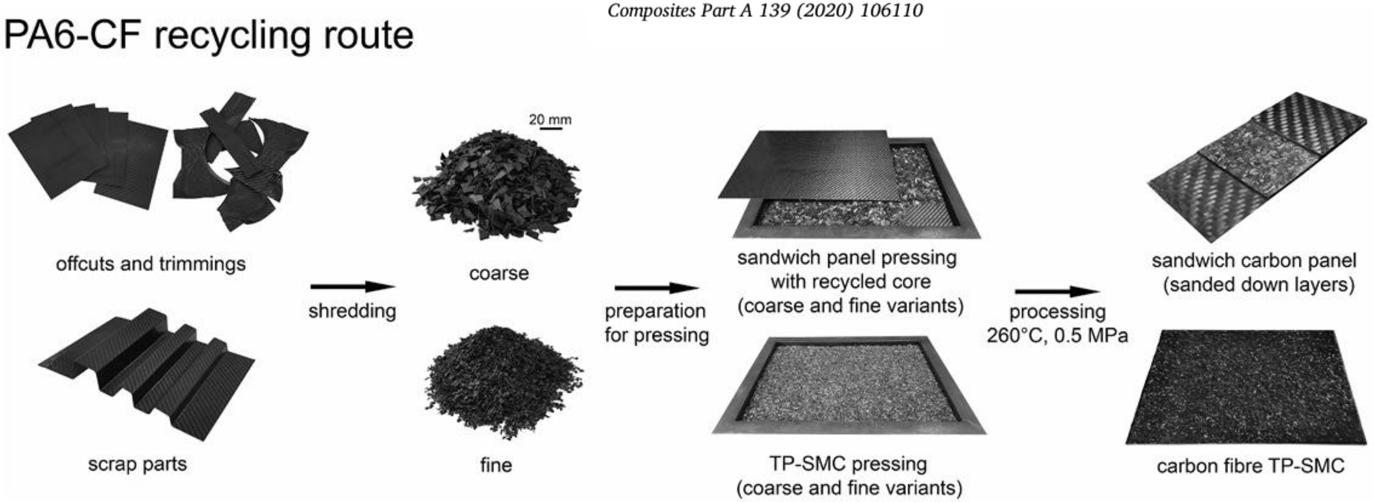
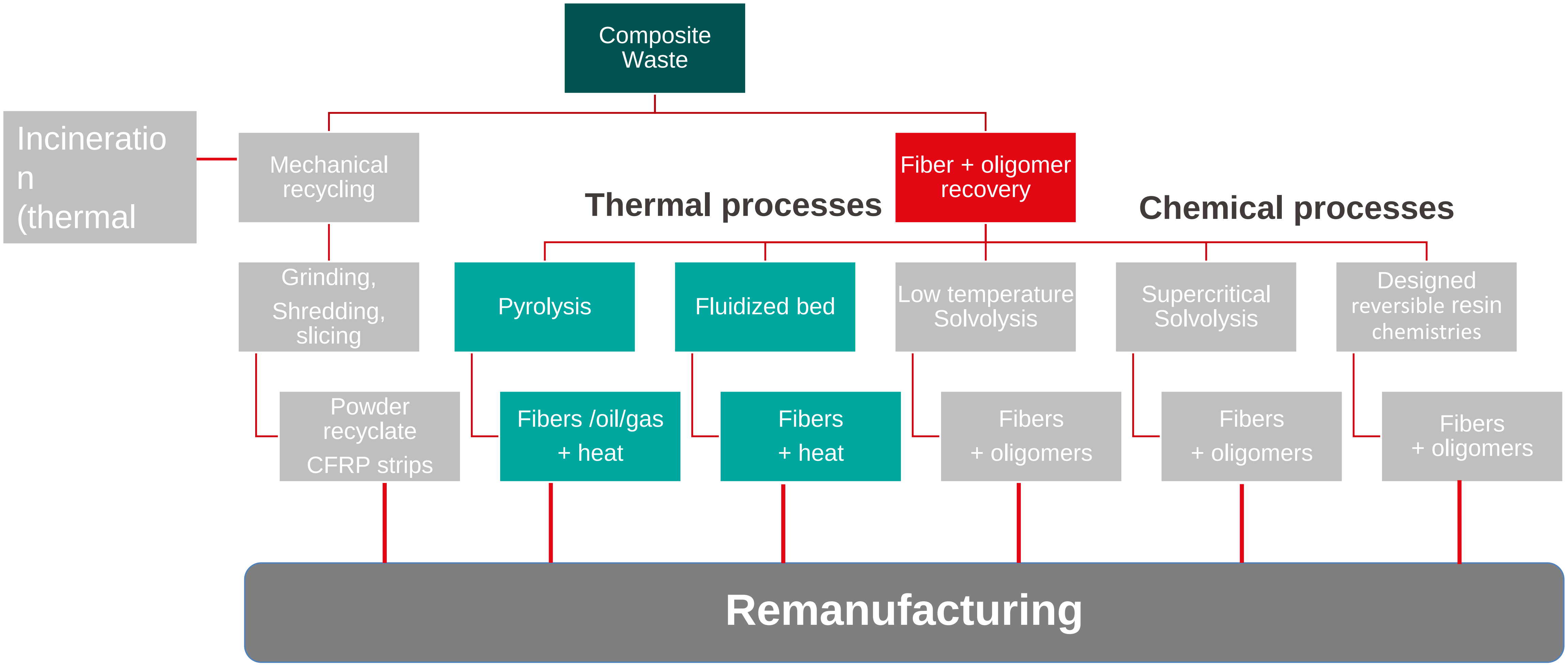


Fig. 1. Recycling routes for PP-GF and PA6-CF based TPCLs.

Journal of Polymer Science, Volume: 61, Issue: 17, Pages: 1937-1958, First published: 19 May 2023, DOI: (10.1002/pol.20230154)

In-house recycling of carbon- and glass fibre-reinforced thermoplastic composite laminate waste into high-performance sheet materials - ScienceDirect

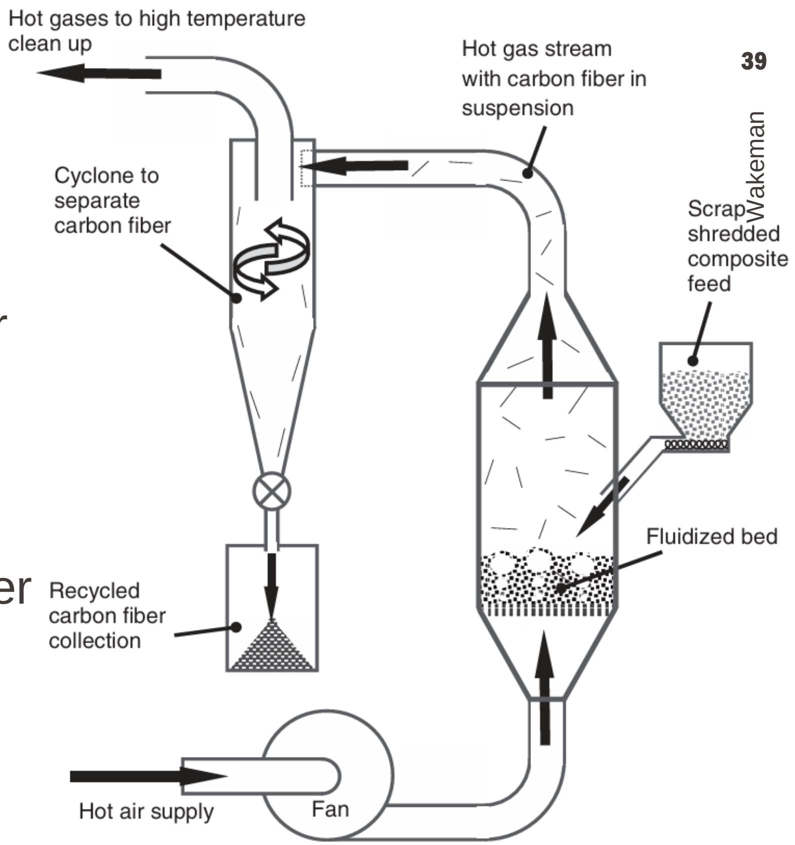
Recycling Routes



Adapted from Pickering, Composites Part A (2006)

Fluidized bed recovery

- Waste composite material shredded 25mm in size
- 450◦C glass / polyester; 550◦C CFRP
- Fibers separate from one another, carried out of fluidized bed in the hot air stream
- Cyclone used to separate fibers from the gases which then pass to a high temperature combustion chamber for full oxidation.
- Energy recovery from high temperature gases leaving combustion chamber
- Tolerant of contaminated and mixed materials, suitable for end-of-life components



CF strength retention 46-62%, modulus not affected

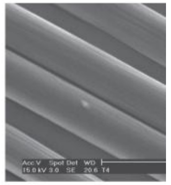
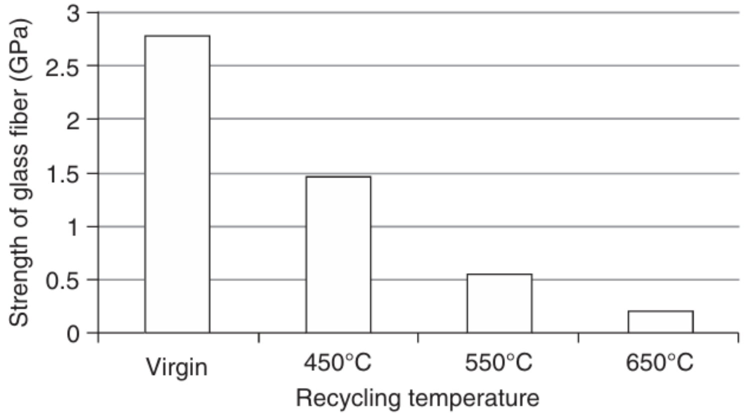


Figure 4. Recycled carbon fiber in a fluffy form.



Boeing funds carbon fibre recycling research – Research, The University of Nottingh

PhD thesis_Fanran Meng_4201331_after correction_final.pdf (nottingham.ac.uk)

Recycling Thermoset Composite Materials - Pickering - Major Reference Works - Wiley Online Library

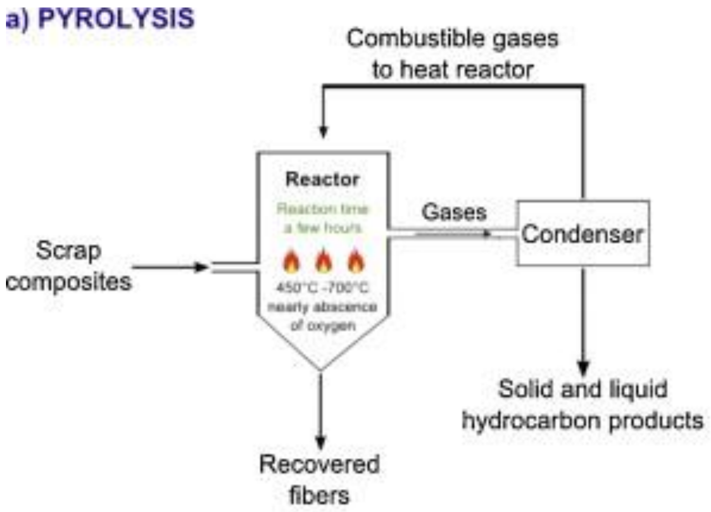
Pyrolysis

- Thermal degradation of resin / polymeric additives
  (absence of oxygen / nitrogen atmosphere)
- 400-500ºC
- Calcification / oxidation step in air to remove char on fiber surface
- 1 or 2 step process
- Fibers recovered
- Gas / oil / char

[Recycling of carbon fibre-reinforced plastics (youtube.com)](#)

