

Producing Microfibrillated Cellulose using a Stirred Media Mill Grinder

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Outline

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

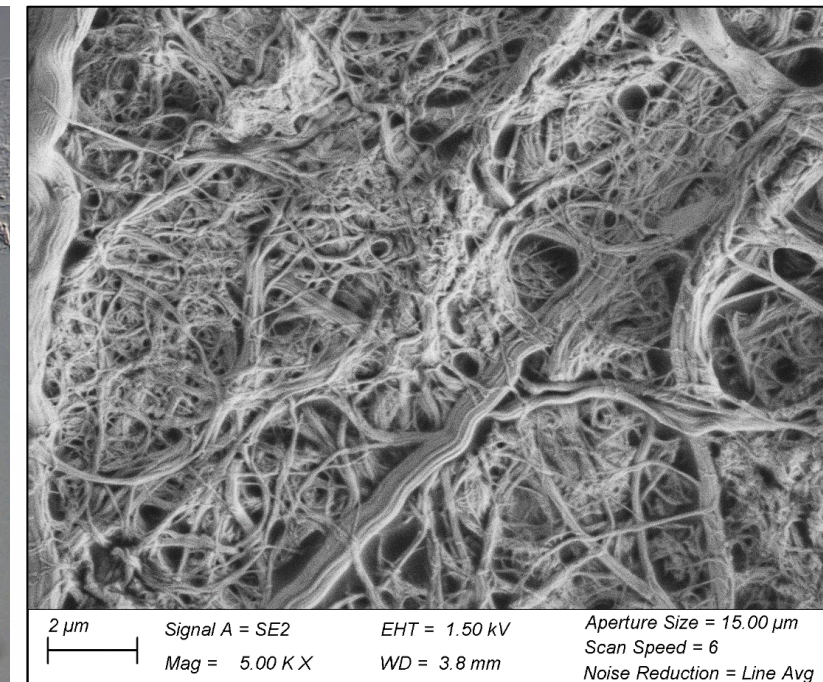
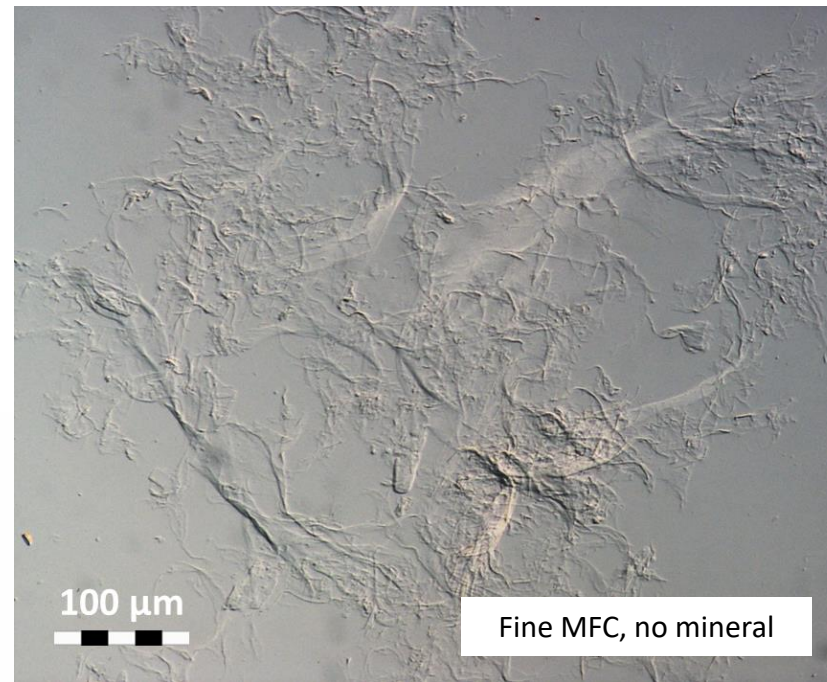
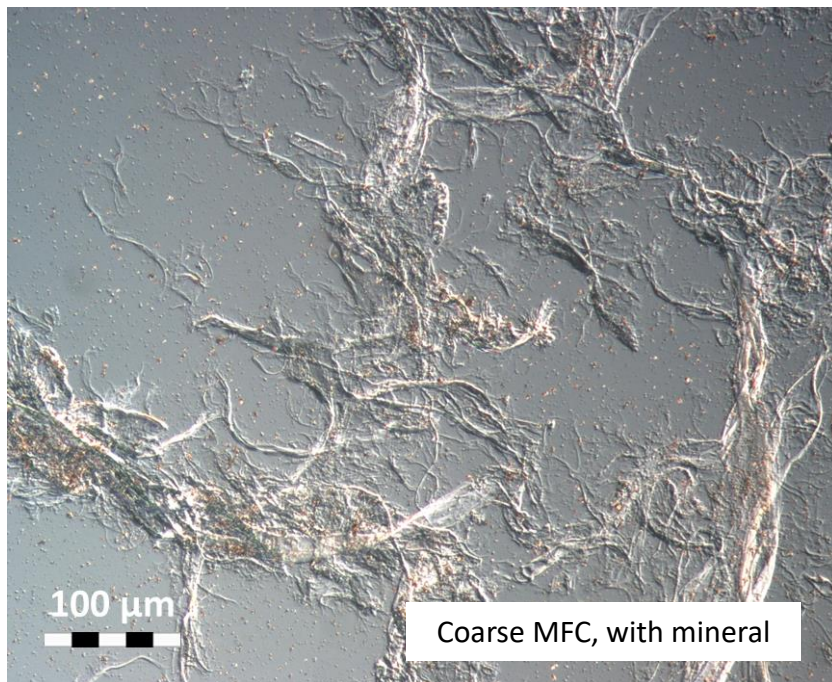


