Production of microfibrillated cellulose using stirred media mills and selected applications

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Outline

- Introduction
- Stirred media mills
- Product characterisation
- Influence on particle size and fibrillation
- Optimisation for various fibre substrates
- Applications of MFC
- Product stewardship

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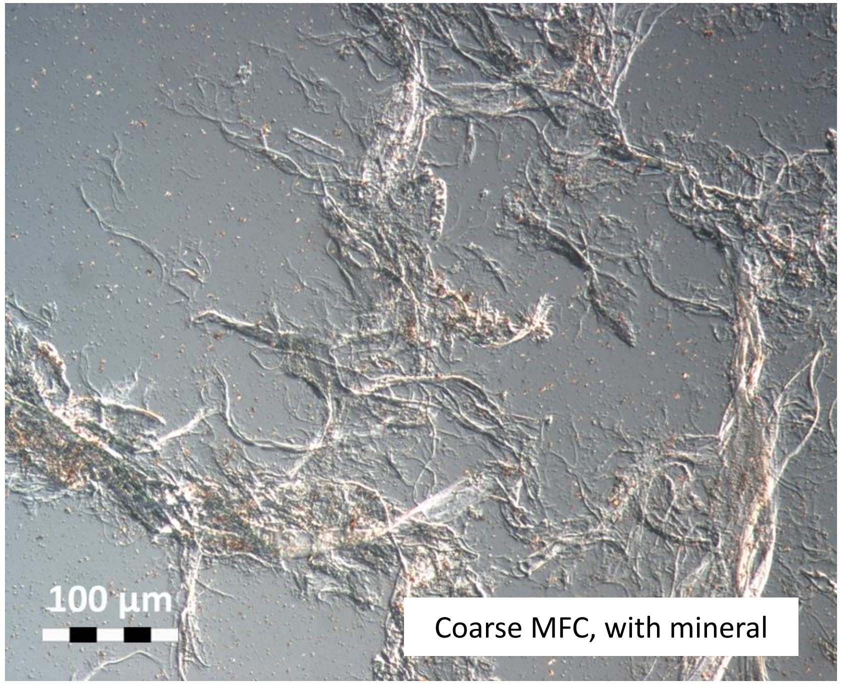
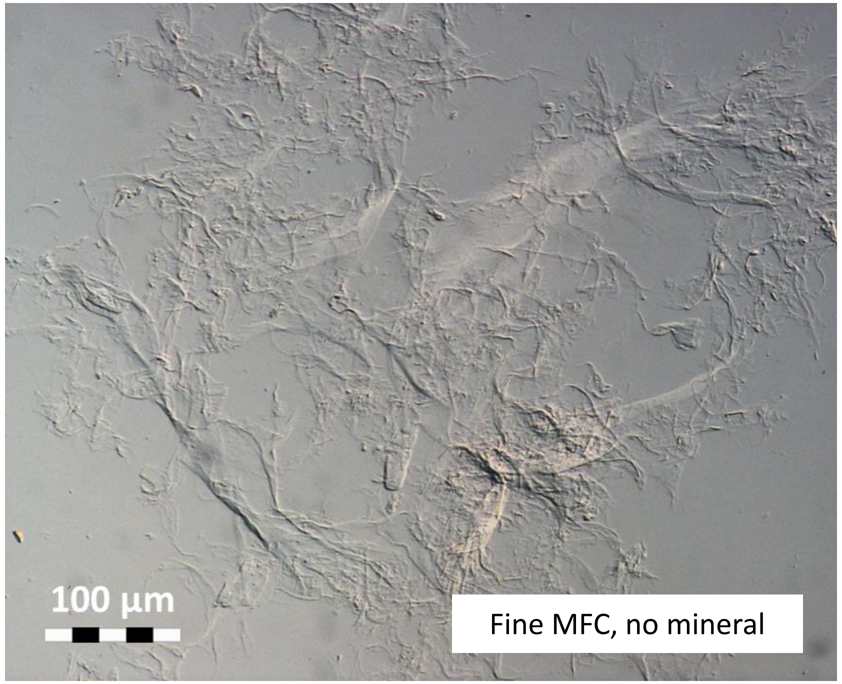
Introduction

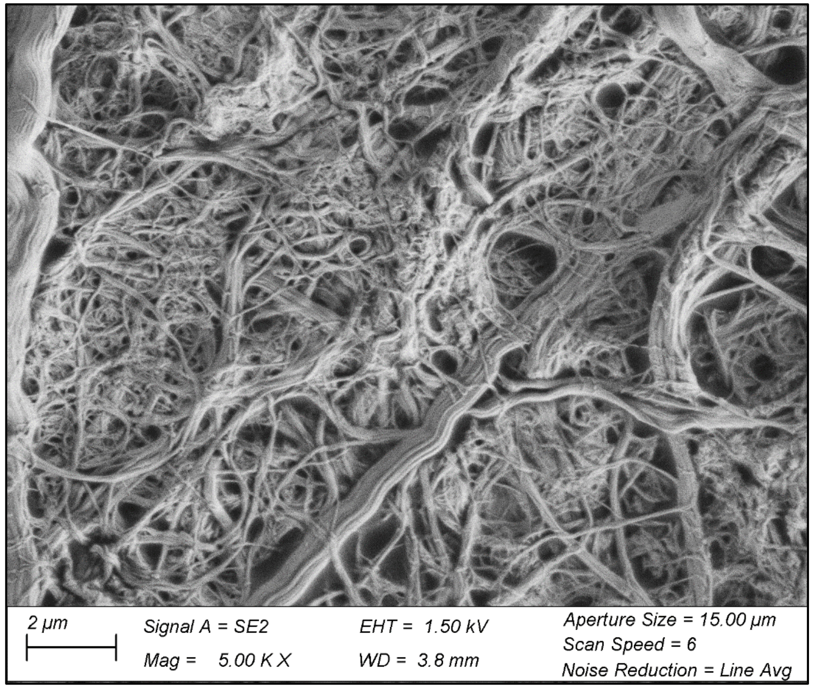
- **MFC** – produced by **mechanical treatment** of cellulose
- Highly viscous aqueous suspension
- Typically **satellite production** adjacent to final use location
- **Continuously produced at large scale using stirred media mills**
- **Flexible process enables a wide variety of product characteristics**

Product families

- **MFC from 100% virgin pulp**
- **MFC from recycled fibres**
- **MFC mineral composites**
- **NB Two of these families have no added minerals. MFC only**