1:45s – 6:11

Wakeman



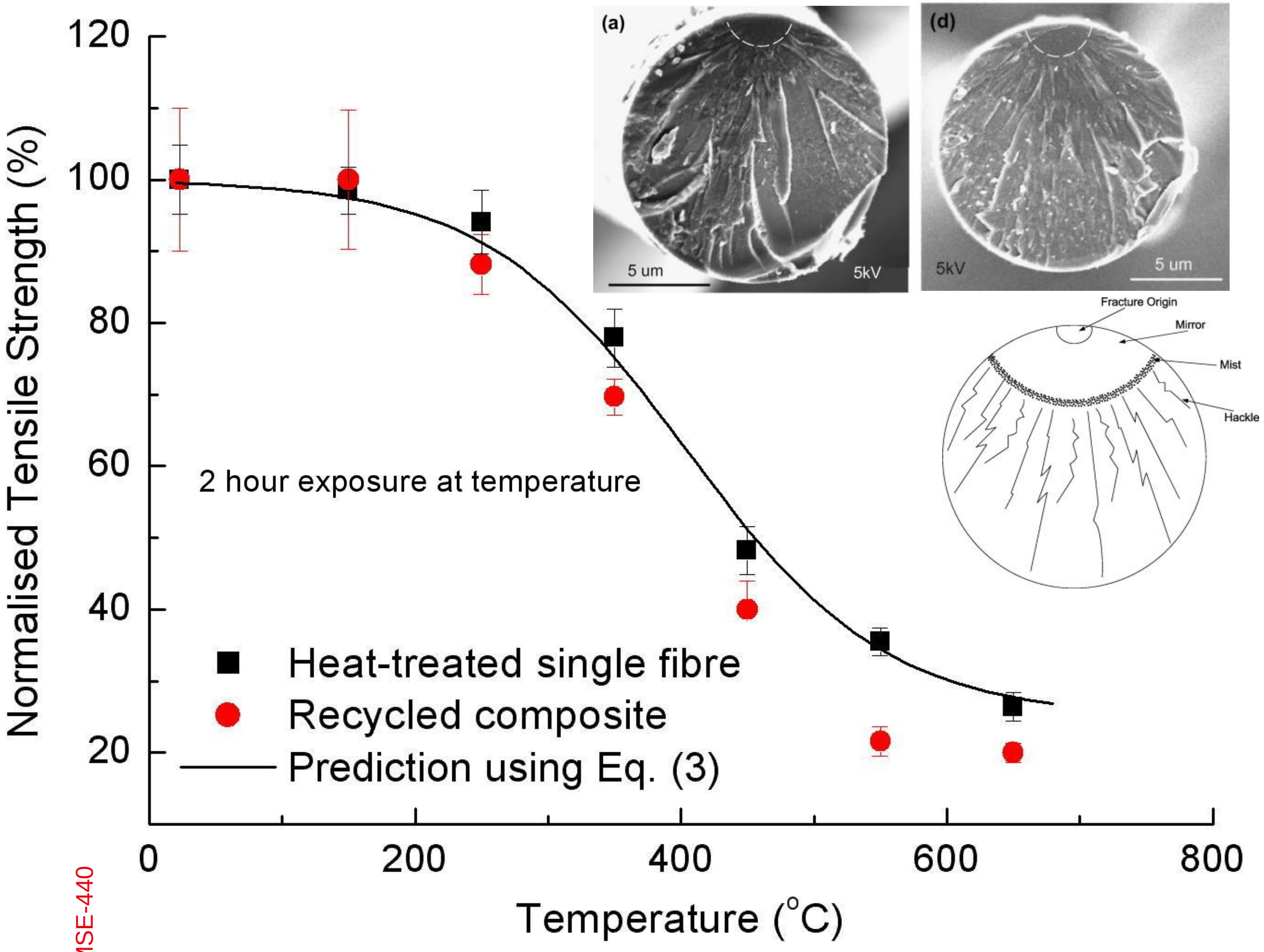
500ºC in Nitrogen

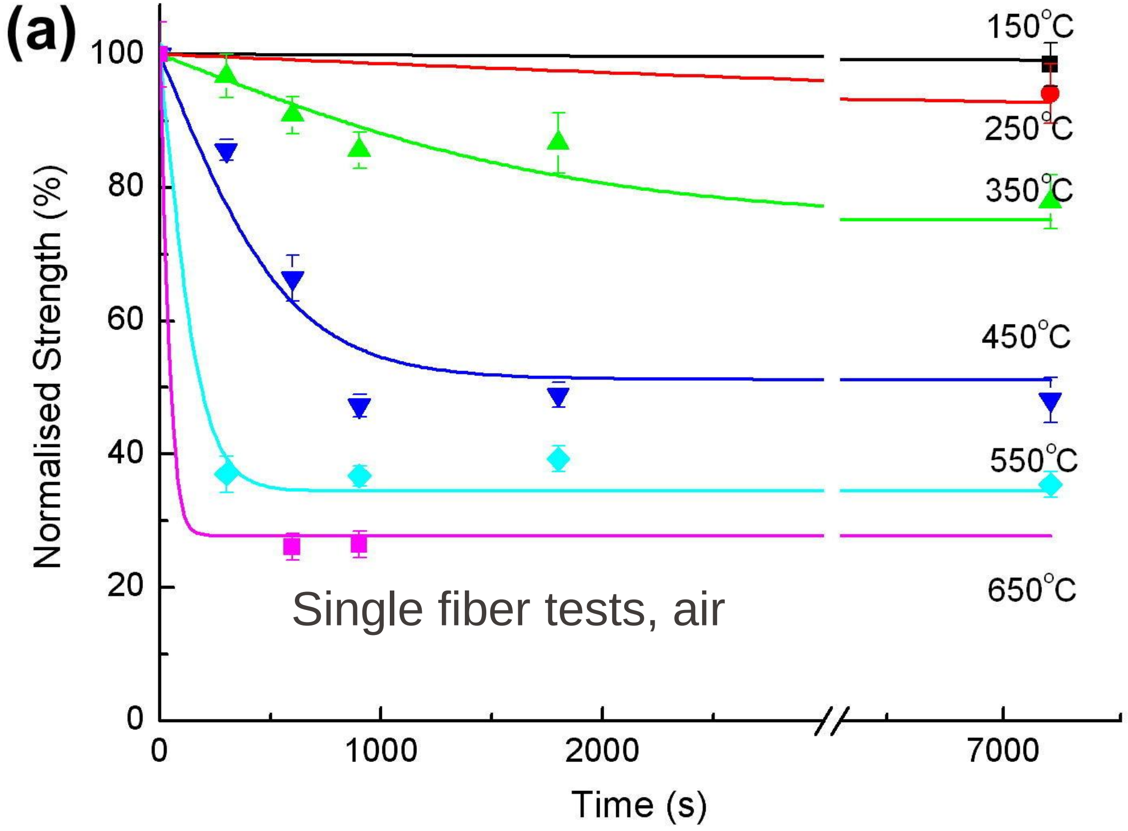
MITSUBISHI CHEMICAL ADVANCED MATERIALS | carboNXT

[100% CARBON FIBERS (carbonxt.de)](#)

Effect of pyrolysis on glass fiber properties

- Modulus not affected
- Strength loss after heating due to surface defects, annealing/stress relaxation, chemical diffusion





Mechanical properties of thermally-treated and recycled glass fibres - ScienceDirect

EPFL

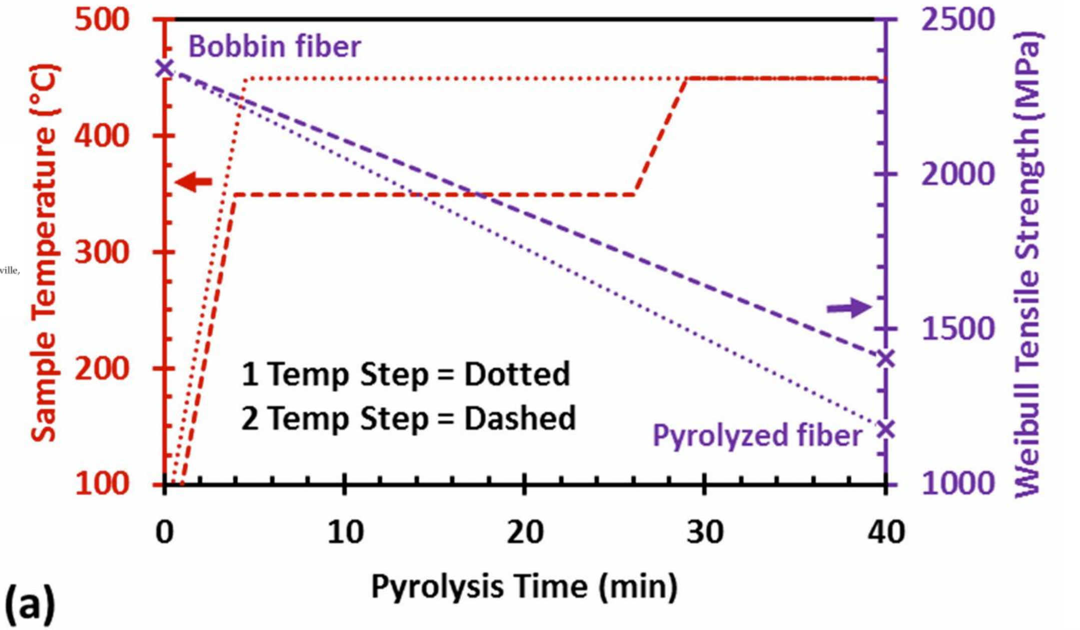
Two step pyrolysis

- Two step
  - 350ºC for 22minutes
  - 450ºC for 11 minutes
- Glass fiber structural changes

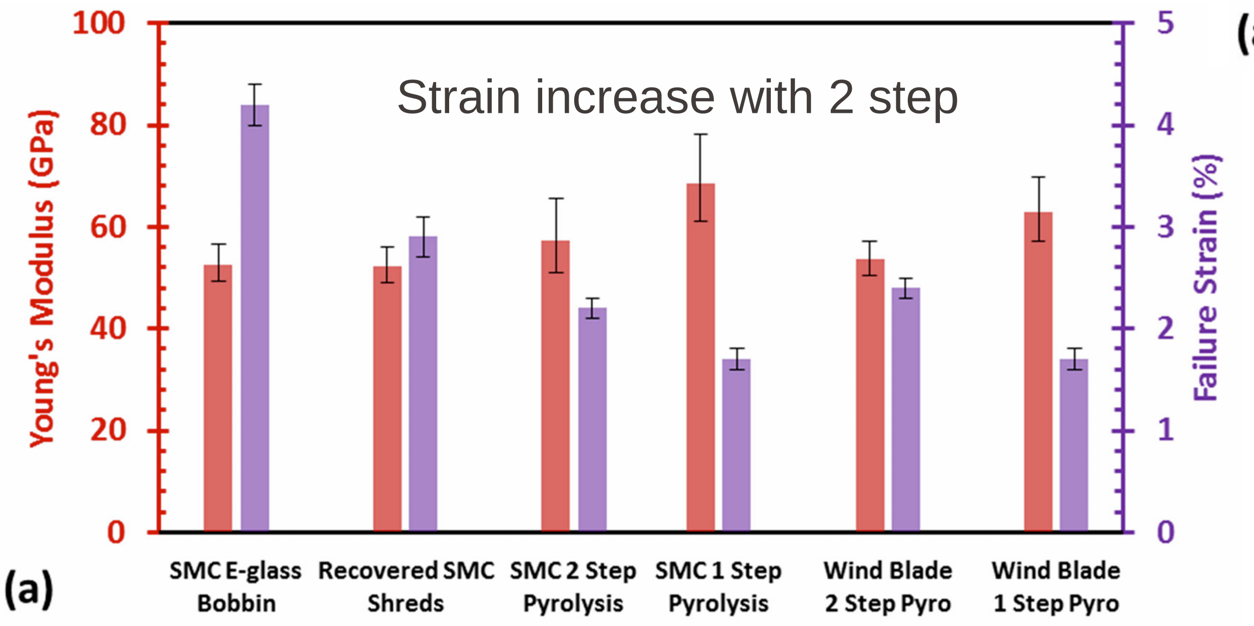
Recycling of Commercial E-glass Reinforced Thermoset Composites via Two Temperature Step Pyrolysis to Improve Recovered Fiber Tensile Strength and Failure Strain

Ryan S. Ginder [1,2,*] and Soydan Ozcan [1,2]

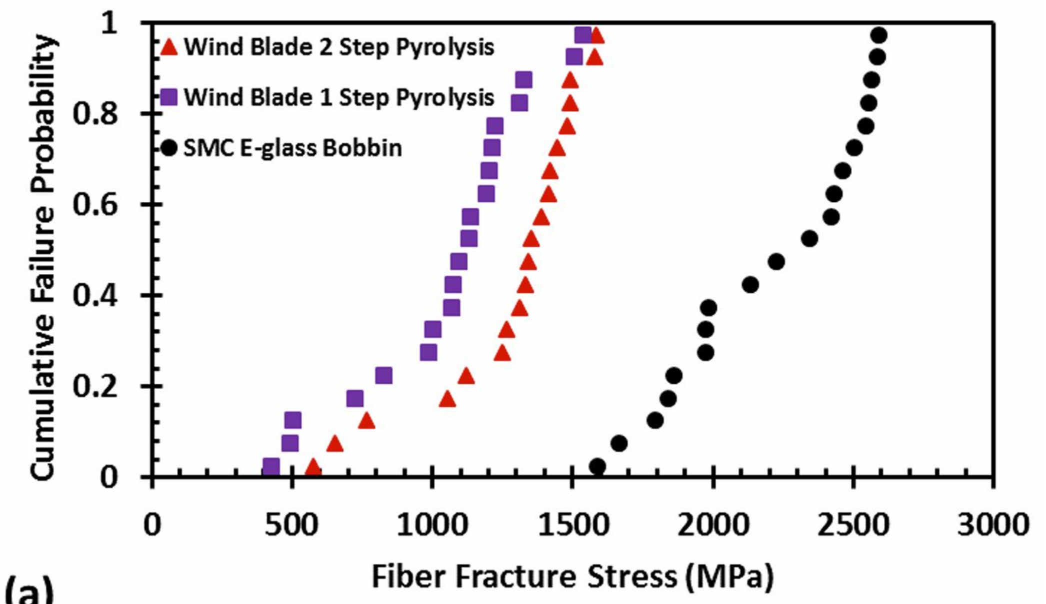
1 Department of Mechanical, Aerospace and Biomedical Engineering, University of Tennessee, Knoxville, TN 37996, USA; ozcans@ornl.gov
2 Chemical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA
* Correspondence: rginder@vols.utk.edu; Tel.: +1-865-574-9040



(a)



(a)

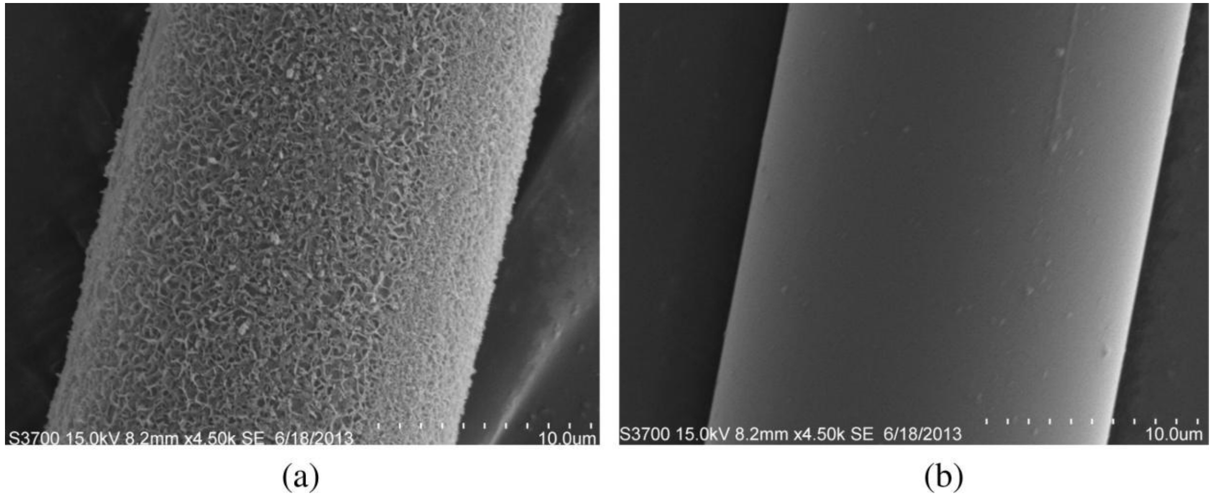


(a)

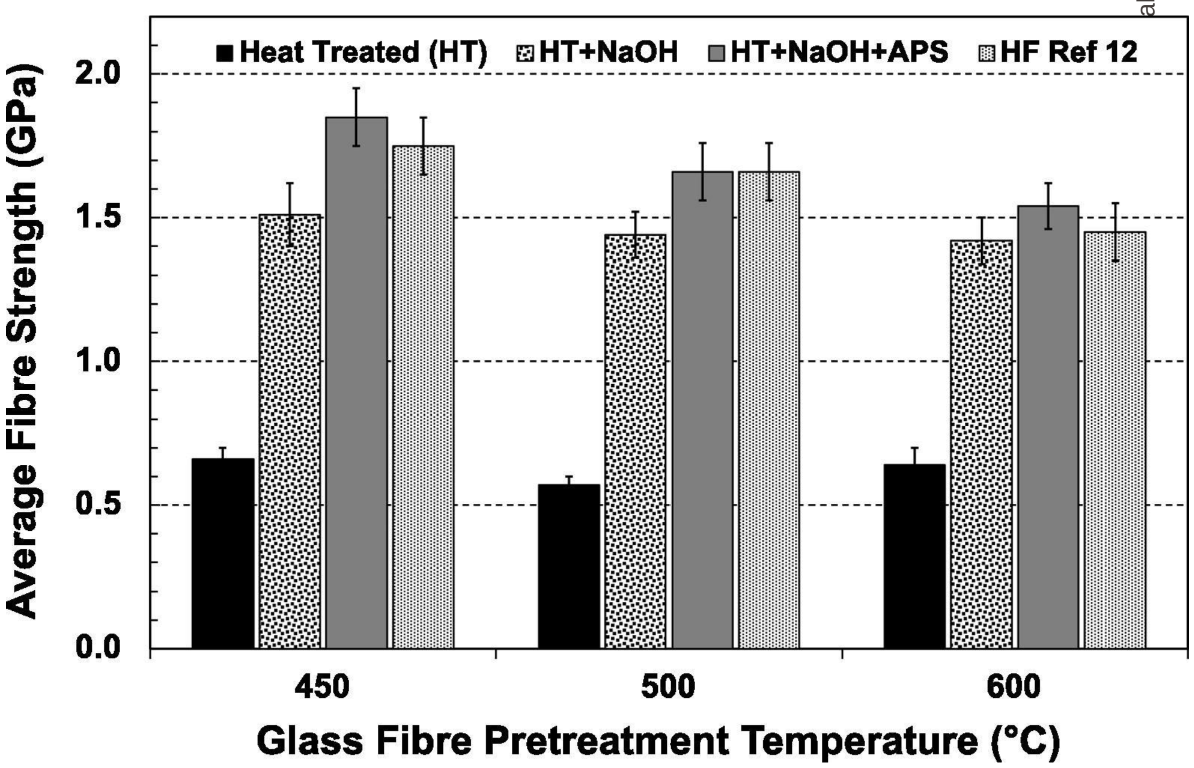
Wakeman

Glass fiber post treatment after pyrolysis

- Immersed in a sodium hydroxide solution (NaOH) for 10 min at 90 °C
- Fibrers drained & rinsed in hydrochloric acid (HCl) to neutralize, followed by silane sizing treatment
- Recover compatibility of fiber to resin and interfacial stress transfer



SEM images of heat conditioned fibre after (a) NaOH treatment, (b) NaOH treatment + HCl rinse + Silane coating.