Introduction

- **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

Slurry



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

Press cake



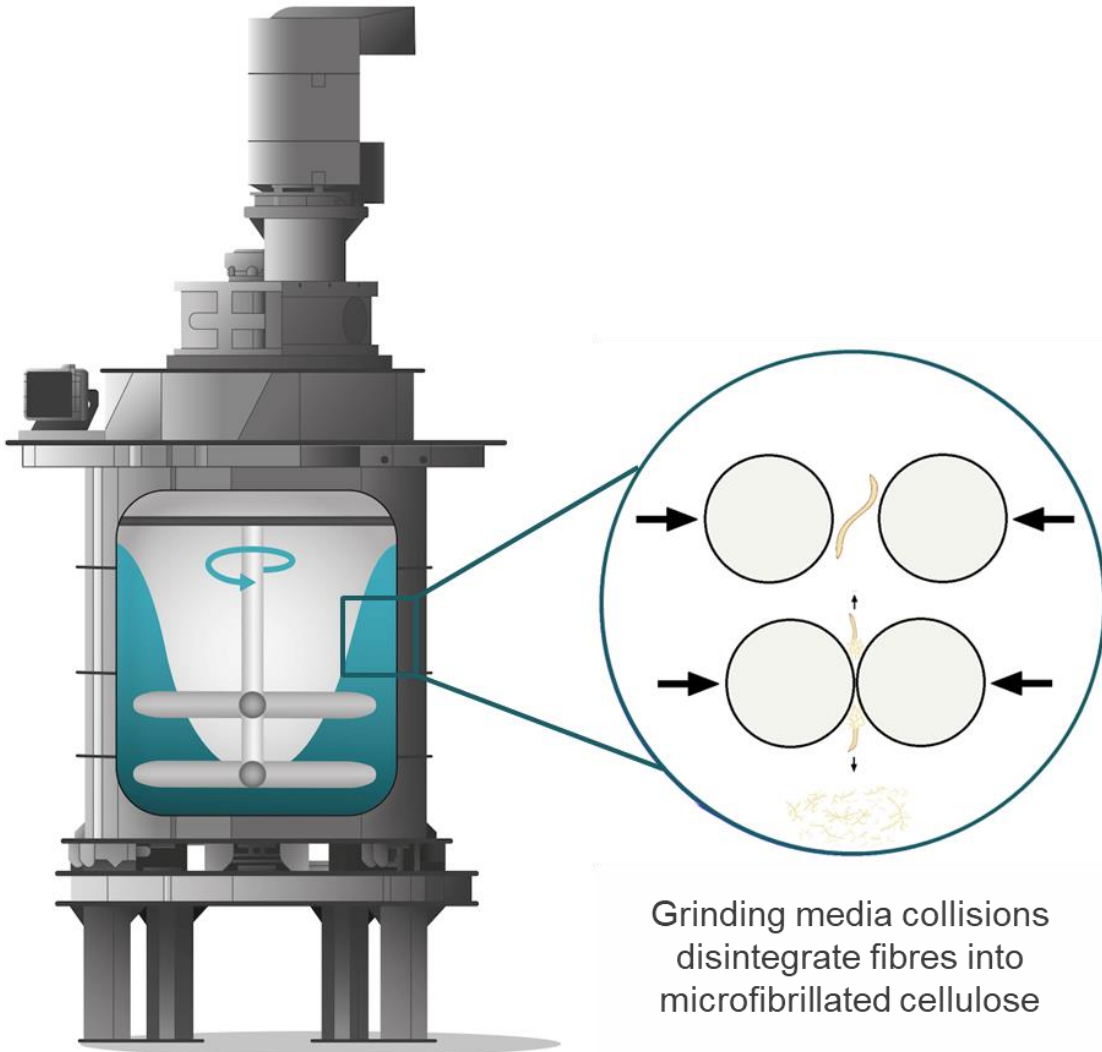
MFC Applications

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.
- **Agriculture** – in fruit coatings to delay ripening and increase shelf life / reduce food waste.
- **Rheological additives** – highly shear-thinning, robust to pH / salt / degradation.

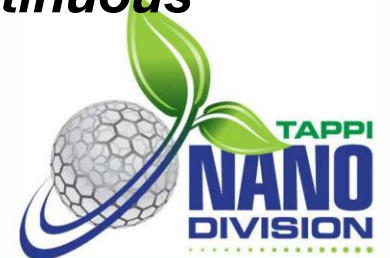


Stirred Media Mills – Introduction



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.