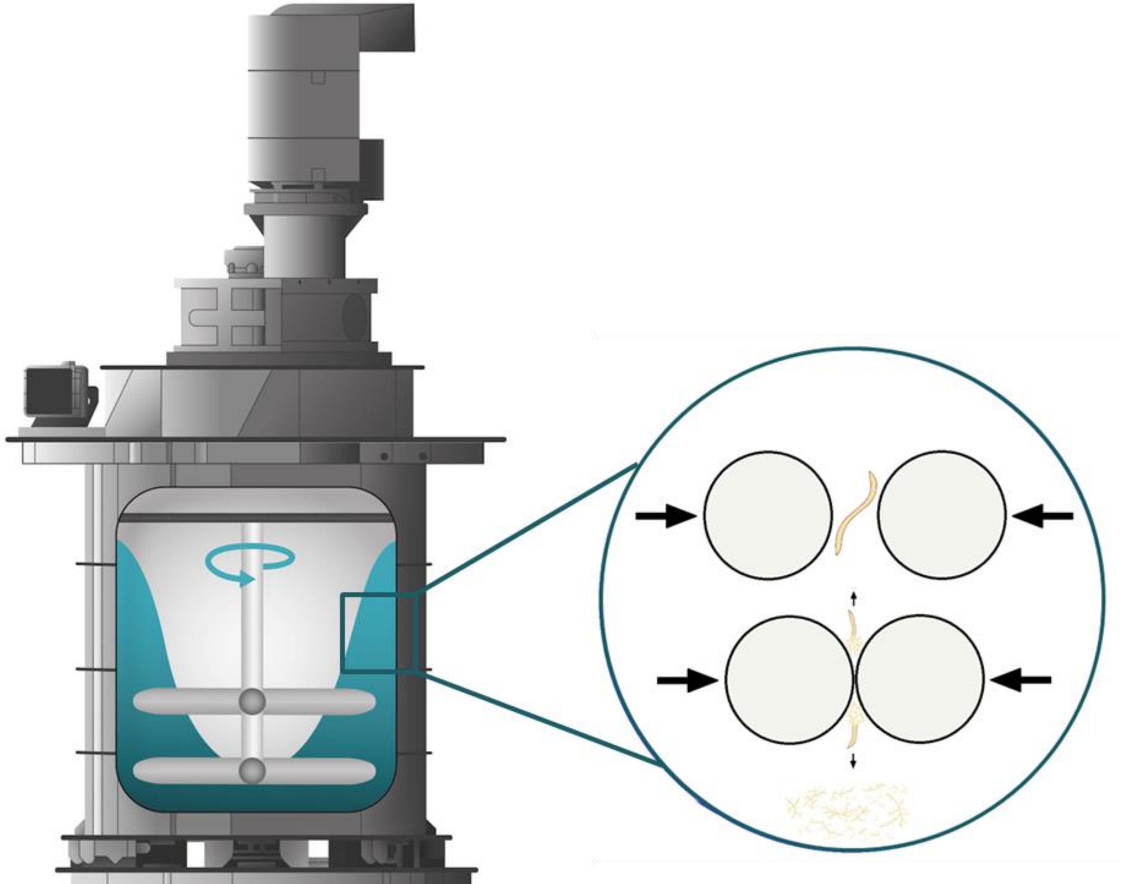
Pilot-Plant Production Facility, and Product Forms

Production plant in the UK, **2000 dry metric tonnes pa of fibril capacity**. Operational since Q4 2013: **Slurry** (< 2% fibre solids) and **press-cake** (10 – 20% fibre solids) product forms

Slurry

Press cake

Stirred Media Mills – Introduction



Grinding media collisions disintegrate fibres into microfibrillated cellulose

- ***Stirred vessel***, where ***collisions between grinding media beads*** break intervening particles.
- Widely used in minerals and mining industry due to efficiency, scale, and flexibility.
- We have adapted this technology to ***break and fibrillate fibres into MFC***; requires modifying theory and operating principles.
- ***Very high active surface area*** of media and ***inherent scalability of stirred vessels*** permits ***high throughput*** and ***continuous production*** of MFC.

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Stirred Media Mills – Advantages

For large-scale MFC production, stirred mills confer many benefits:

Video of Grinder

- **Robust technology, operational since 1950s for minerals processing, since 2013 for MFC**
- **No close tolerances or precision engineered components**
- **Continuous single stage process**
- **Availability > 95%**
- **Low capital and running costs**
- **High throughput in a small footprint (typically >1000 dry tonnes / annum per grinder)**
- **Modular easily-scalable design**
- **No additives or pre-treatments**
- **Flexibility in tailoring MFC properties**

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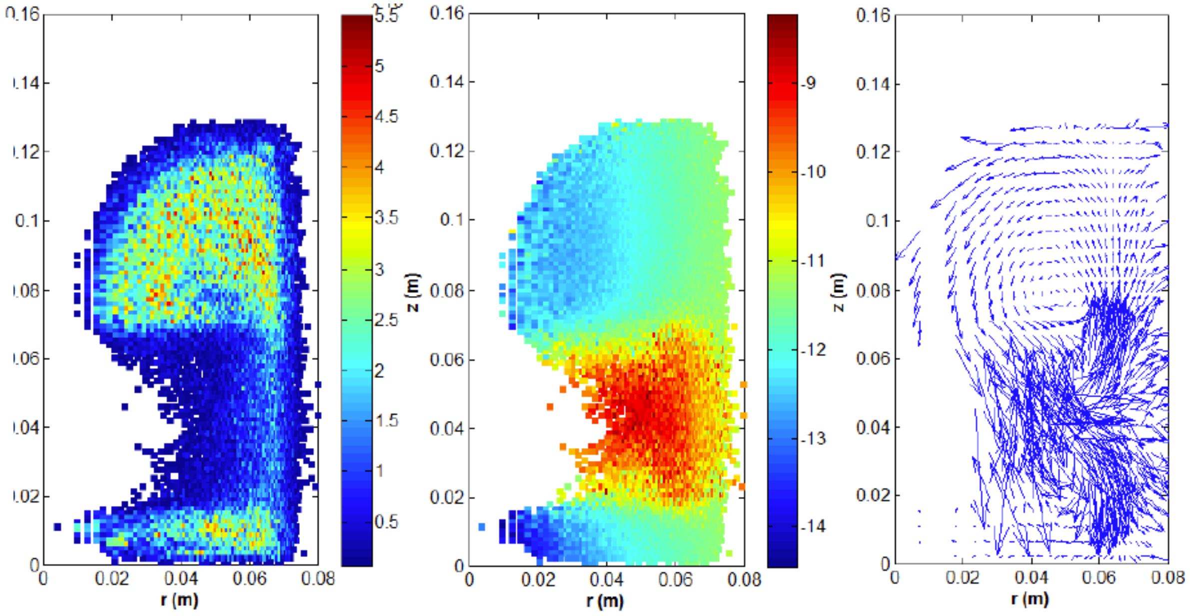
Stirred Media Mills – Optimisation

- Unlike minerals processing, where minimising particle size is usually the goal, effective MFC production requires ***high surface area generation whilst maintaining fibril aspect ratios***.
- Stirred mills are ***conceptually simple***, though ***optimising is complex*** due to the number of parameters (charge formulation, grinding media properties, machine operation parameters, grinder geometry); a purely empirical approach is not sensible.
- Effective optimisation requires the following:
    1. An ***intimate understanding of the feed fibre properties*** (i.e. what forces are required for breakage and fibrillation).
    2. Tailoring the ***type, frequency and magnitude, of forces*** applied by the media to the fibres.
    3. Modifying the energy distribution within the vessel by ***controlling flow patterns***.
- Stirred mills have the key advantage that the ***strength of forces can be varied by many orders of magnitude with little to no equipment modifications.***



PEPT tracking *of a lab-scale grinder – (left) occupancy, (middle) kinetic energy distribution, (right) velocity vectors.*

Product Characterisation

- **Particle size and morphology analysis -** Microscopy, fibre analysers, laser diffraction
- **Viscosity / rheology -** Over a range of shear conditions
- **Permeability and drainage**
- **In-application testing**
- **Mechanical properties** – “**FLT**” – (FiberLean Tensile) strength test (hereafter referred to as ***high loading tensile index***) - Good correlation with in-application mechanical properties.