Influence of heat treatment temperature and ReCoVeR chemical treatments on the average glass fibre strength at 20 mm gauge length.

Regenerating the strength of thermally recycled glass fibres using hot sodium hydroxide - ScienceDirect

Effect of pyrolysis on carbon fiber

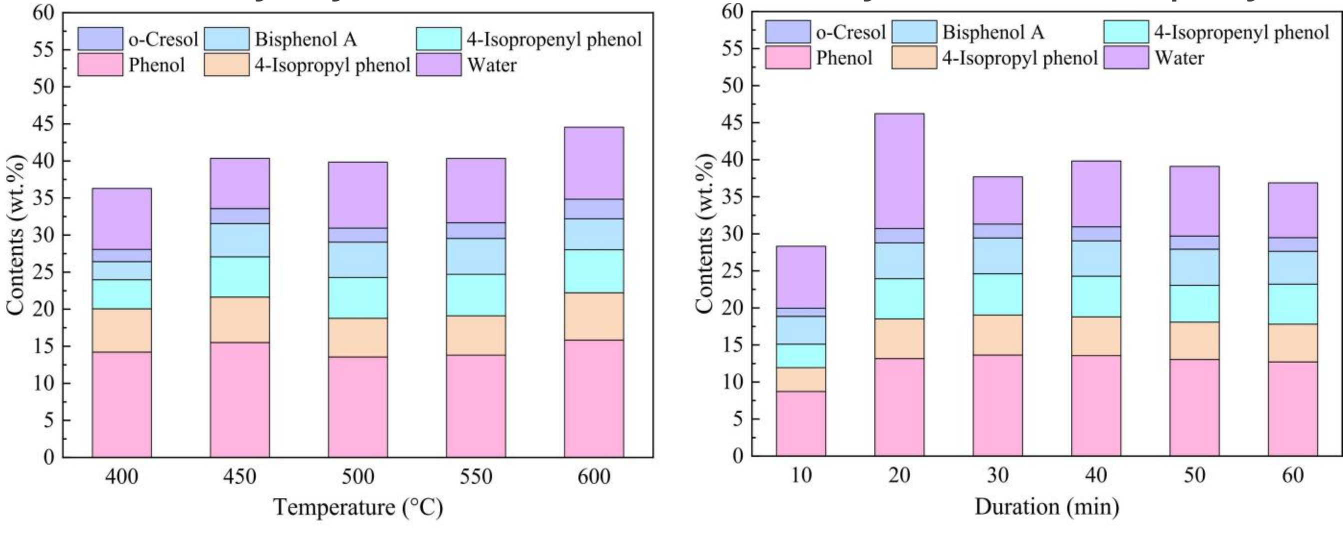
- Modulus generally maintained
- Strength reduction, process and parameter dependent
  - Lower end: SM 80%, IM as much as 55%
  - Upper end: pyrolysis temperature of 500ºC / 20 mins, 94%.
- Surface chemistry similar to virgin CF
- Some post heat in air/oxygen at 500-600ºC to remove pyrolytic char from CF surface
- Microwave heating can reduce avoid char formation on fiber surface or gasification, heated in oxygen to 600ºC

Table S1 The tensile strength retention of carbon fibers recovered by different methods.

Method	Tensile strength retention* (%)	Sources
Mechanical method	50-65	Zhu, et al [1]
Fluidized bed method	80.00	E. Pakdel, et al [2]
Pyrolysis method #1	92.00	O. Zabihi, et al [3]
Pyrolysis method #2	75.86	Lopez, et al [4]
Chemical method #1	93.55	Pei, et al [5]
Chemical method #2	Almost 100	Jiang, et al [6]

* Tensile strength retention was the ratio of tensile strength between recovered and virgin carbon fibers [7].

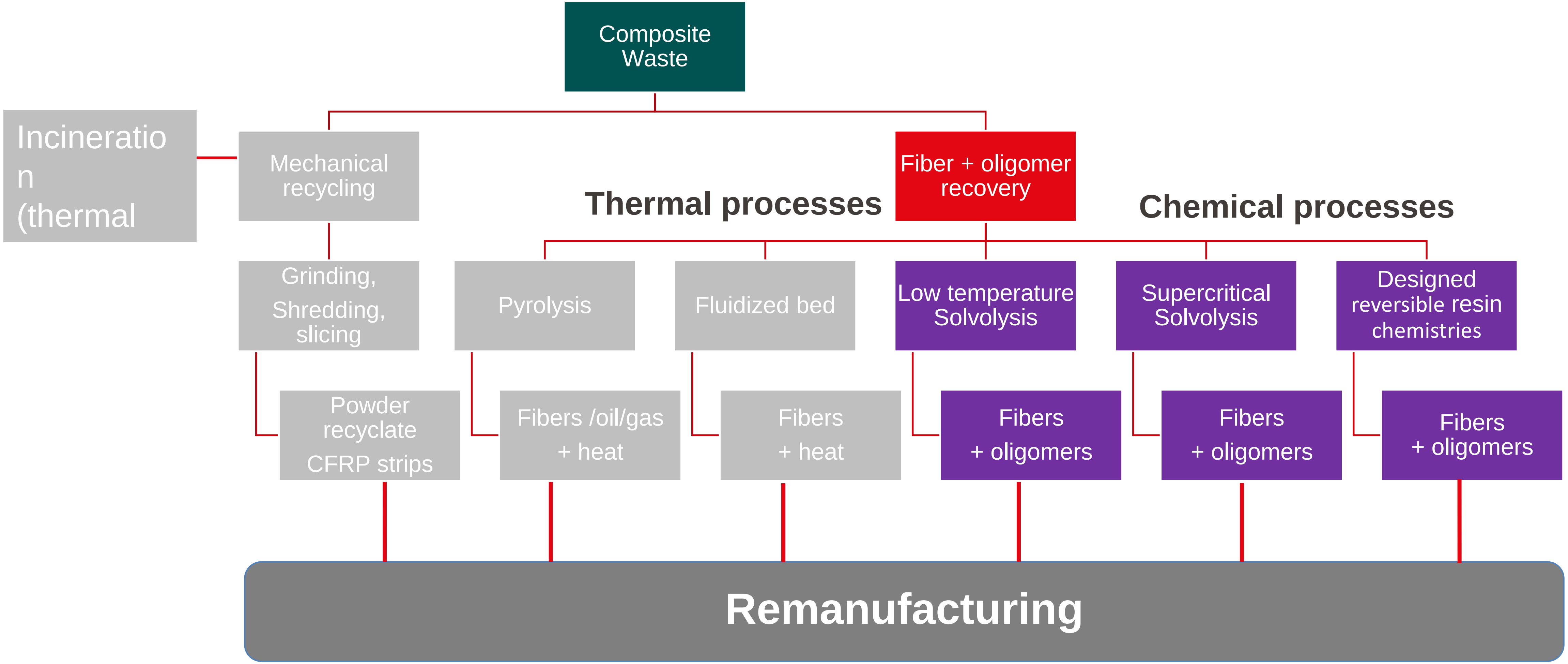
Pyrolysis oil candidate for re-synthesis into epoxy



(a) (b)

Evolution of pyrolysis char during the recovery of carbon fiber reinforced polymer composite and its effects on the recovered carbon fiber - ScienceDirect

Recycling Routes



Adapted from Pickering, Composites Part A (2006)

CF tensile strength retention by process

Table S1 The tensile strength retention of carbon fibers recovered by different methods.

Method	Tensile strength retention* (%)	Sources
Mechanical method	50-65	Zhu, et al [1]
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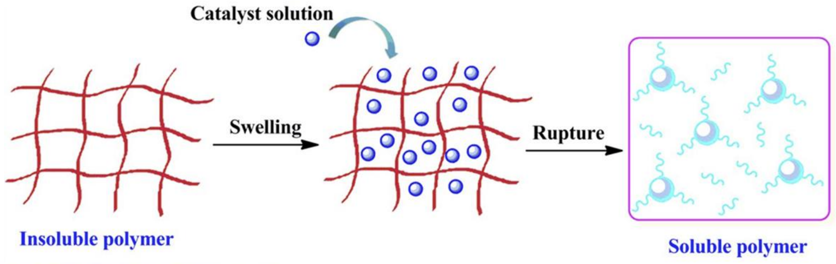
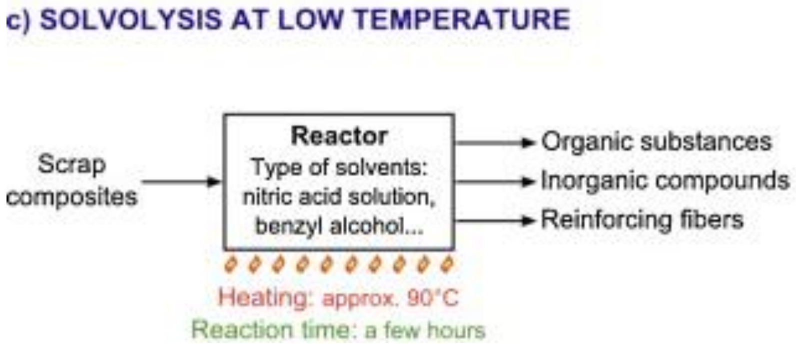
Evolution of pyrolysis char during the recovery of carbon fiber reinforced polymer composite and its effects on the recovered carbon fiber - ScienceDirect

EPFL

Low-Temperature Solvolysis





- Uses reactive solvents such as alcohol, ammonia or glycol
- Break down the chemical bonds of the epoxy or phenolic matrix
- Solvents can be toxic
- Yields fibers and organic liquid (mixture of monomers and excess reactive solvent)
- e.g. Hitachi Chemical
  - 180°C, benzyl alcohol solvent and tri-potassium phosphate catalyst, 5-20hrs
  - Used for tennis rackets
  - 63 MJ/kg vs 286 MJ/kg virgin CF

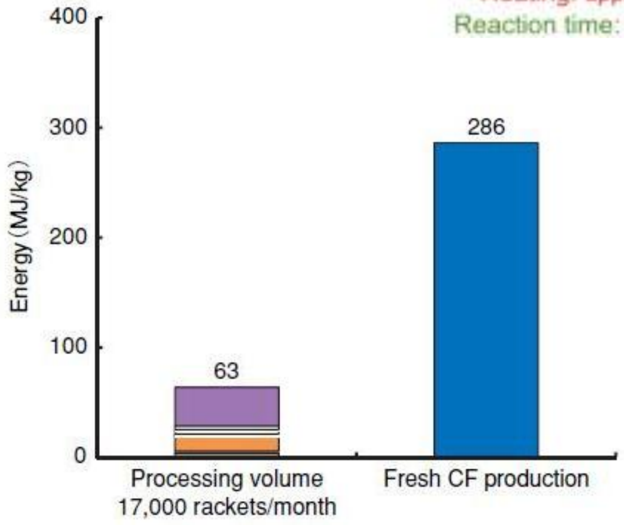


Figure 13 A comparison of energy of a recovered CF by dissolving method under ordinary pressure with a fresh CF on the market

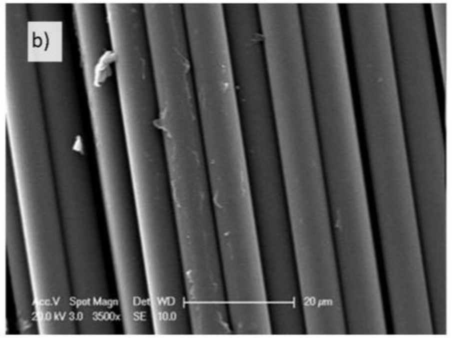
Item	Depolymerization under ordinary pressure
Organization	Hitachi Chemical
Temperature	200 ℃
Pressure	Ordinary pressure
Solvent	Benzyl alcohol
Catalyst	Alkali metal salt
Preprocessing	None
Processing capacity	12 tons/year (200 L x 2 baths)

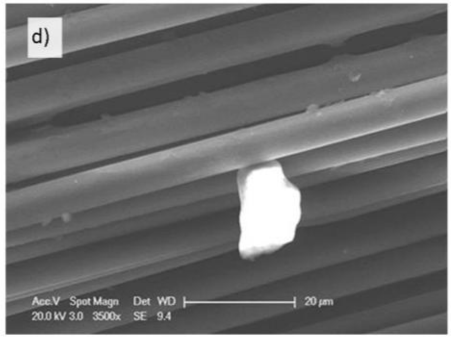
Near- and supercritical solvolysis of carbon fibre reinforced polymers (CFRPs) for recycling carbon fibers as a valuable resource: State of the art - ScienceDirect