Stirred Media Mills – Advantages

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

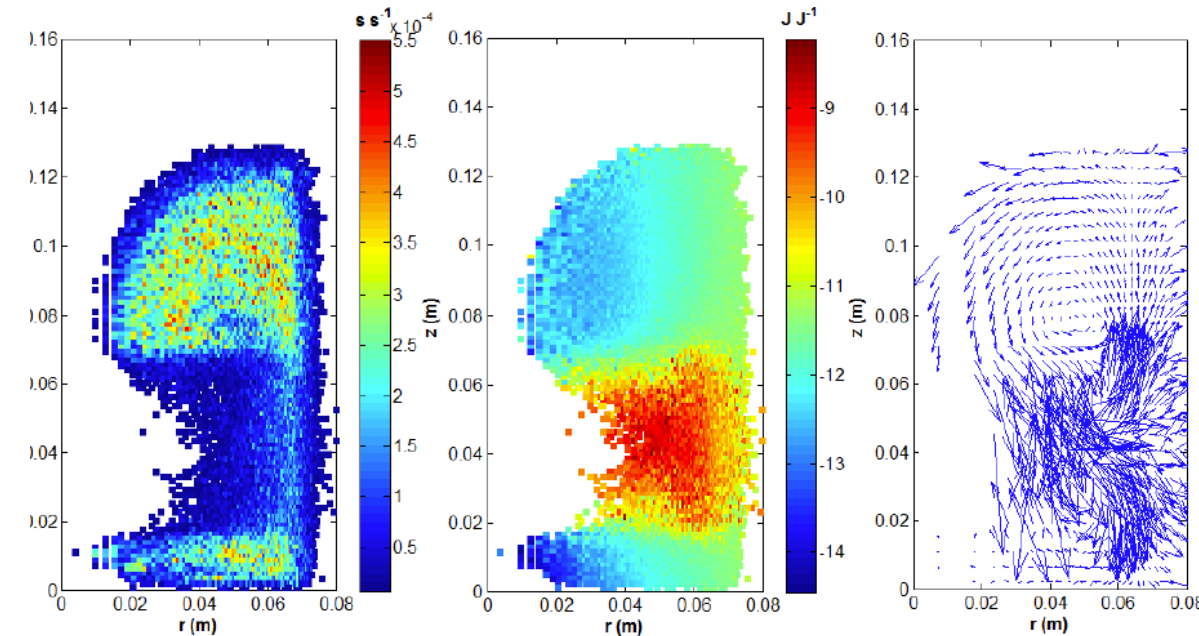
Video of
Grinder

- 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