Particle size alone is not sufficient to characterise MFC performance.

A test of performance is also required

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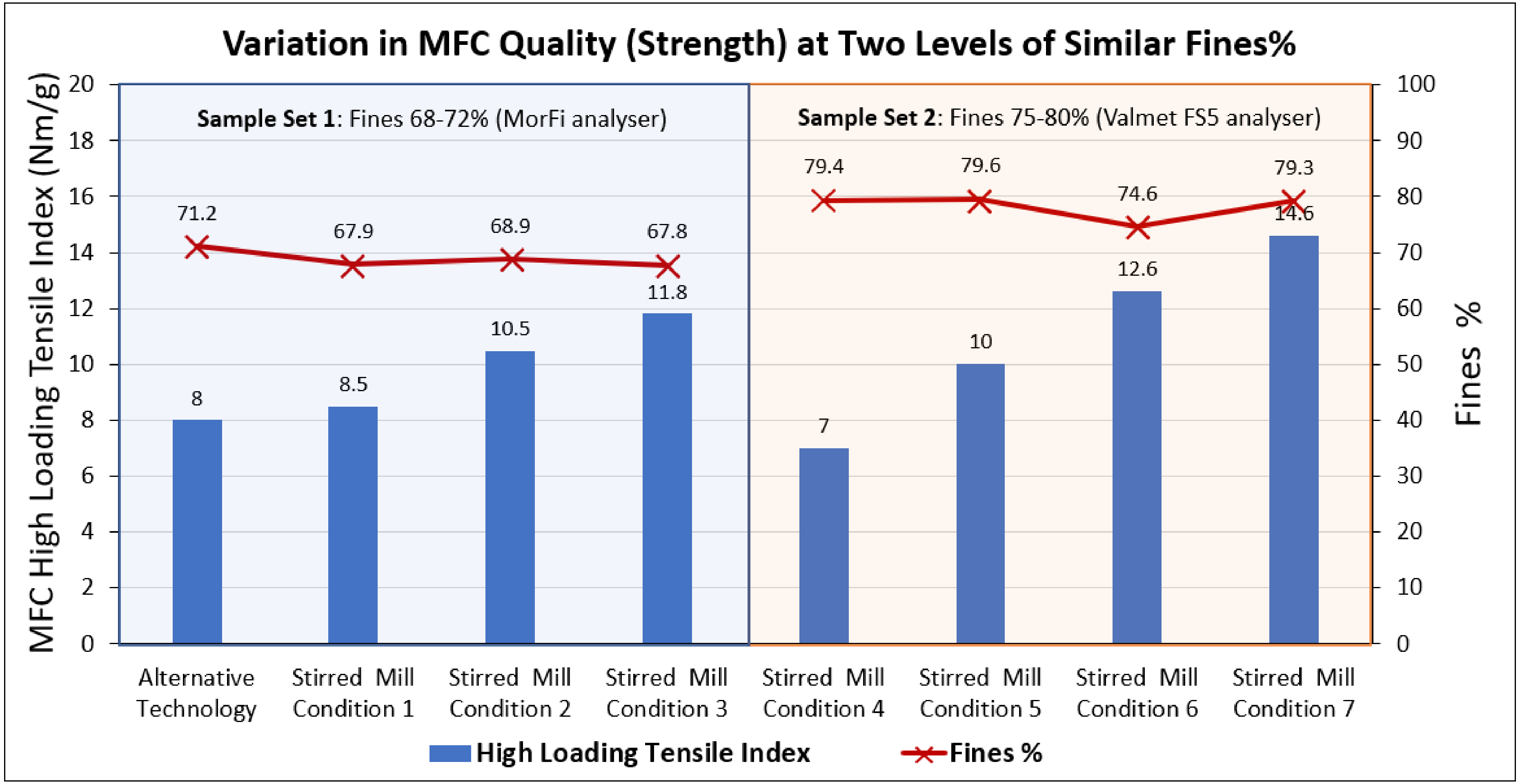
High Loading Tensile Index

- Since ***particle size alone*** (e.g. laser diffraction d_{50}, Fines%) ***says nothing about the extent of fibrillation or quality of fibrils***, we instead use such measurements largely to aid understanding of the process, and for process control and diagnostics.
- Many MFC applications rely on the ***bonding ability of the MFC***; measuring a proxy for this can be expected to correlate more generally with performance.
- The high loading tensile index test does this using a direct measurement of the ***tensile strength of an MFC - mineral film.***
  - A sheet of 100% MFC will be so heavily bonded that the sheet will largely fail by breakage of fibril cross-sections (i.e. zero-span strength) rather than bonding failure.
  - Therefore, the ***high loading tensile index test is performed at extreme mineral loadings*** (many times more mineral than fibre) to greatly weaken sheet bonding, thereby ***forcing bonding failure*** to be the dominant failure mechanism.
- Such a measurement gives a ***good general correlation with performance*** in many applications, that is largely ***robust to changes in pulp type and processing conditions***.

Fibre Breakage and Fibrillation (i)

- Using a stirred media mill, parameters can be changed to ***decouple fibre breakage from fibrillation***, and control them independently based upon application requirements.



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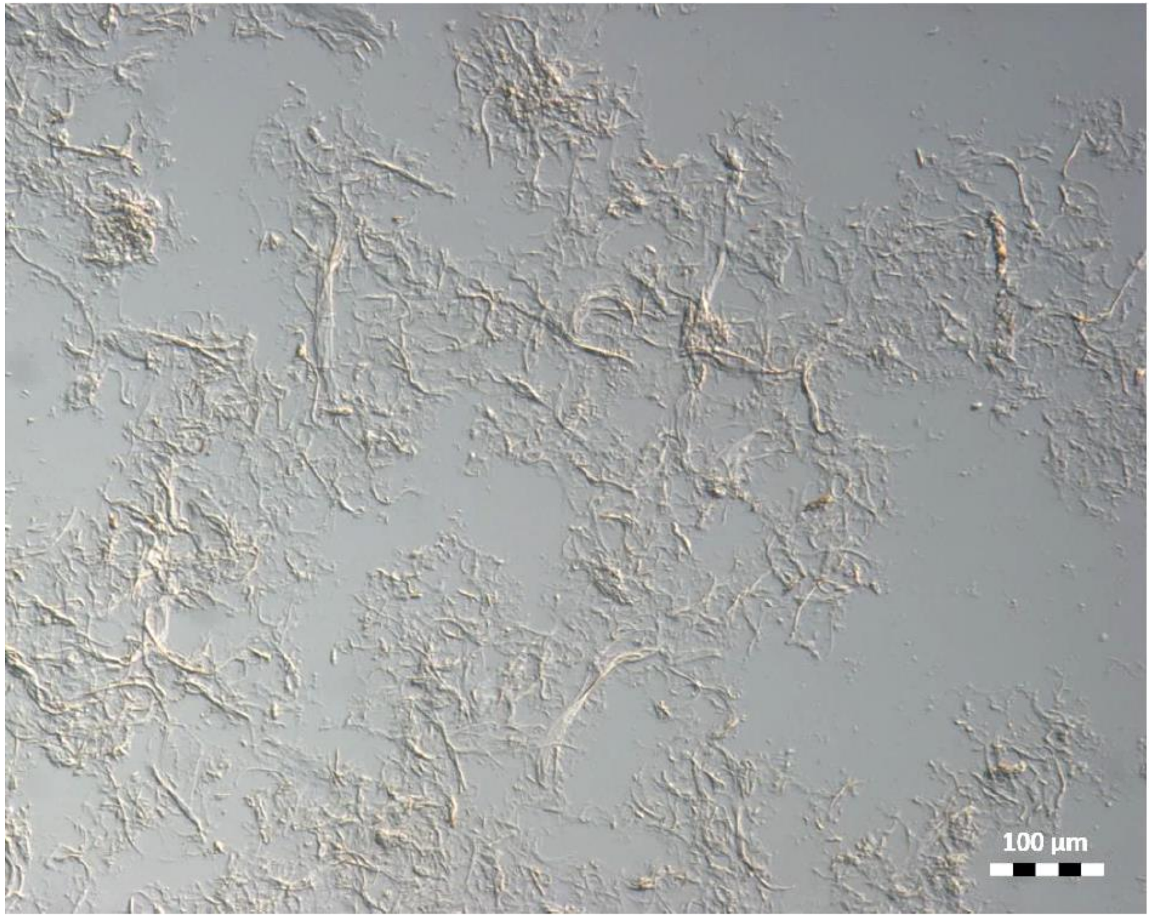
Fibre Breakage and Fibrillation (ii)