Recycling carbon fiber for structural applications | CompositesWorld

Supercritical solvolysis

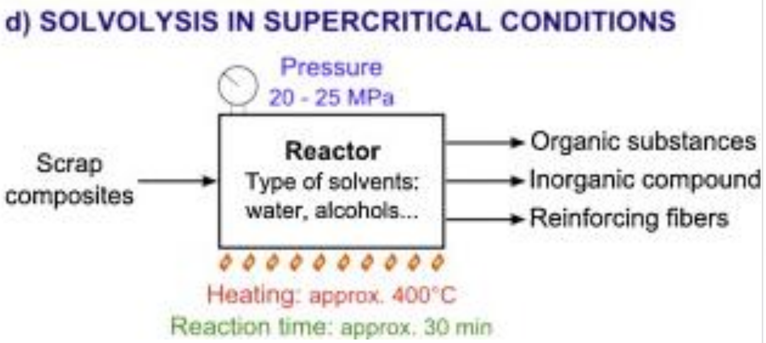
- Supercritical fluid
  - *Any substance at a temperature and pressure above its critical point, where distinct liquid and gas phases do not exist, but below the pressure required to compress it into a solid*
  - e.g. water 373°C, 220bar, alcohols, used as alternatives to organic solvents, and catalysts.
- Non toxic / low cost solvents
- 310-440°C, 15-30 MPa
- Excellent fiber property retention
- CF, GF, TP, TS materials
- End products: fibers, monomers, gas emissions (CO2, CO)

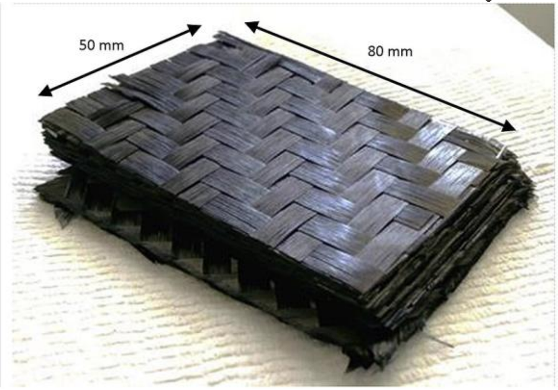


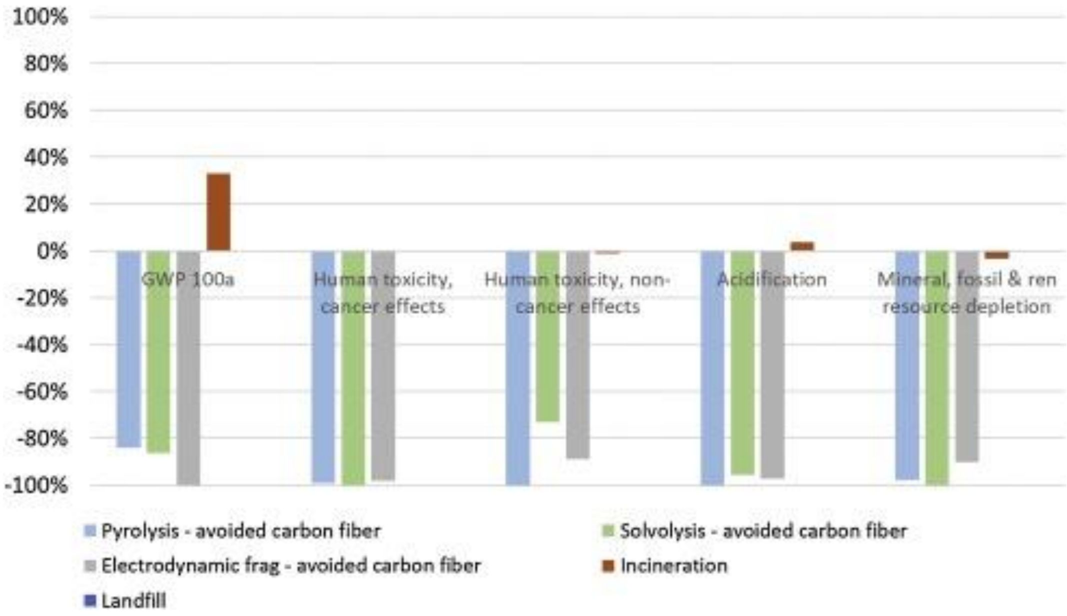


b) unwashed carbon fibres recovered after solvolysis; d) solid particle of partially degraded resin on slightly washed carbon fibres.

Recovery and reuse of discontinuous carbon fibres by solvolysis: Realignment and properties of remanufactured materials - ScienceDirect







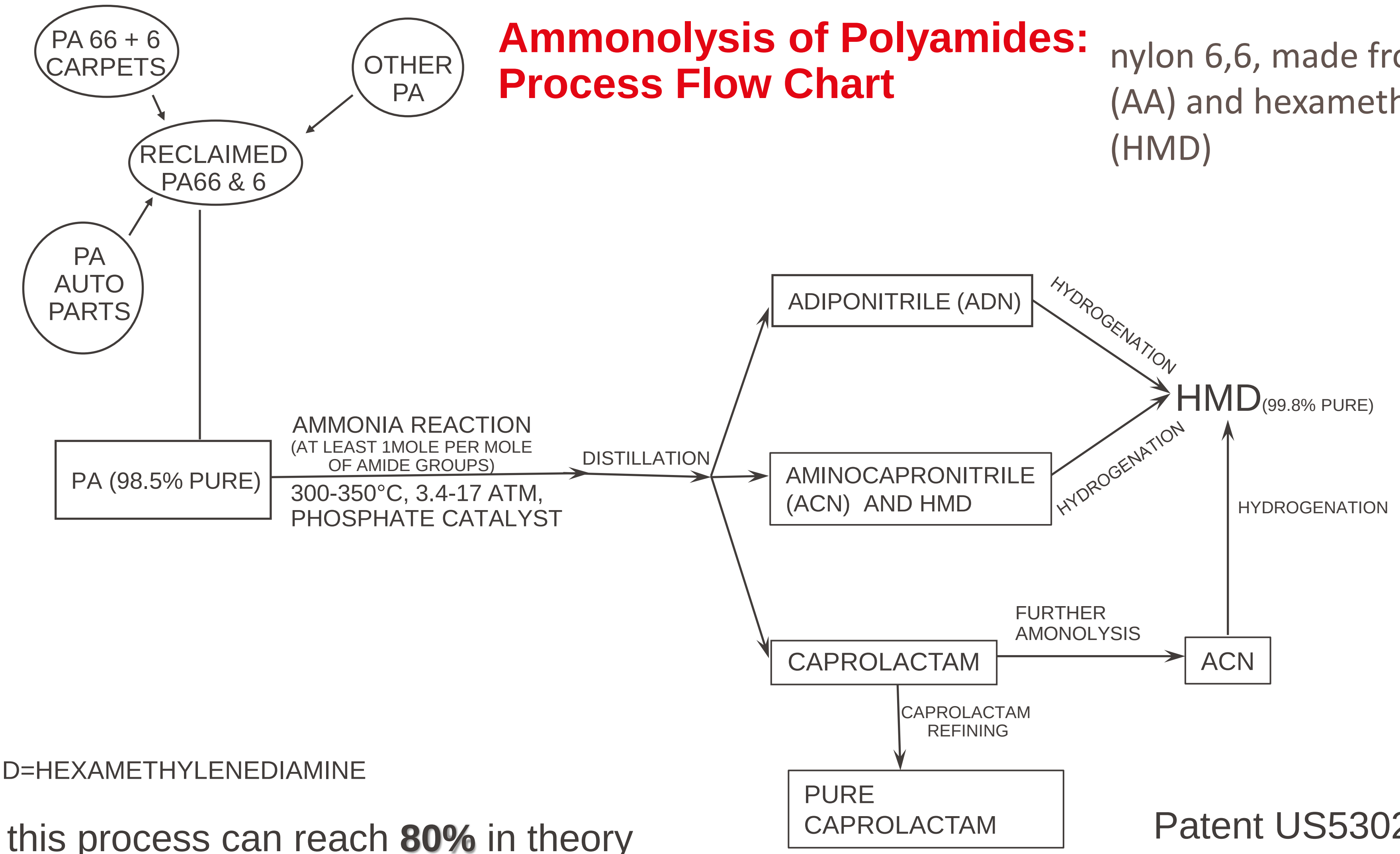
Near- and supercritical solvolysis of carbon fibre reinforced polymers (CFRPs) for recycling carbon fibers as a valuable resource: State of the art - ScienceDirect

Recycling of carbon fiber with epoxy composites by chemical recycling for future perspective: a review | Chemical Papers (springer.com)

PA chemical recovery (molecular recycling)

Ammonolysis of Polyamides: Process Flow Chart

nylon 6,6, made from adipic acid (AA) and hexamethylene diamine (HMD)



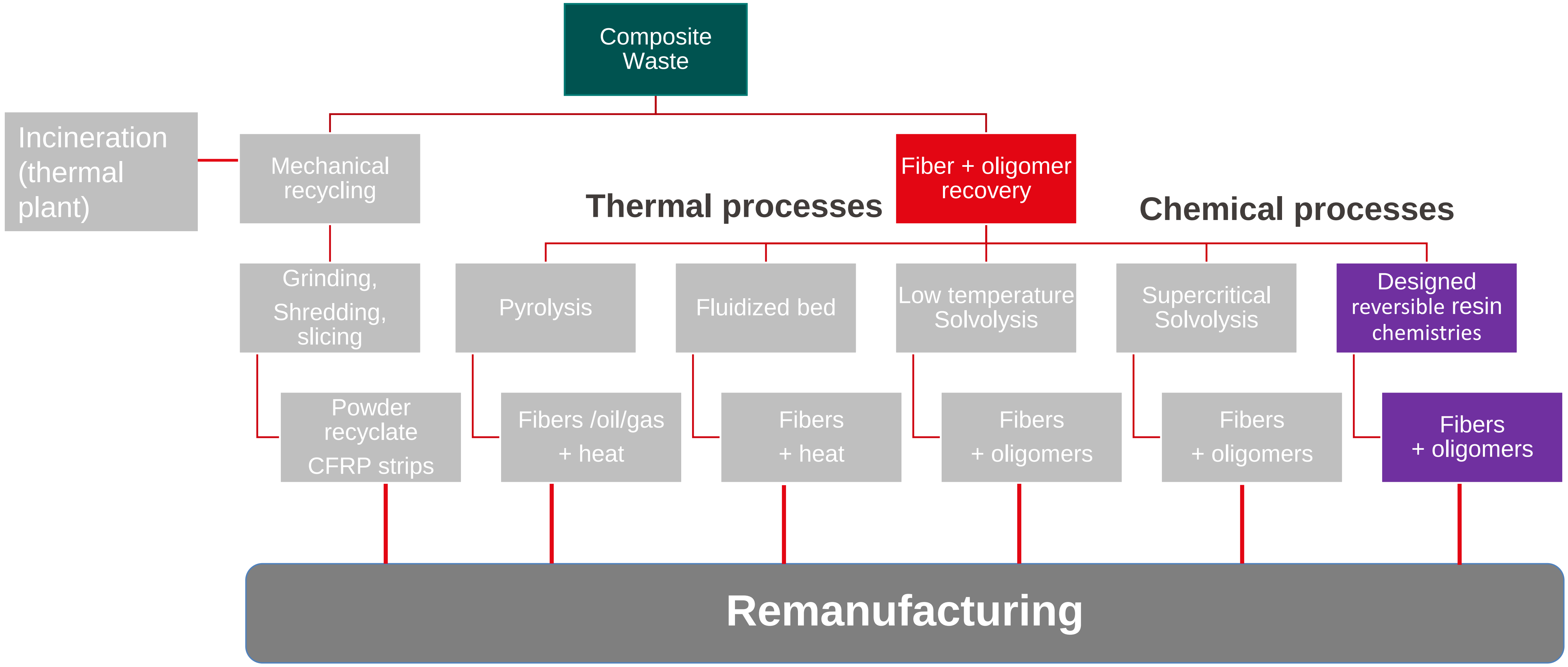
HMD=HEXAMETHYLENEDIAMINE

Yields on this process can reach **80%** in theory

Patent US5302756, Ammonolysis of Nylon, DuPont, 1994, McKinney

Wakeman

Recycling Routes



Adapted from Pickering, Composites Part A (2006)

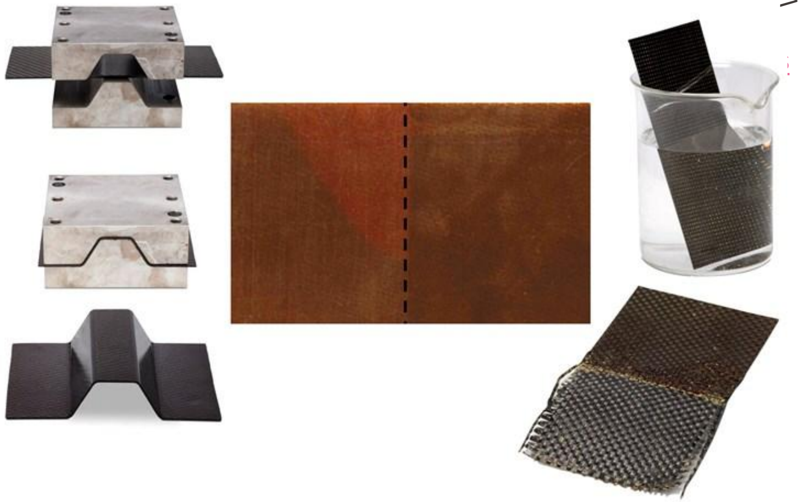
Chemical recovery of epoxy resins

Dissociative chemistry (the crosslinks are broken)

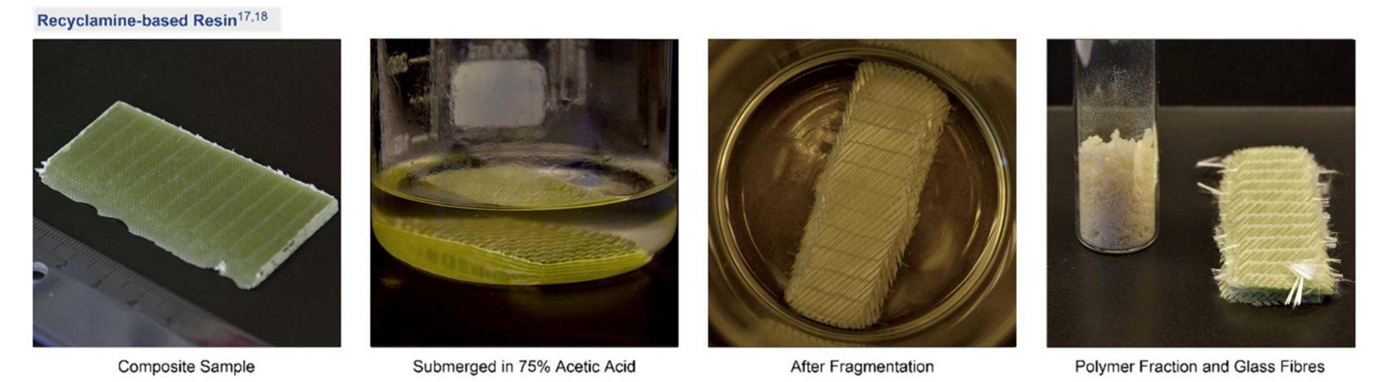
- Recyclamine
  - Low-pH solvent 25% vol acetic acid at elevated temperature converts resin to a thermoplastic epoxy while freeing the continuous fiber reinforcement
  - Both resin and fiber can be reused
- Epotec recyclable epoxy resins
- Thermoreversible Crosslinkable Thermoplast-Thermoset Hybrid (Evonik)

Associative chemistry (cross-links never broken)

- Epoxy vitrimer resins
- Provide cure cycles and properties comparable to conventional aerospace epoxies *but also allow a range of behaviors more akin to thermoplastics*
- thermoset properties below T_g, yet behave like a *thermoplastic* at elevated temperatures, such as 80°C above T_g



reprocessing via thermoforming (left), joining via welding, repair of delaminations using heat and pressure (center) and recycling by solvolysis (right) as well as by remolding ground up scraps



Reversibly crosslinking thermoset-thermoplastic hybrid polymers | CompositesWorld

Wind energy: recyclable blades

- Siemen’s RecycleableBlades (81m long) [Cleaver]

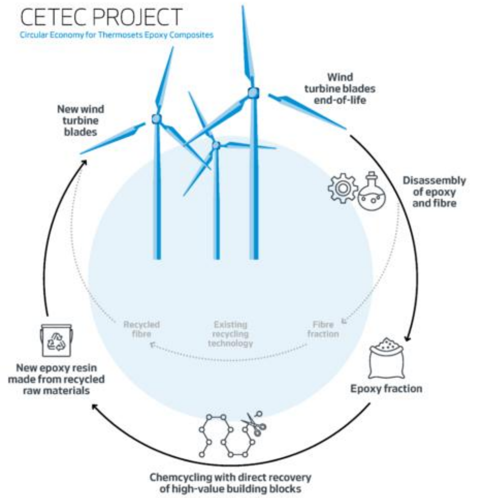
Novel resin	End of life	Recovery
• **Dissociative chemistry** • Ketal diamines (recyclamine) in hardner • Can efficiently separate the resin from the other components	• Blade immersed into a heated mild acidic solution (solvolysis) • 140ºC , 4-5hrs	• Separates resin from the fiber glass, plastic, wood and metals • Oligomer recovered, directly re-used

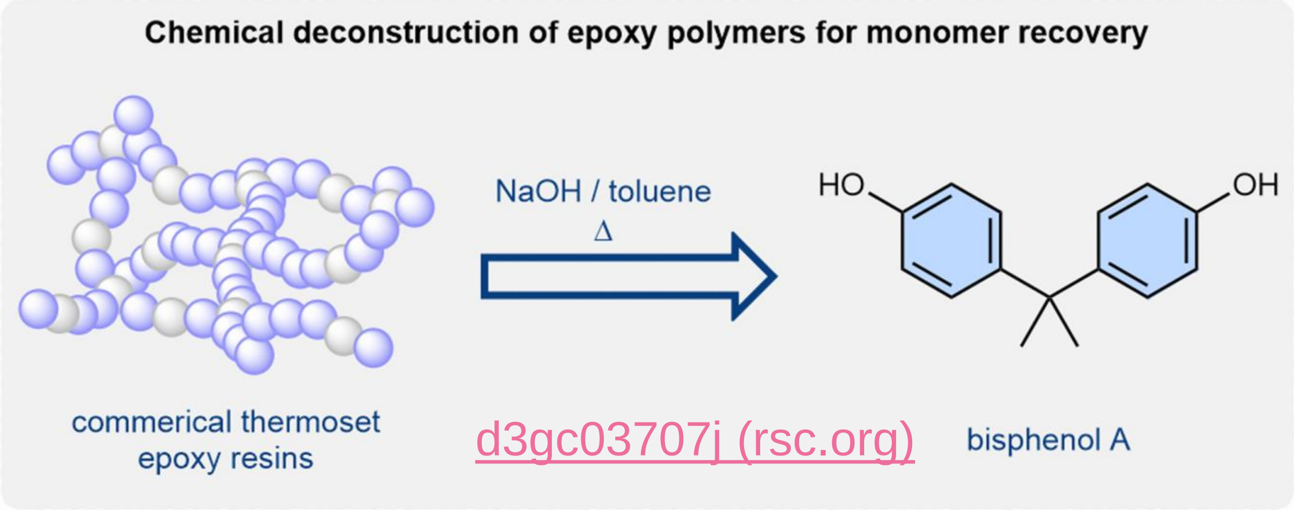
Swancor, Siemens Gamesa solidify recyclable wind blade partnership | CompositesWorld
Swancor launches recyclable thermosetting epoxy resin | CompositesWorld
SWANCOR: Leading into the Zero-Carbon Era: Recyclable Thermosetting Resin System EzCiclo and CleaVER (youtube.com)

World's first "fully recyclable" wind turbine blades roll off production line | RenewEconomy

Vestas (legacy & current)

- Recover BPA using solvent base mismatch (alkaline base with apolar solvent)
- 190ºC, 2hrs





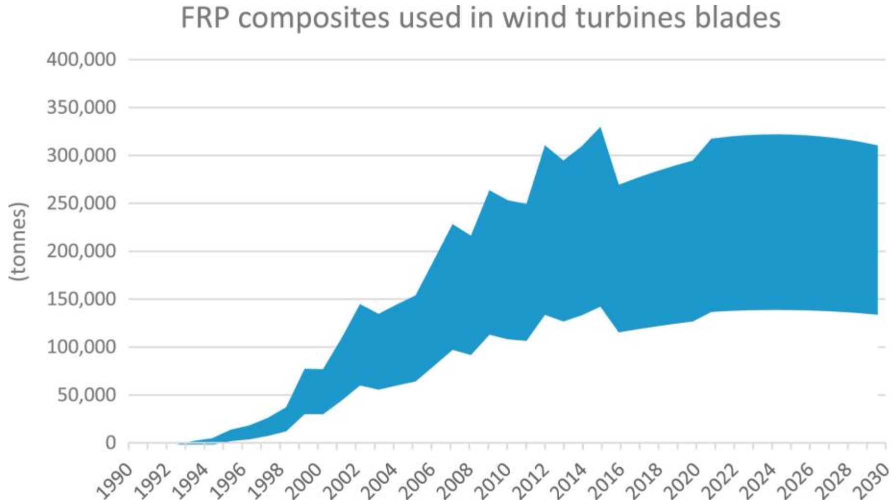
[d3gc03707j (rsc.org)]

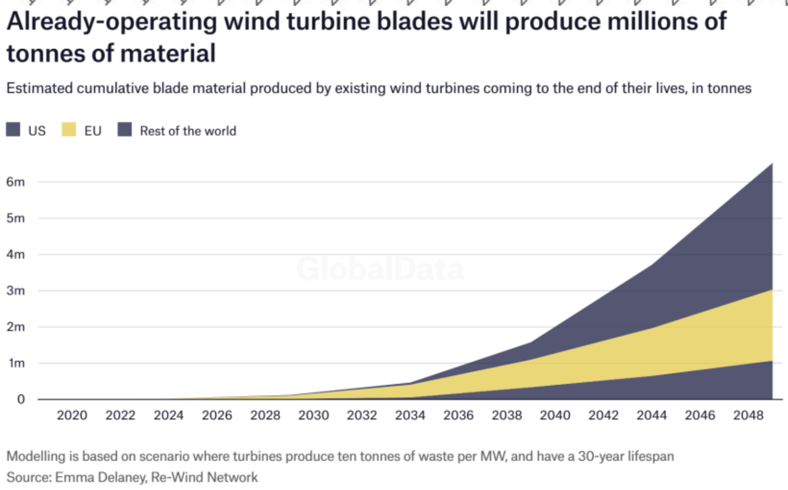
Renewables

Vestas hails breakthrough for recyclable wind turbines

A new chemical recycling process can break down the epoxy resin in wind turbine blades into virgin-grade materials.

Oliver Gordon | February 21, 2023

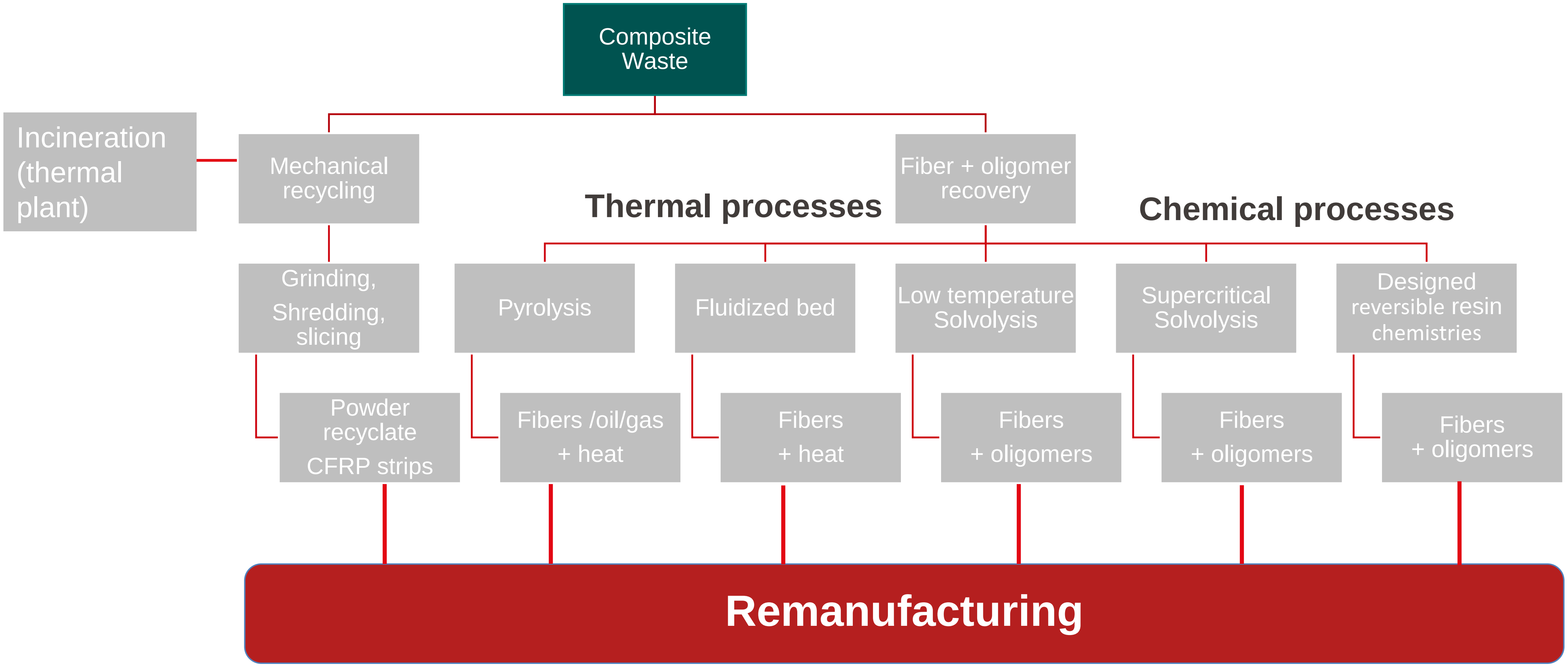




[Solvent–base mismatch enables the deconstruction of epoxy polymers and bisphenol A recovery - ScienceDirect]

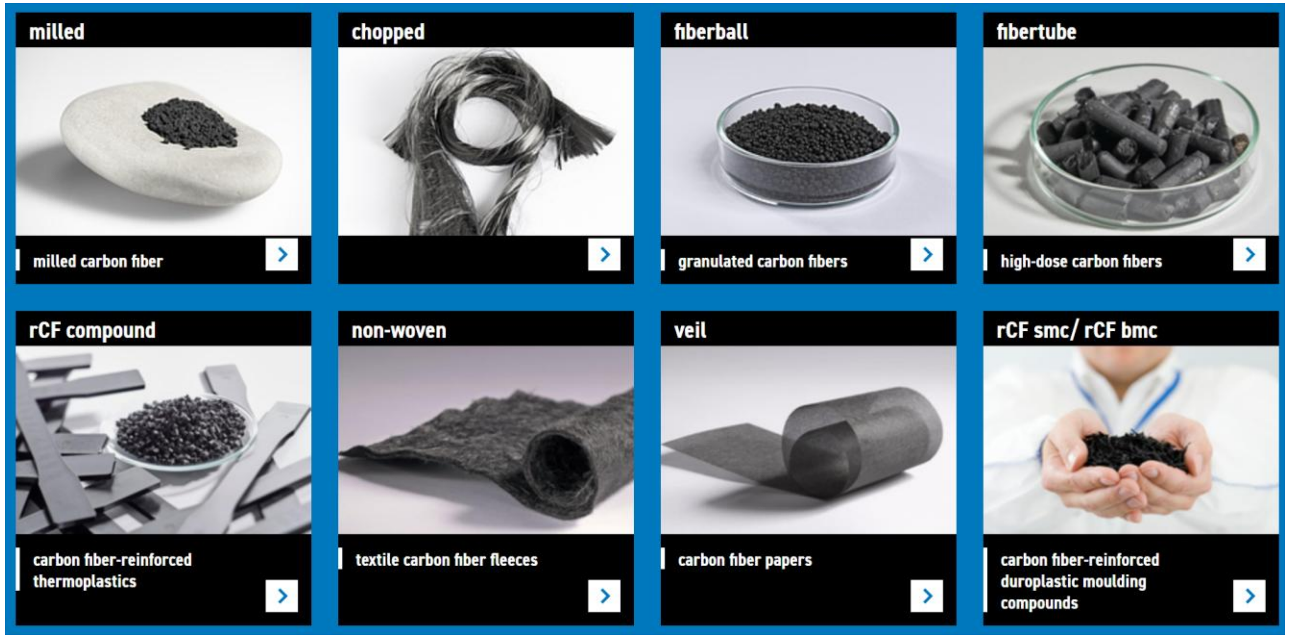
[Wind turbine blade recycling: Experiences, challenges and possibilities in a circular economy - ScienceDirect]