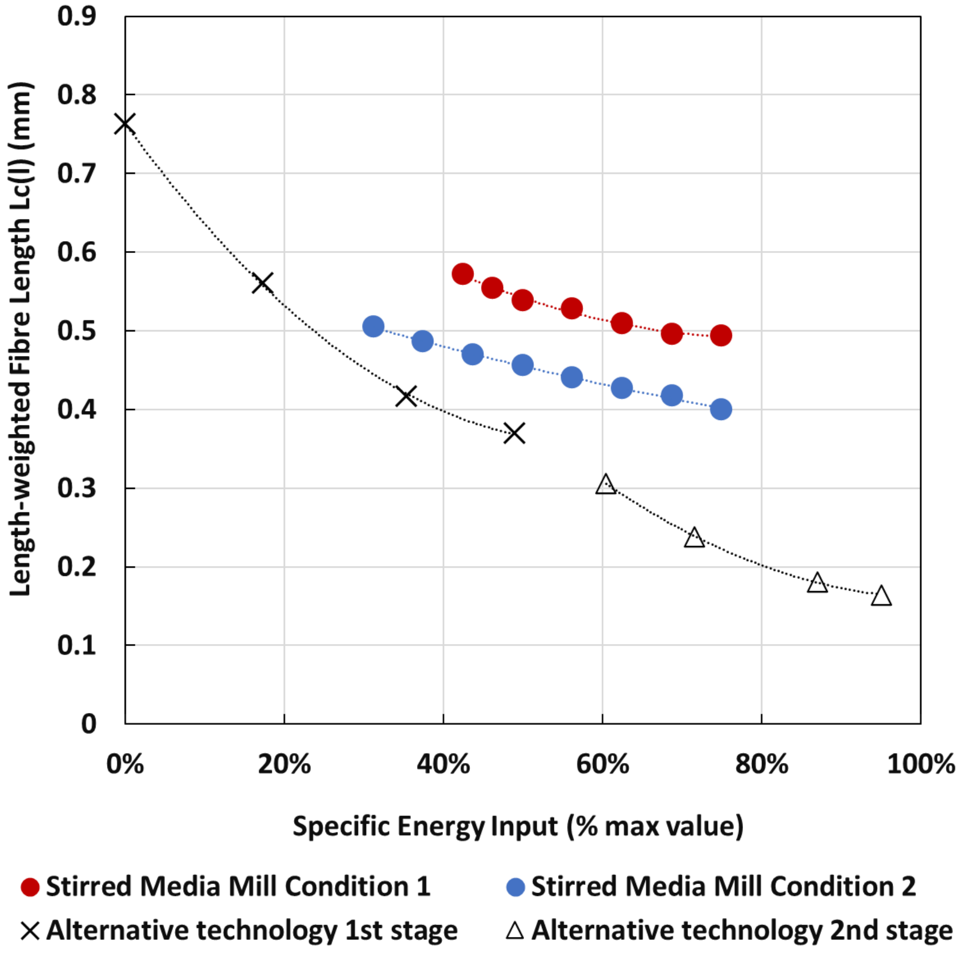
Very different product morphology possible with the same feedstock.

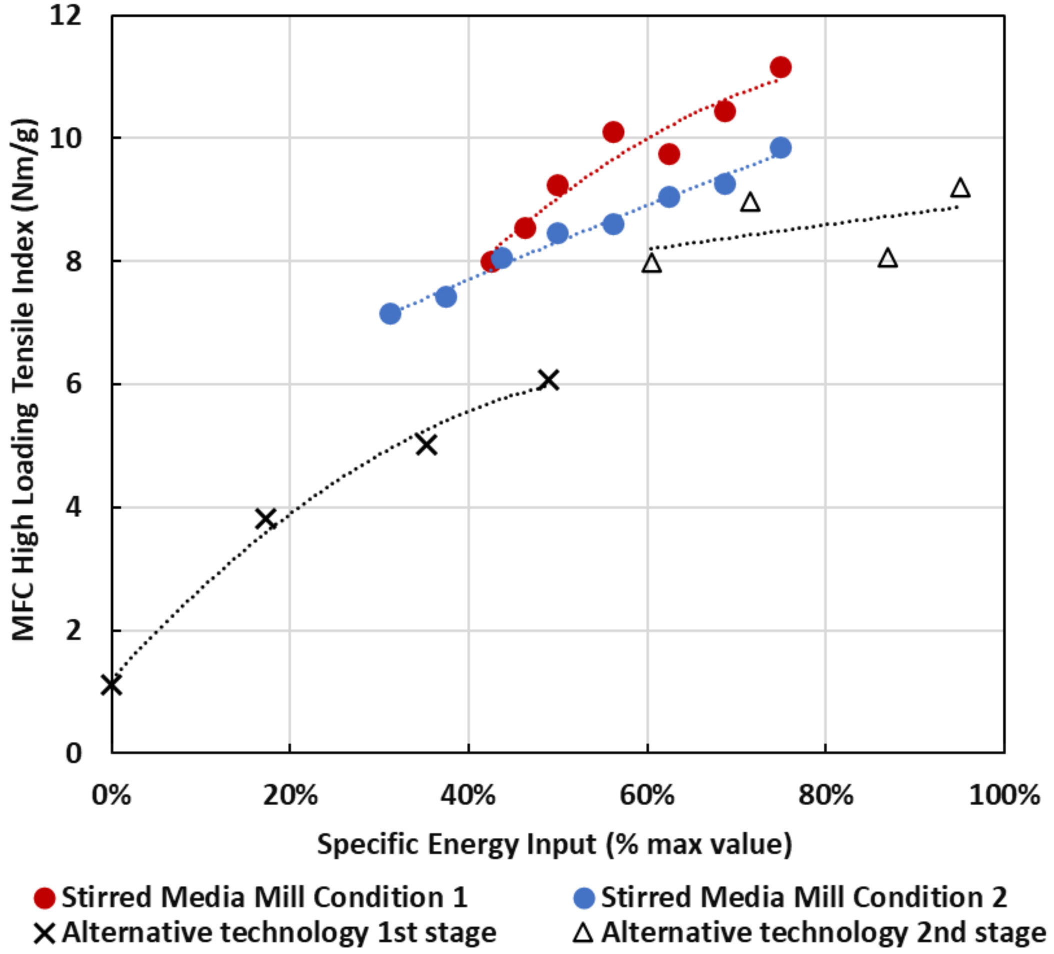
- Below have very different particle sizes, but similar high loading tensile index (bonding) values.



Performance / Energy Balance

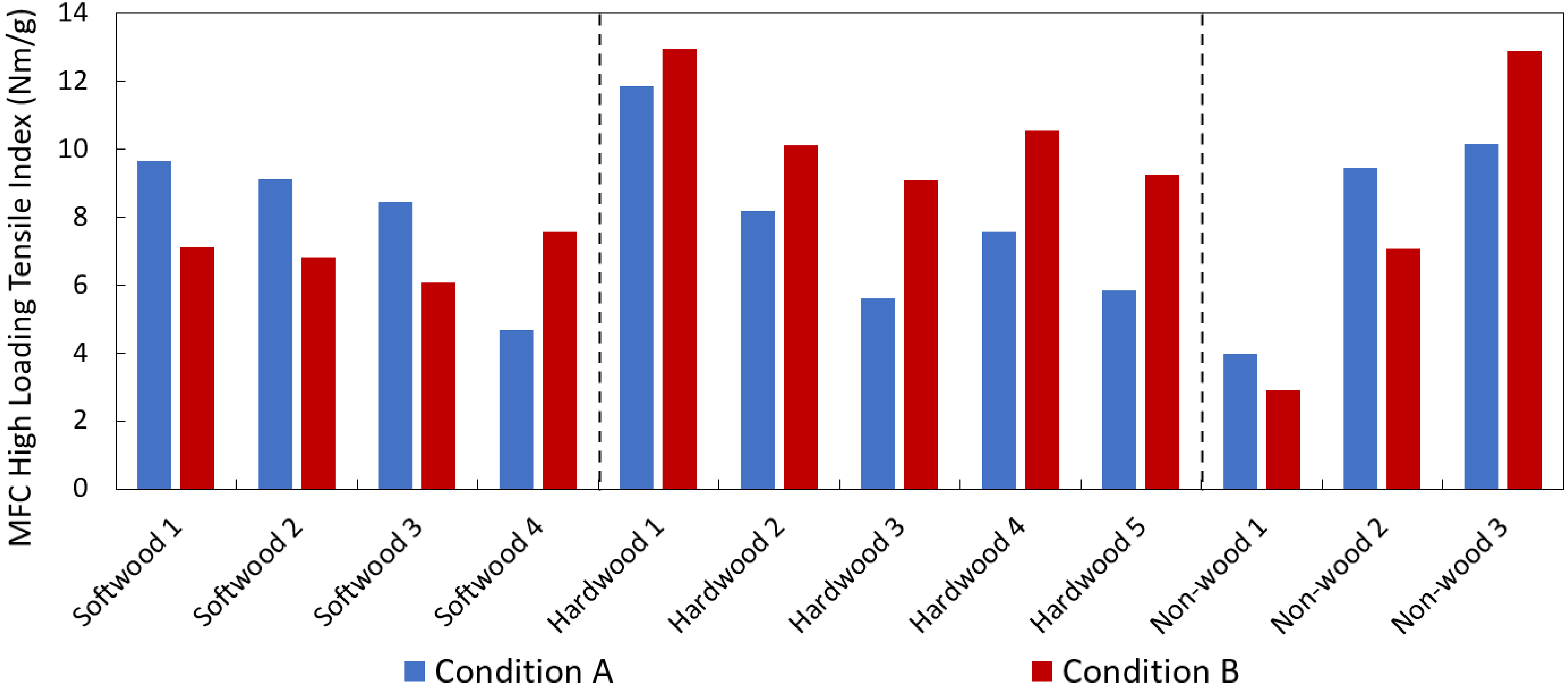
Stirred media mills are economical at generating a highly fibrillated product compared to alternative technology, though tends to maintain larger particle dimensions.





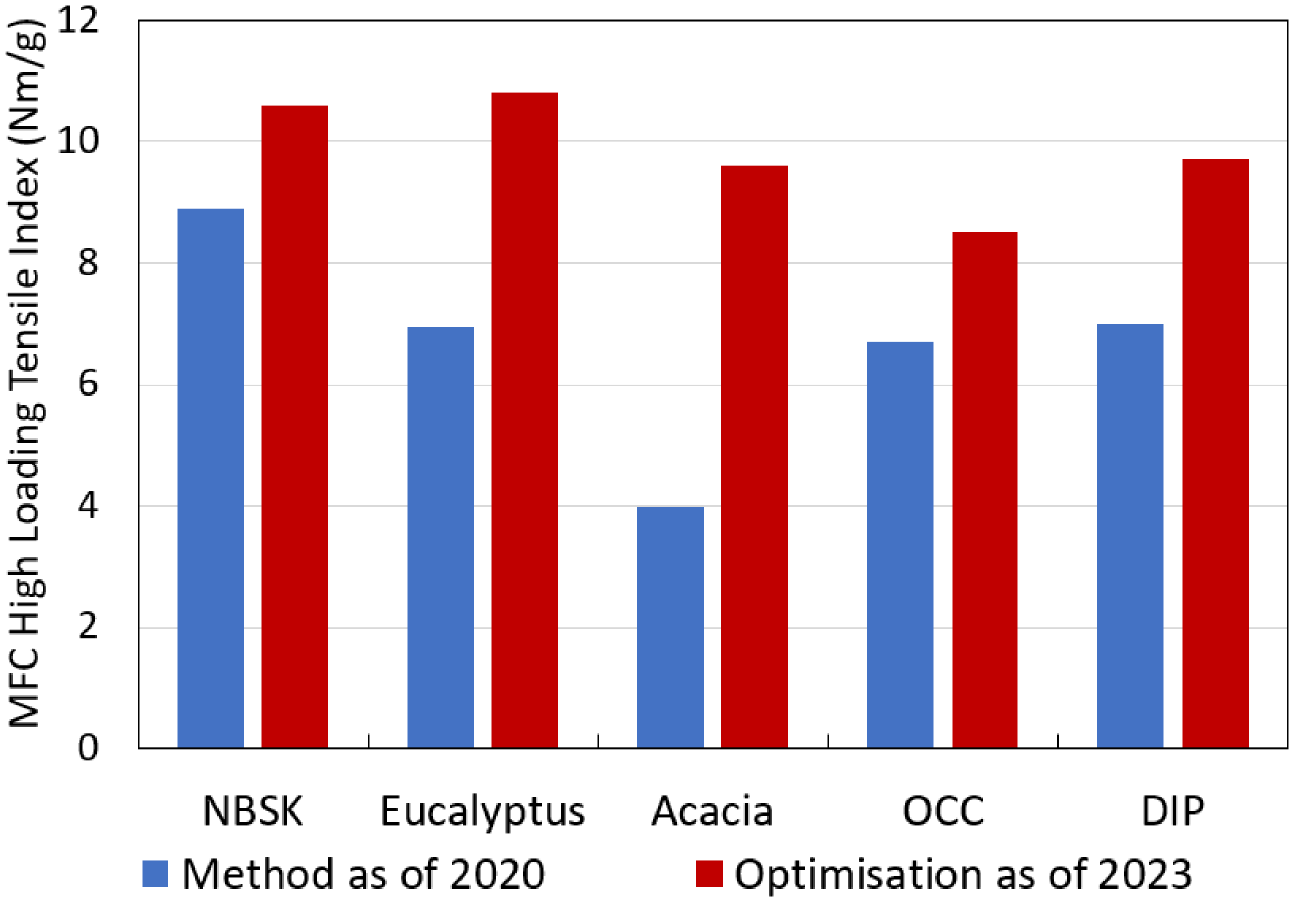
Optimisation for Fibre Substrate

Optimum conditions change based upon fibre type and properties



Improvements Since 2020

- Understanding how to adapt the process conditions based upon the properties of the feed fibres has:
  - ***Improved product quality*** at a given energy input.
  - ***Lowered the energy required*** to obtain a target quality.
  - Produced good quality MFC out of ***previously nonviable substrates.***
  - Enabled us to produce ***100% MFC products without requiring minerals*** as a co-grinding aid.