Recycling Routes



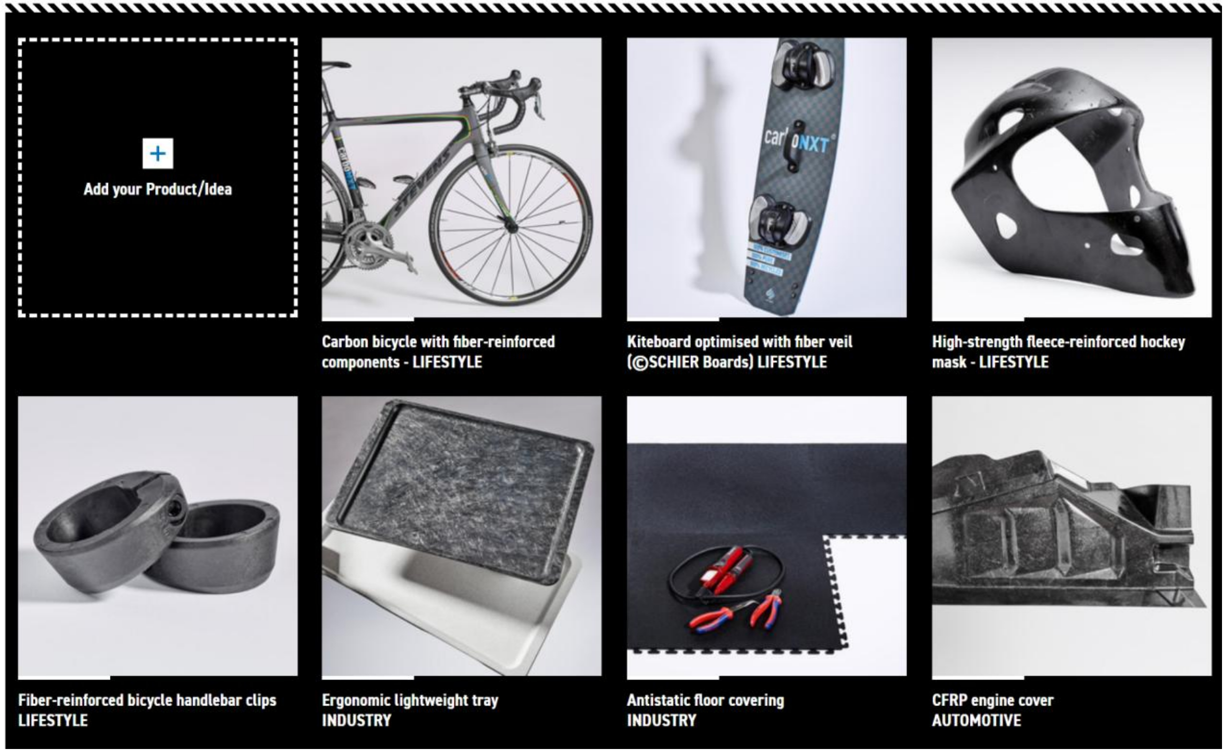
Adapted from Pickering, Composites Part A (2006)

Carbon fiber recycling & reuse



- Recovered CF material forms, 1000 T/yr

- Products using recovered CF



[100% CARBON FIBERS (carbonxt.de)](carbonxt.de)

MITSUBISHI CHEMICAL ADVANCED MATERIALS | carboNXT®

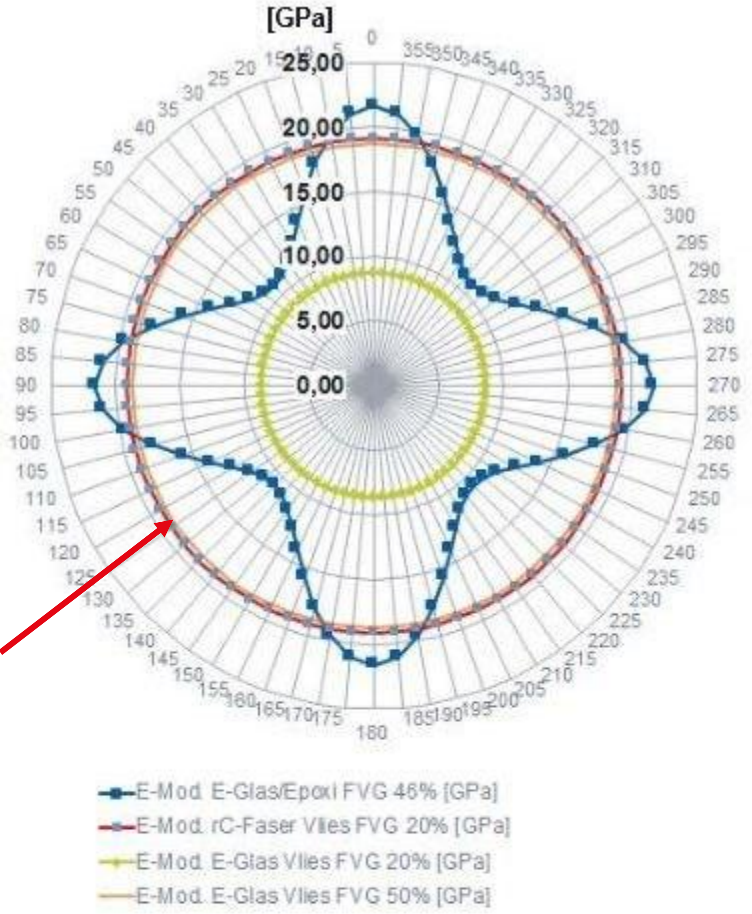
Procotex

- 2000 T/yr capacity
- Range of short fiber carbon materials
- Milling / cutting of CF waste
- Powder (60-300µm)
- Granulates
- Cut and resized fibers (300µm to 120mm)
- Fiber bundles (strands) 6mm

Recycled Carbon - Procotex

Gen 2 carbon

- Uses recycled carbon fiber to make
- Non-woven mats 50-500gsm, 1-2.5m wide
- Commingled with PP/PA/PEI/PPS
- Random fiber (isotropic)



Recycling carbon fiber for structural applications | CompositesWorld

G-TEX M - Gen 2 Carbon

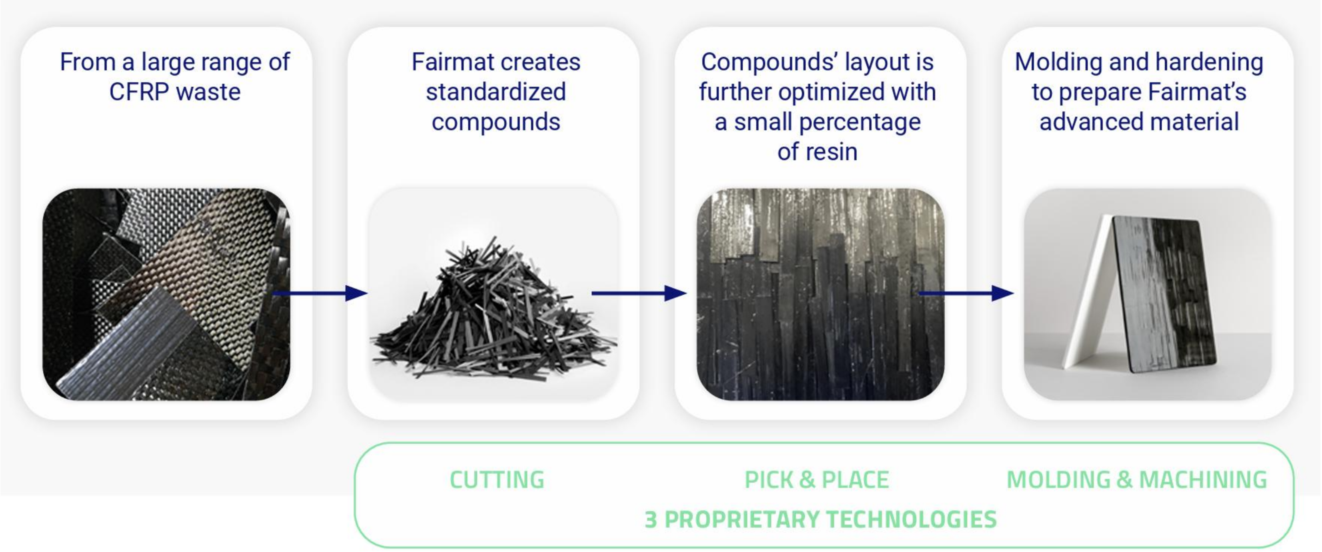
EPFL Fairmat

- 5000 T/yr
- Recycle post-industrial waste from carbon fiber prepreg production
- Mechanical treatment, robotics & machine learning to cut
- Maintain fiber length

Published 11/29/2021

Hexcel, Fairmat partnership aims to recycle carbon fiber prepregs

Cuttoffs generated by Hexcel's European plants will be repurposed by the end of 2022 into high value-added products



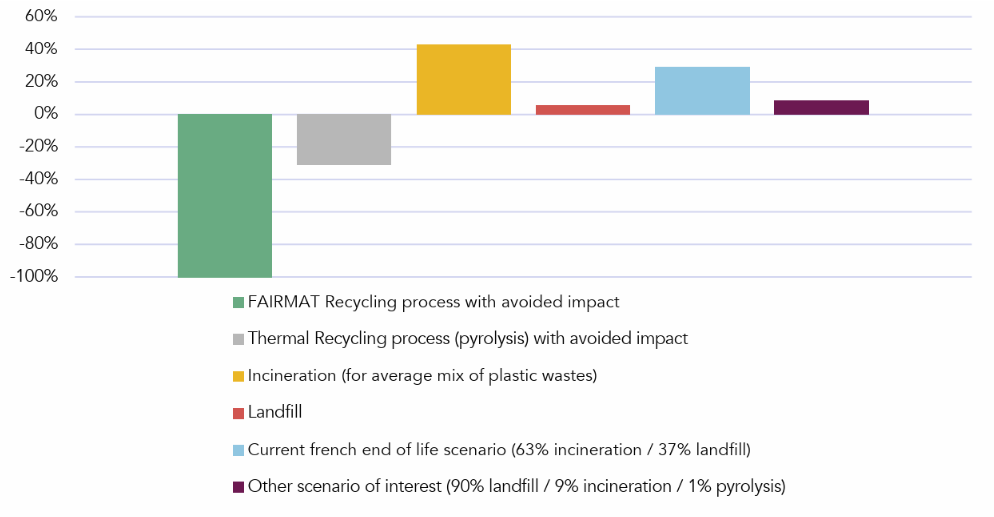
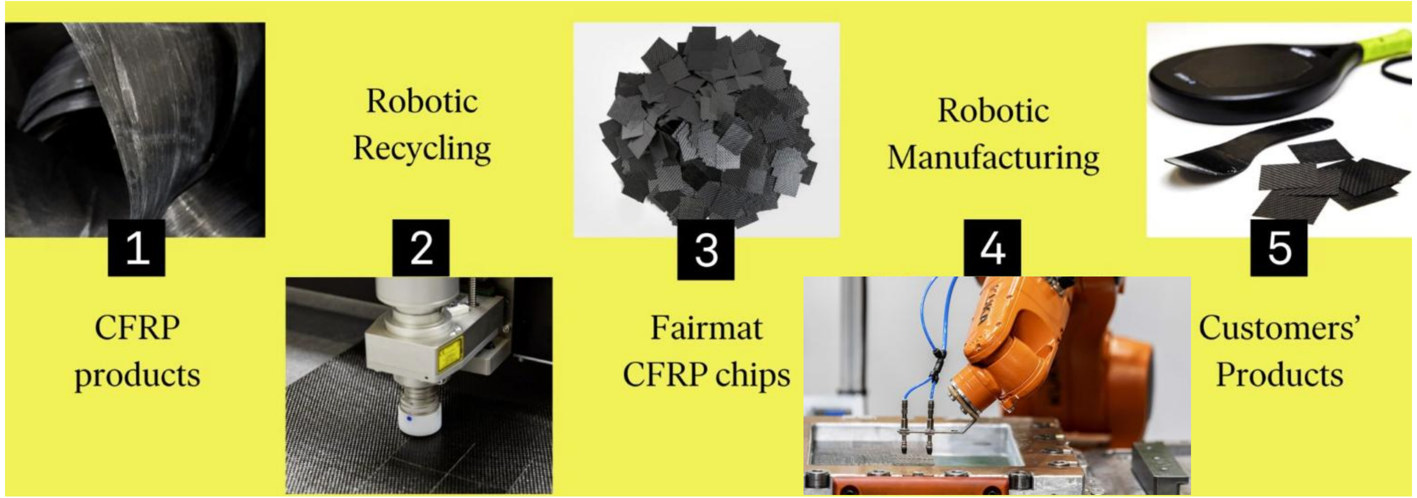


FIGURE 6: CARBON IMPACT COMPARISON BETWEEN FAIRMAT RECYCLING PROCESS AND OTHER END OF LIFE SCENARIOS CONSIDERING PRODUCTION OF RECYCLED MATERIAL



3.8 kgCO2e/kg Fairmat laminate

[FAIRMAT - Unlock new possibilities with recycled carbon fiber]

(re) aligning recycled and recovered carbon fiber

- HiPerDiF (University Bristol)
  - Uses water jets to align fibers 1-12mm (4mm today, 6mm trade-off formability vs. strength, >8mm forms flocks and blocks)
  - Tensile modulus of 115 GPa and tensile strength of 1509 MPa Vf 55%
  - Tapes 6.35-50.8mm wide, kg/hr
  - Used for 3D printing, potential for filament winding