*MFC quality produced in **continuous mode** at **pilot scale** with various substrates. Specific energy input is maintained as constant.*

MFC Applications

The strengthening and viscosifying properties of MFC have shown benefits in applications such as:

- **Paper and board** – generally improved mechanical properties, increased filler, softwood replacement, lightweighting, new products and grade development.
- **White top liner:**
  – Improved optical properties from formation and filler increase, significant reduction in fibre use.
  – Wet-end coating of MFC to upgrade brown boxboard to WTL with minimal capex.
- **Barriers** – MFC forms a barrier layer which greatly improves oil and grease resistance and oxygen barrier properties for food packaging, is a recyclable and compostable alternative to PFAS.
- **Specialty papers** – various (e.g. low porosity improves coating holdout in thermal papers; significant increases in wet web strength enables low GSM papers on machines configured for much higher GSM).
- **Construction materials** – binders in furniture (MDF, particle boards, substitutes), ceiling tiles.
- **Rheological additives** – highly shear-thinning, robust to pH / salt / degradation.

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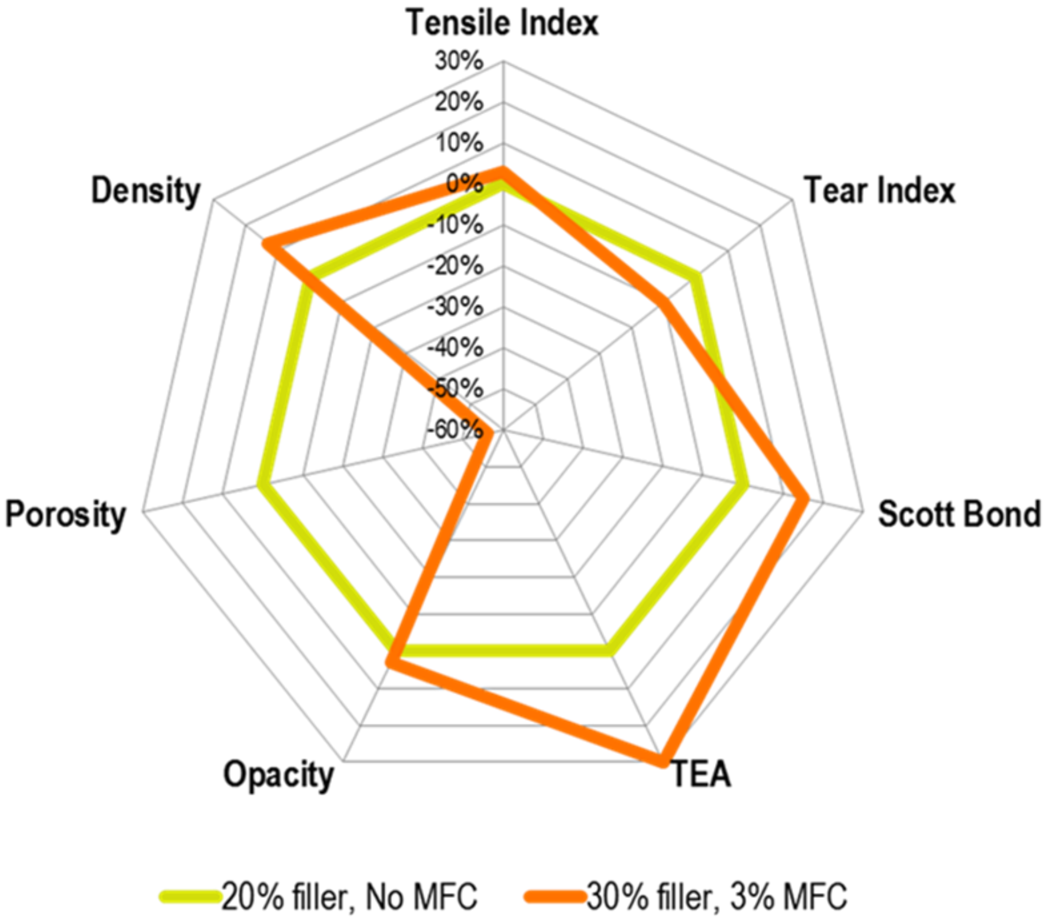
Use of MFC in paper and board

Typically, use of MFC in a web-based system is associated with:

- Improved performance stability
- Increased initial wet web strength
- Minimal impact on wet end chemistry
- Overall positive impact on drainage (when there is a filler increase)
- Improved dry mechanical properties
- Improved opacity
- A much tighter sheet (reduced porosity)
- Improved coating hold out
- Improved smoothness
- Maintaining bulk when fibre is replaced is a challenge but can be managed



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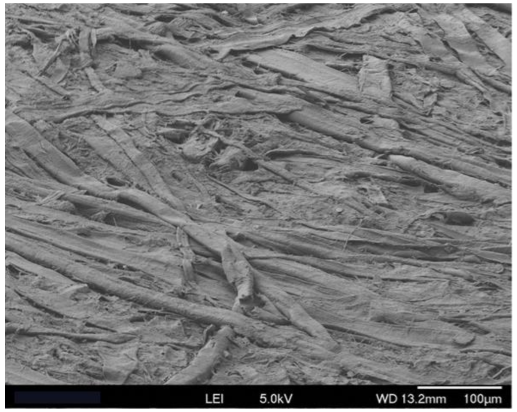
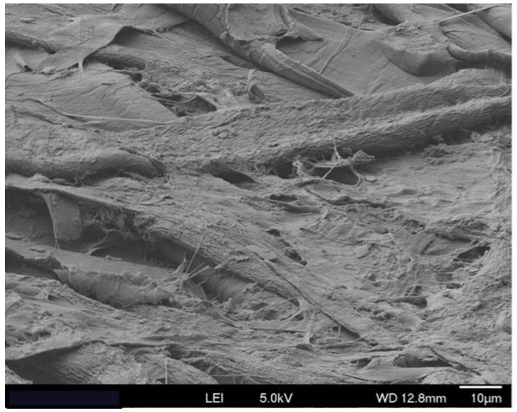
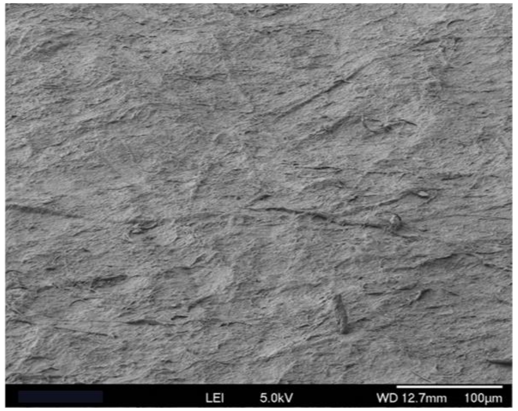
Barriers: Scanning Electron Microscope (SEM) Imaging of MFC coated papers

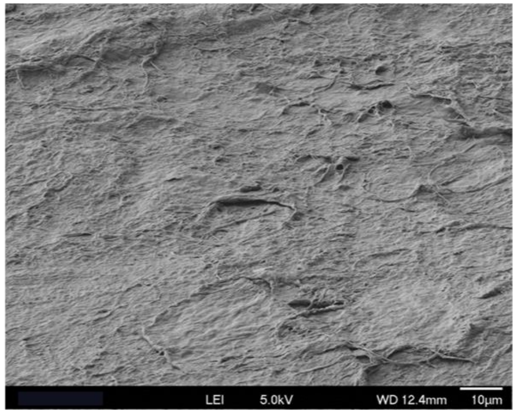
0 g/m² (No coating)



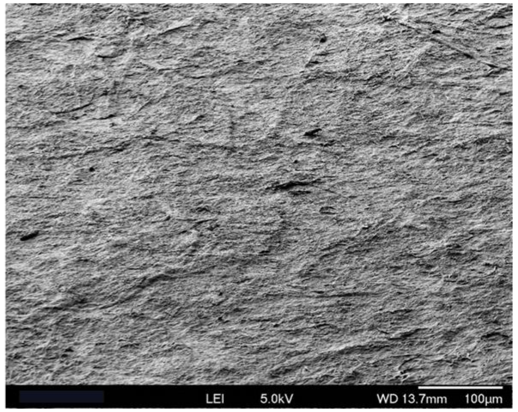


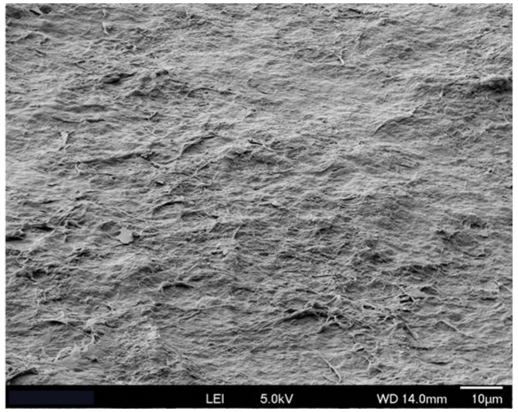
4 g/m² (MFC coating)





12 g/m² (MFC coating)





- The lowest coat weight, 4 g/m² provided substantial changes to the surface topography and structure.
- By 12 g/m², the MFC has formed a film and reached sufficient thickness to achieve high barrier properties.

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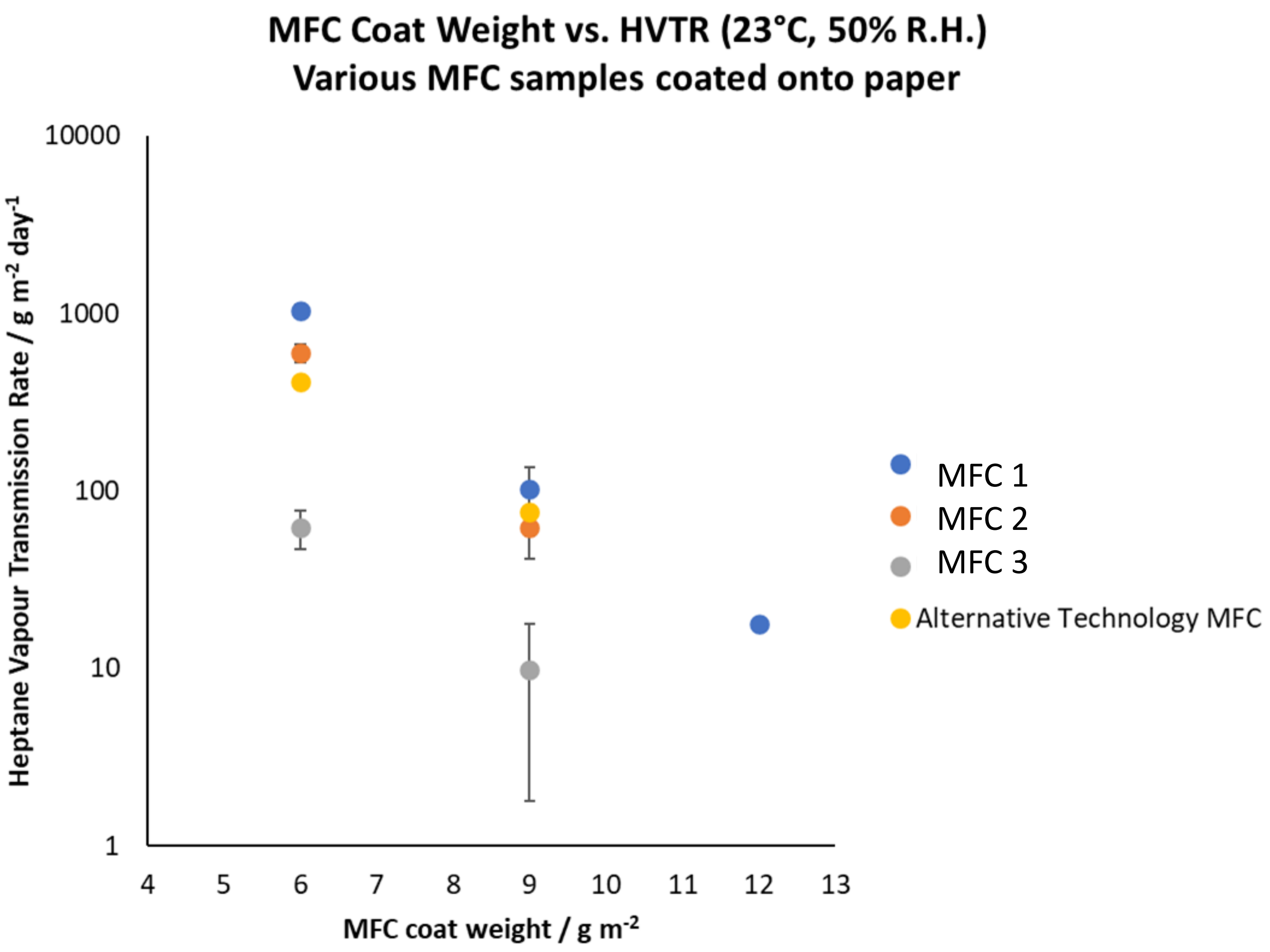
Barriers: Heptane vapour transmission



Optimised MFC samples offer improved barrier properties

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Regulatory clearances are essential for many applications

Current status

USA

EPA – existing substance under TSCA. Not subject to reporting under EPA nano rule

Food contact clearance through FDA (5wt.% fibrils in packaging), FCNs 1582 and 1887

Covers all ratios of mineral: MFC including mineral-free

Food coating FCN 2022

FDA GRAS – in progress, part of Vireo led consortium. For food use

Canada

Environment and climate change Canada – existing substance under CEPA

Health Canada opinion – “…we see no reason to object…to the use of FiberLean in food contact packaging, under conditions as described on the FDA website in the FCN 1582”