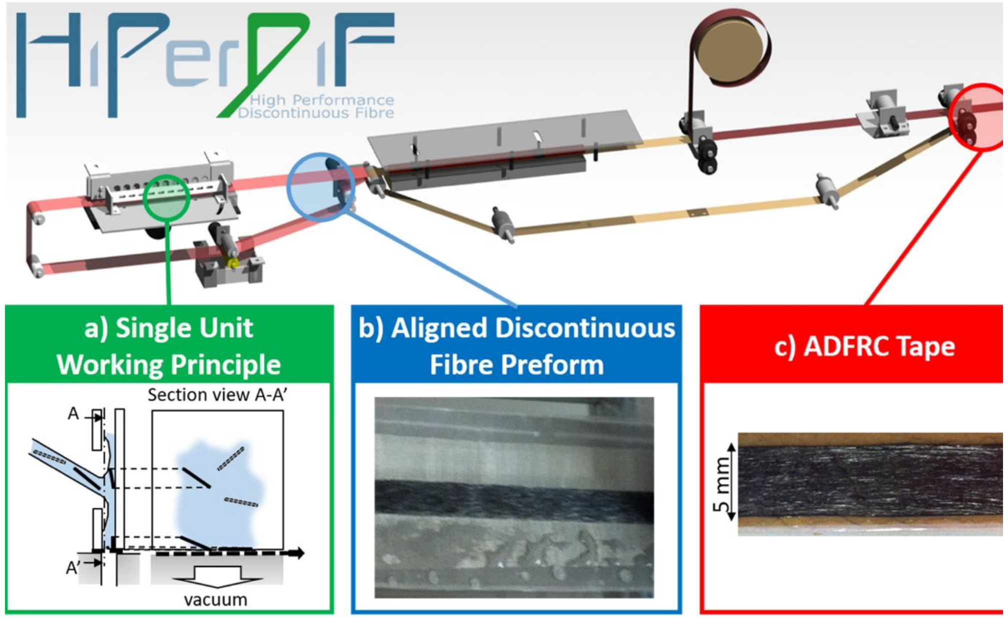
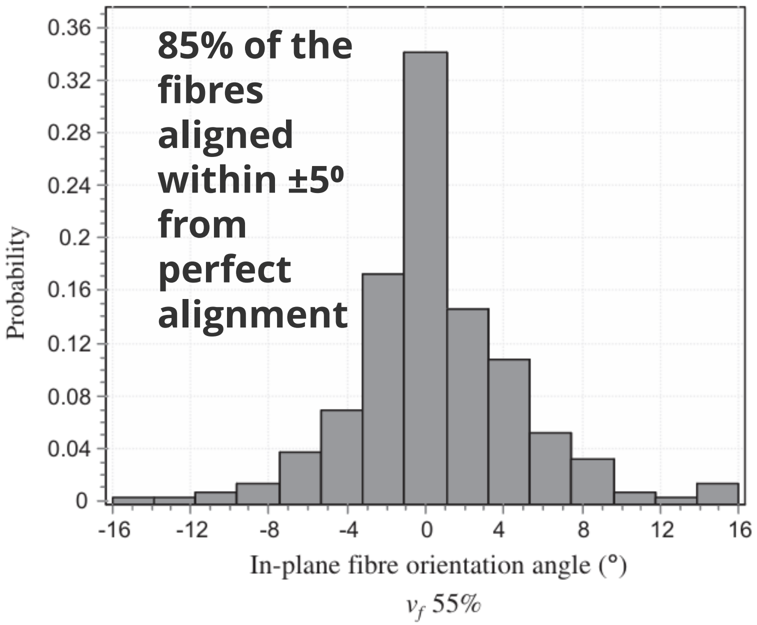
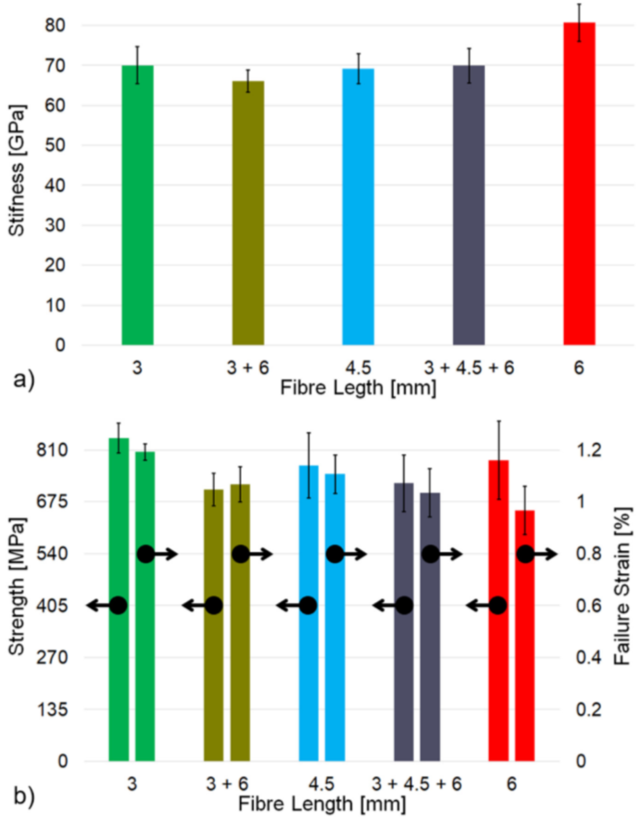


Figure 1. Schematic of the HiPerDiF alignment process.



v_f 55%

(b)



[J. Compos. Sci. | Free Full-Text | Remanufacturing of Woven Carbon Fibre Fabric Production Waste into High Performance Aligned Discontinuous Fibre Composites (mdpi.com)]

[A novel manufacturing method for aligned discontinuous fibre composites (High Performance-Discontinuous Fibre method) - ScienceDirect]

VIDEO PRODUCTION

FORMING DEMONSTRATOR – with NCC

National Composite Centre
Automated Tape Laying (ATL)
75mm width
Continuous fibre tape

ATL with continuous fibre

National Composite Centre
Automated Tape Laying (ATL)
75mm width
Lineat AFFT tape

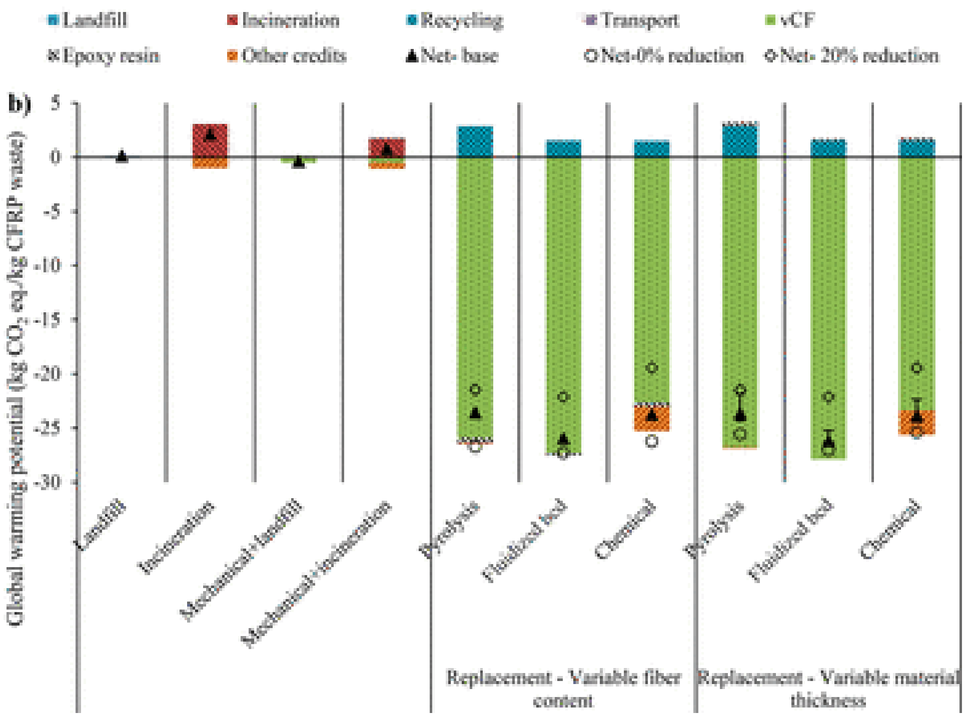
ATL with LINEAT tape

Critical steering radius | **8m** | **0.8m**

LINEAT

Recycled CF LCA

- Recycling processes can achieve
  - Global warming potential (GWP) from −19 to−27 kg CO2eq
  - Primary energy consumption (PED) from −395 to−520 MJ per kg CFRP
  - Superior environmental performance to conventional composite waste treatment technologies
  - All process significant improvement vs. landfill and incineration

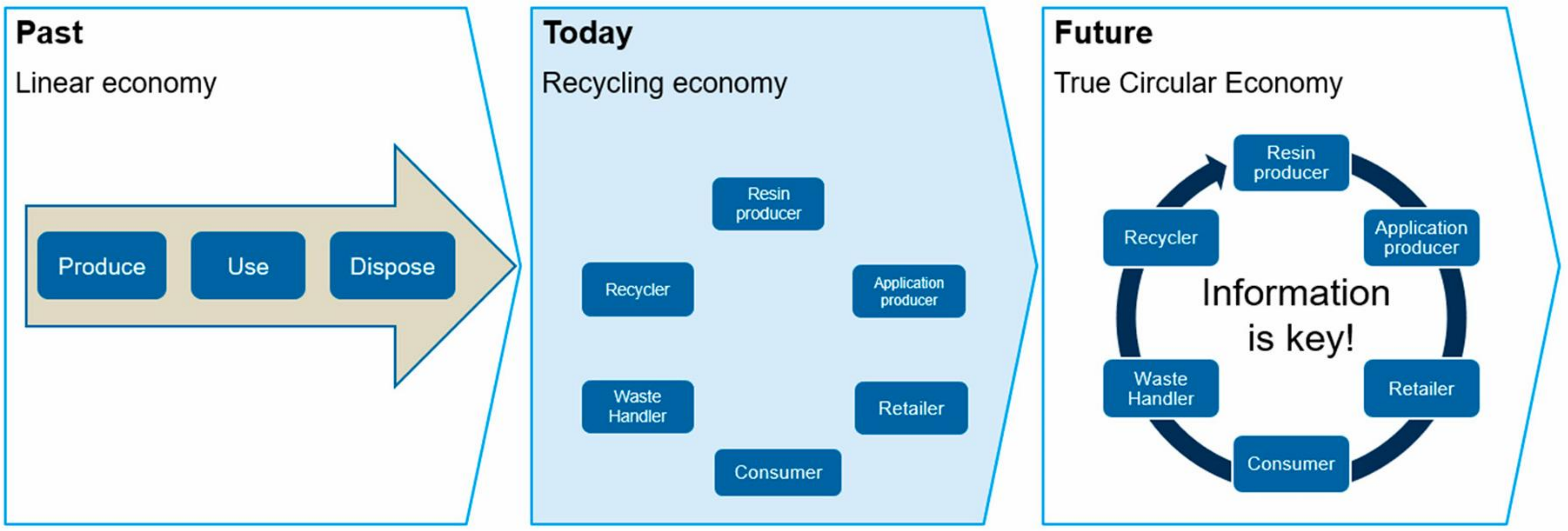


[Comparing Life Cycle Energy and Global Warming Potential of Carbon Fiber Composite Recycling Technologies and Waste Management Options (acs.org)]

Question

- How to go from non-standardized waste to standardized raw materials?
- Traceability / supply chain …

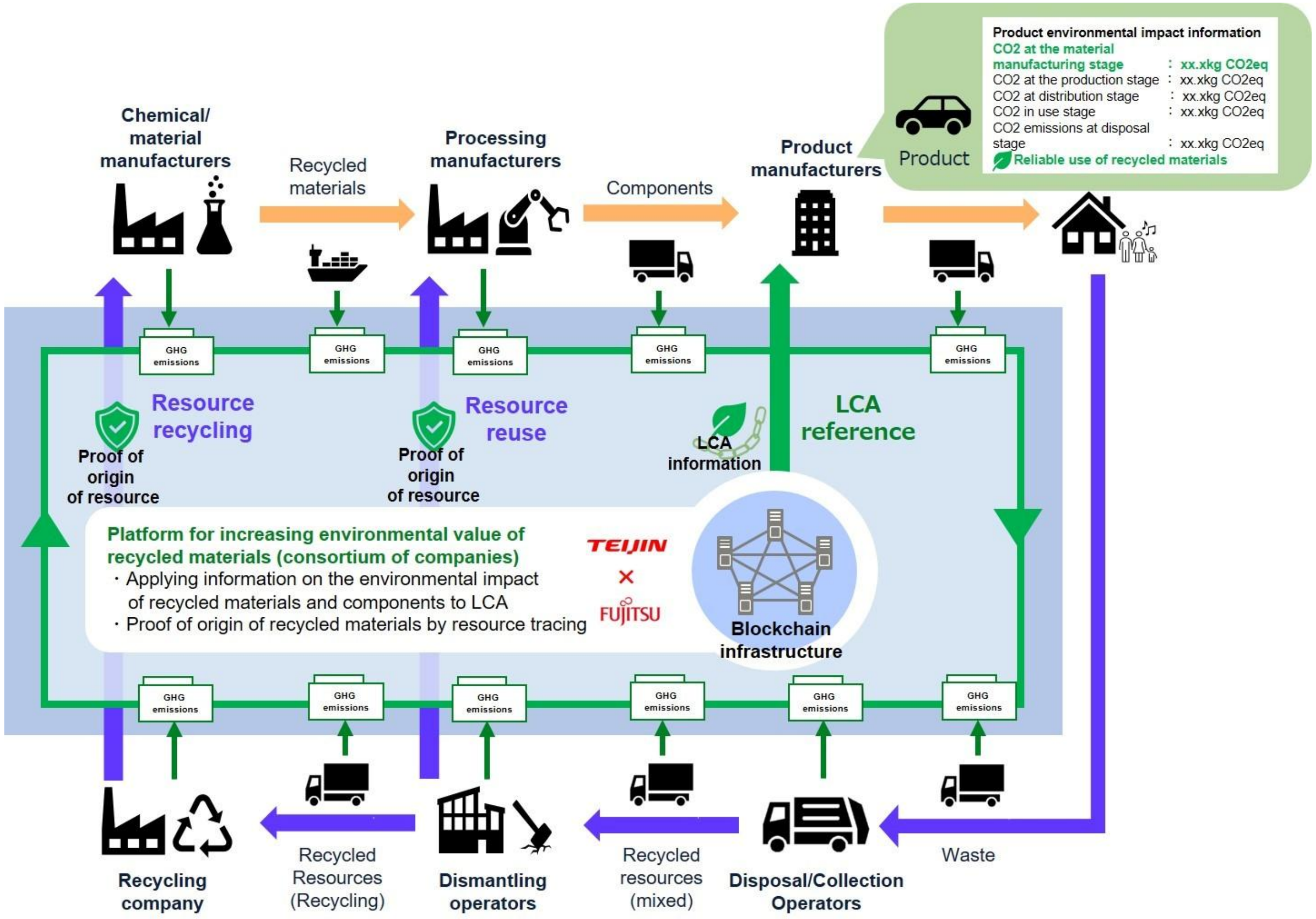
We need a transition in business models ...