Covers all ratios of mineral: MFC including mineral-free

China

The National Health Commission of the People’s Republic of China approved microfibrillated cellulose pulp (CAS 65996-61-4) as an additive in paper and paperboard used for contact with all types of food, subject to a maximum usage of 5% (based on the dry weight of fiber) and no specific migration level requirement

Covers all ratios of mineral: MFC including mineral-free

Germany

Acceptance confirmed for BfR XXXVI and XXXVI/2 at up to 5 wt.% fibrils when produced with minerals at between 50% and 83% mineral content

Mineral-free application has been filed with BfR

Netherlands

Cellulose microfibres produced with calcium carbonate, kaolin and/or other permitted mineral fillers are included in Chapter 2 (Paper and board) of the Dutch commodities act regulation at up to 5wt.% fibrils

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Environmental Impact

- PTS-RH 021:2012 Recyclability testing was carried out by PTS
- Two samples tested: MFC coated paper with bleached and unbleached base sheet

Sample description		Sample 1: Bleached	Sample 2: Unbleached
Disintegratability	Non-paper constituents	No information: Not quantified	No information: Not quantified
	Total reject	< 1%	< 1%
	Recyclable percentage	> 99%	> 99%
Sheet formation	Adhesive impurities	Absent	Absent
	Optical inhomogeneities	Absent	Absent
Overall rating: Recyclability		Recyclable	Recyclable

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Environmental Impact

- OECD 301B Biodegradability testing was carried out by RespirTek Inc

Sample description	Biodegradation (%)	Classification
MFC slurry - no biocide	75.4	Ready biodegradability
MFC mineral composite - no biocide	70.4	Ready biodegradability
MFC mineral composite - with biocide	76.6	Ready biodegradability

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Environmental Impact

- ISO 20200 (precursor to EN13432) packaging compostability standard testing was carried out by Impact Solutions
- Two samples tested: MFC coated paper with bleached and unbleached base sheet
- Still await compostability data but high level of disintegration is observed.

Material	R factor	Material recovered from sieving (g)	Degree of disintegration (%)
White paper	57.5	0.36	94
White paper	57.9	0.48	92
White paper	55.8	0.05	99
Brown paper	58.1	0.11	98
Brown paper	57.0	0.79	86
Brown paper	57.1	0.14	98

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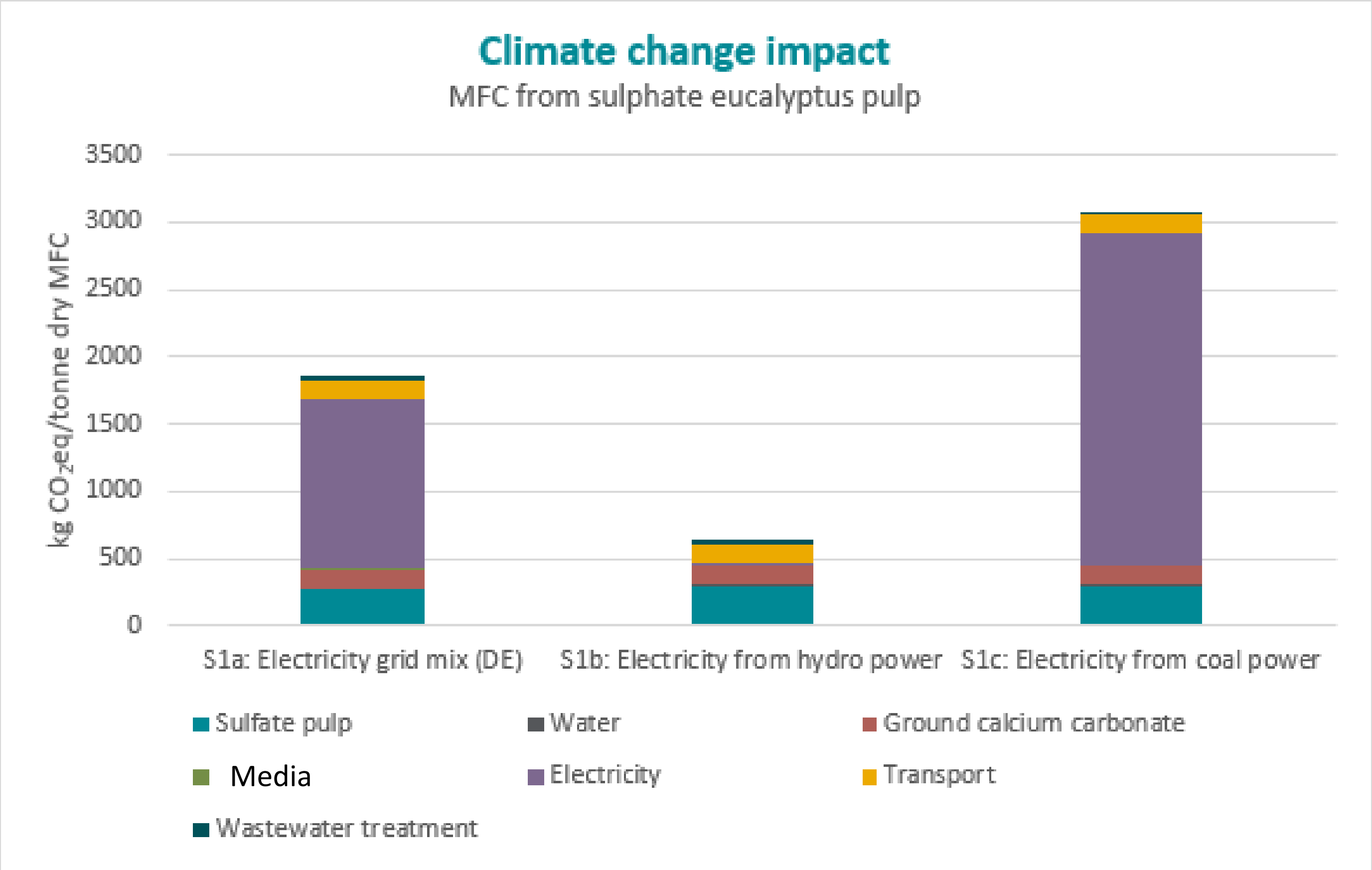
Environmental Impact

- Life Cycle Analysis (LCA)
  - Cradle to grave LCA for the use of FiberLean MFC in paper applications with different pulp sources and plant locations is in progress with IVL
  - Cradle to gate analysis illustrates the importance of electricity consumption and sourcing
  - Used to focus Process Research work on energy reduction



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Conclusions

- ***MFC and mineral / MFC composites*** are produced from ***virgin and recycled pulps***, and are important additives for a wide range of paper, board, and other applications.
- Stirred media mills efficiently and continuously produce MFC at large scale.
- Their nature allows for effective ***decoupling of fibre breakage and fibrillation.***
- Stirred media mills are ***highly tuneable***, giving flexibility for a ***wide range of product characteristics*** depending on application need.
- Although ***conceptually simple***, they are ***complex to optimise***.
- ***Efficient optimisation requires an intimate understanding of the feedstock and process physics.***
- ***Several key fibre characteristics influence optimum operating conditions***, and adapting the process accordingly has yielded substantial efficiency and quality benefits.
- There are a wide range of applications for MFC in paper and board, building material and other applications.
- A range of regualtory clearances are in place for MFC and the products are both biodegradable and recyclable

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Thank you for your attention

Any Questions / Comments?

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