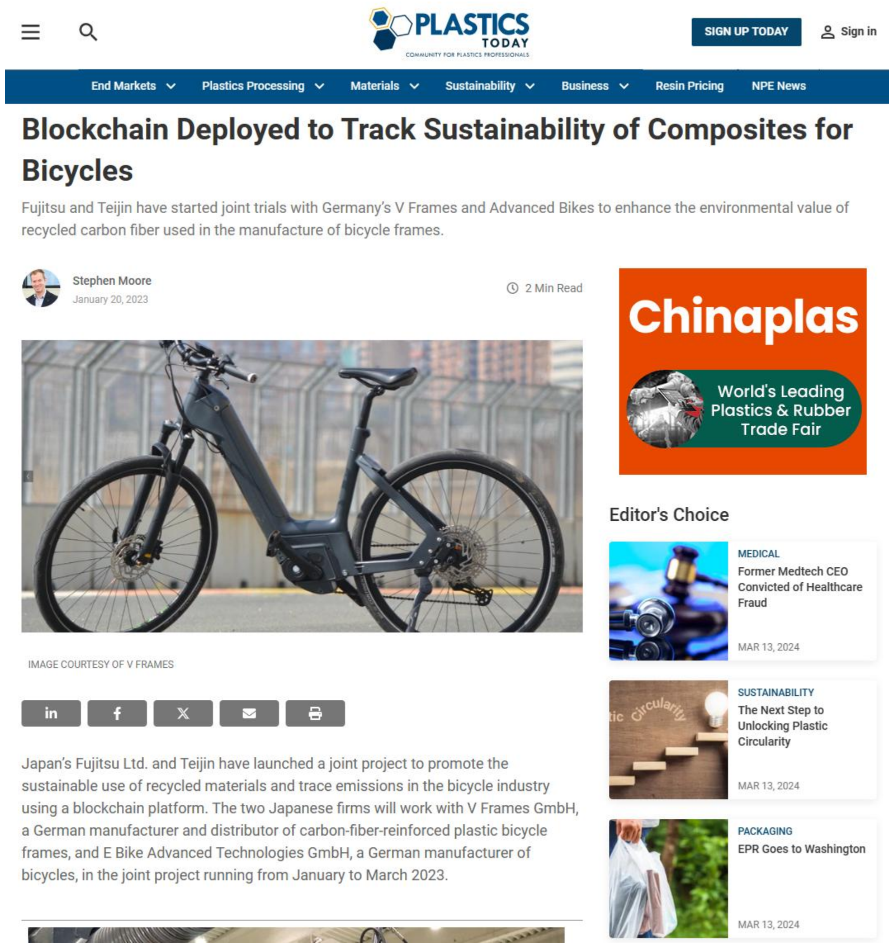


Polymers | Free Full-Text | Information-Based Plastic Material Tracking for Circular Economy—A Review (mdpi.com)

EPFL

Block chain enabled carbon fiber composite recycling





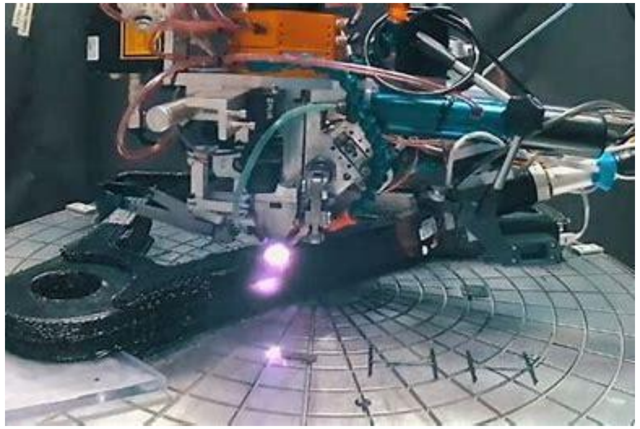
[Teijin, Fujitsu to develop blockchain-based commercial platform for recycled materials manufacturing | CompositesWorld]

[Blockchain Deployed to Track Sustainability of Composites for Bicycles (plasticstoday.com)]

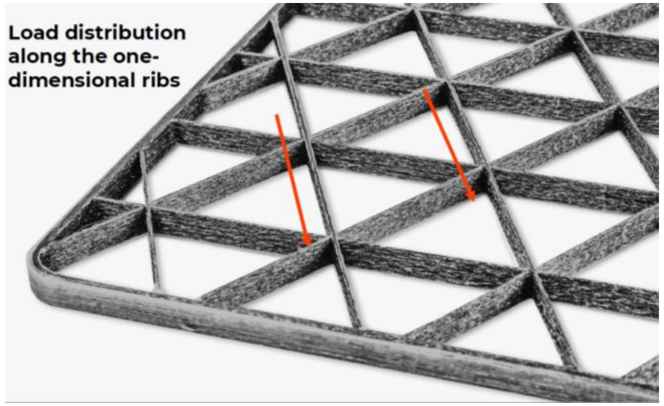
Overview - Towards sustainable composite solutions

- Bio-based / lower impact materials
- End of life and recycling
- Additive manufacturing
- Unmet challenges

Continuous carbon fiber 3D printing examples



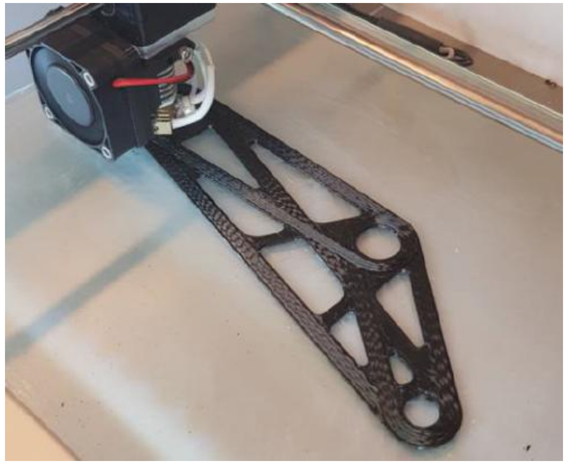
Arevo



Anisoprint

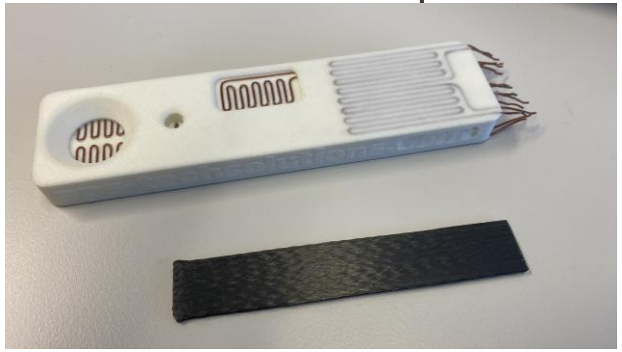
Ingersoll

Arris



9tlabs

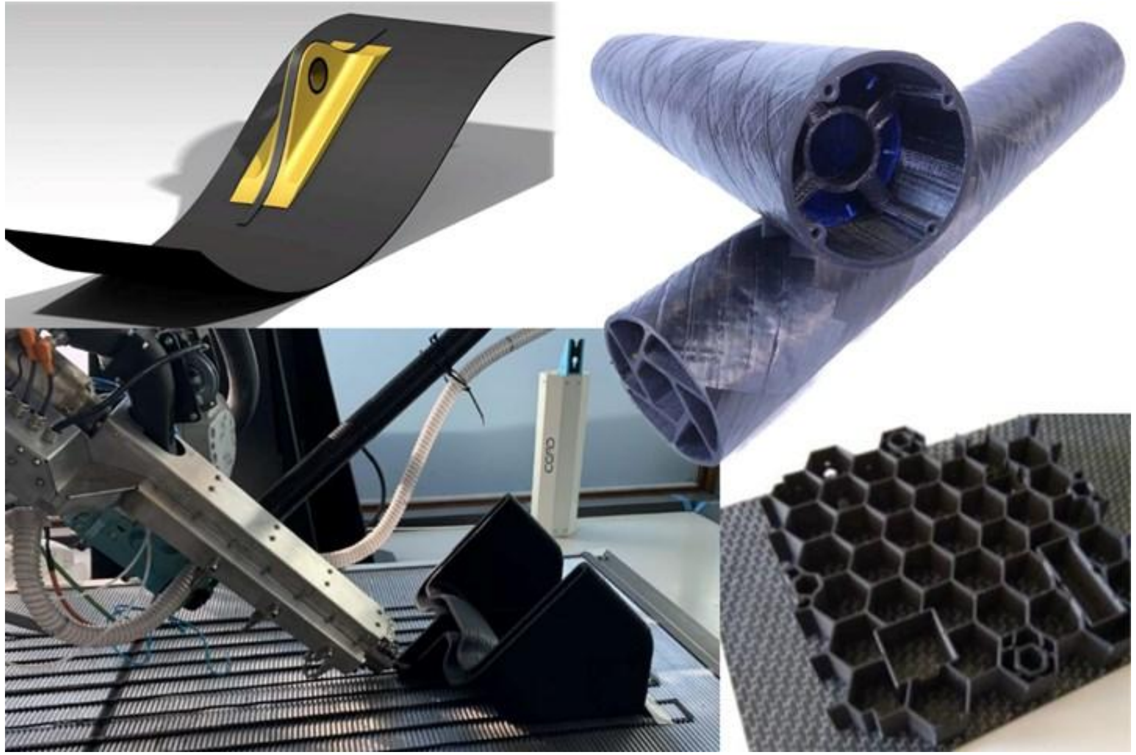
Continuous composites



APS

[(92) 3D printing of continuous carbon fiber | LinkedIn]

Merging of AM with composites manufacturing

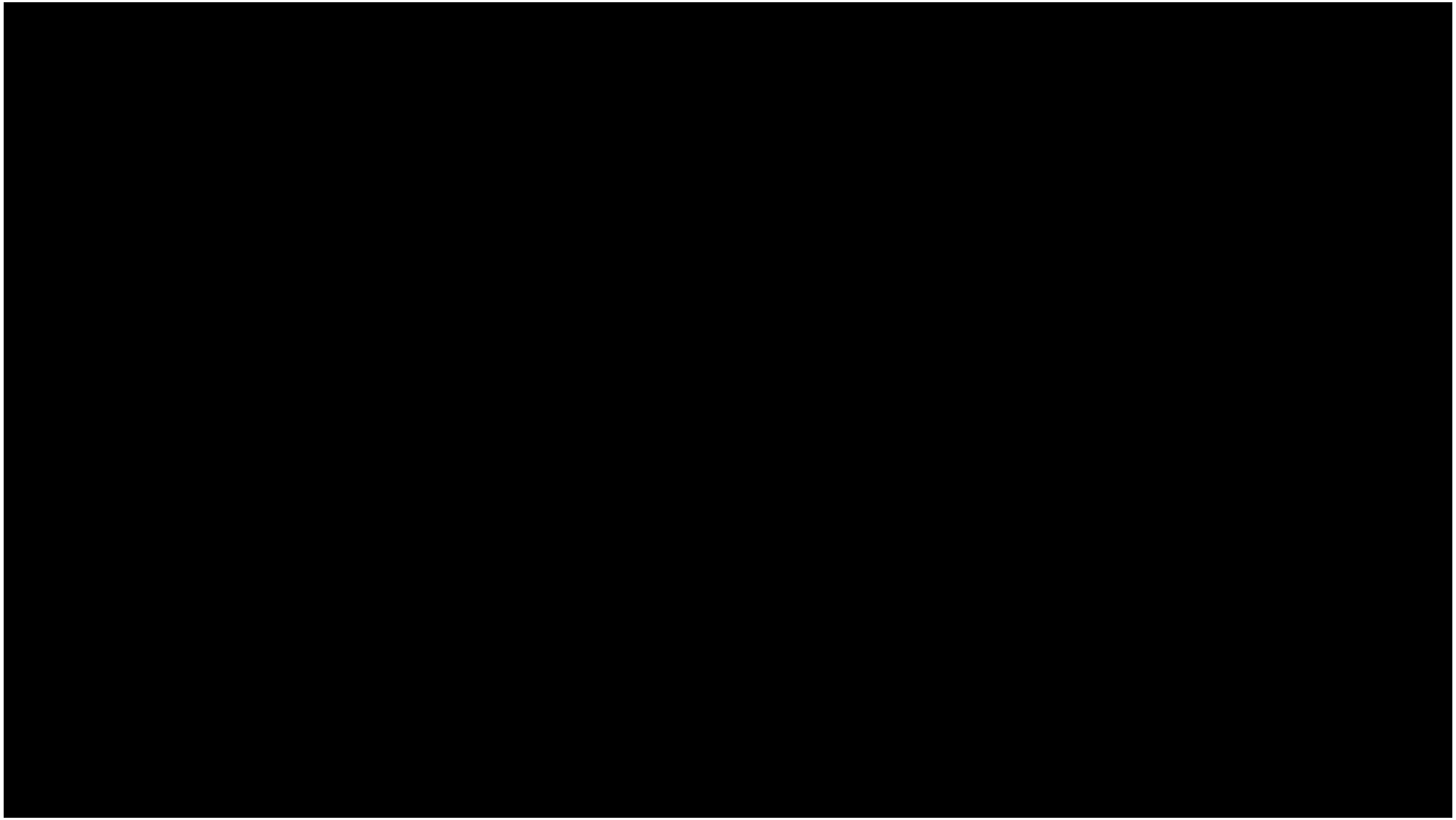


- Overprinting onto pre-made CFRP surfaces and brackets *(top left)*,
- hybridizing processes such as 3D printing and filament winding *(top right)*,
- 3D printing cores onto AFP laminates and AFP skins onto 3D-printed cores *(bottom right)*
- CEAD AM Flexbot 3D printing at 45 degrees *(bottom left)*.

[Coriolis Composites - Cpico #additivemanufacturing machine 🛠 - YouTube]

[Future composite manufacturing - AFP and Additive Manufacturing | CompositesWorld]

Merging of AM with composites manufacturing



Electroimpact Continuous Fiber 3D Printing - YouTube 1min20s

Overview - Towards sustainable composite solutions

- Bio-based / lower impact materials
- End of life and recycling
- Additive manufacturing
- Unmet challenges

EPFL

Challenges and opportunities to FRPC circularity

Technical	Market	Policy
• Limitations of materials • Complexity and dimensions of parts (e.g. bike frames to wind turbine blades) • Wide variety of existing materials, even different CF grades • Endless material combinations • Makes sorting for recycling difficult	• Constraints along value chain • Financial, Social, Behavioral, Logistical • Risk in developing recycling infrastructure versus waste supply heterogeneity • Issues developing economies of scale for recycling • Risk that recovered fiber can cost more (not always) than virgin fibers	• Inadequacy, lack of local national or European regulations • Some are outdated, others deemed too strict • Waste is still considered a hazardous waste not a resource • Limits trade and reuse and treatment • Contradictory agendas

[Circularity of Polymer Composites (baxcompany.com)](baxcompany.com)

Summarizing: a sustainable composites industry?

Efficient use of materials and energy

- Reduced waste
- Energy efficient processing

Smarter use of materials

- Design for recycling
- Net-shape automated manufacturing, AM
- Re-visit material formulation
- Simpler/ standardized
- Virtualized / TRLcomp.

Responsible sourcing

- Global/regional/local supply chains
- Reduce use of hazardous materials/ constituents
- Petro chemical to non-food bio-mass

In service

- Durability, increase life / use phase
- Sensors, IoT Ind4.0, functionalized
- Easy repair
- Lease / maximize use

End of life management

- Recycling coalitions
- Investment in recycling CAPEX
- Standardized processes
- Traceability, Blockchain

Simple recyclate streams

Recover fibers (for lower specification applications)

Cracking to universal small molecules (beyond composites)

Return to raw

Suppress landfill / burning

Leaky loops

Foundations

Legislation & incentives	Collaboration Respect & Trust	NetPositive: Regenerative	Tech. innovation, entrepreneurship	Use renewable energy

Create bigger circles (rather than small complex expensive circles)