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

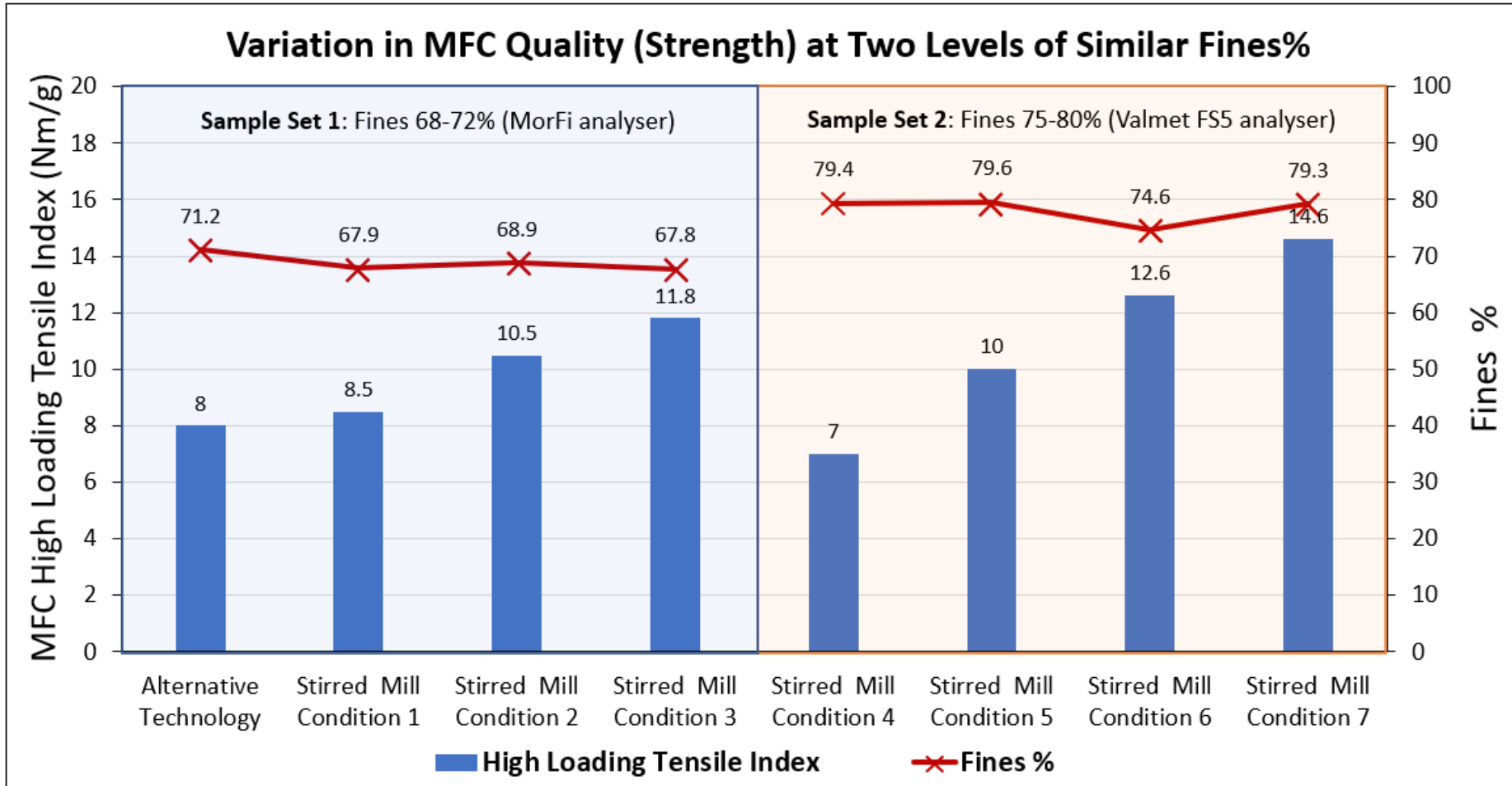


High Loading Tensile Index

- 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)

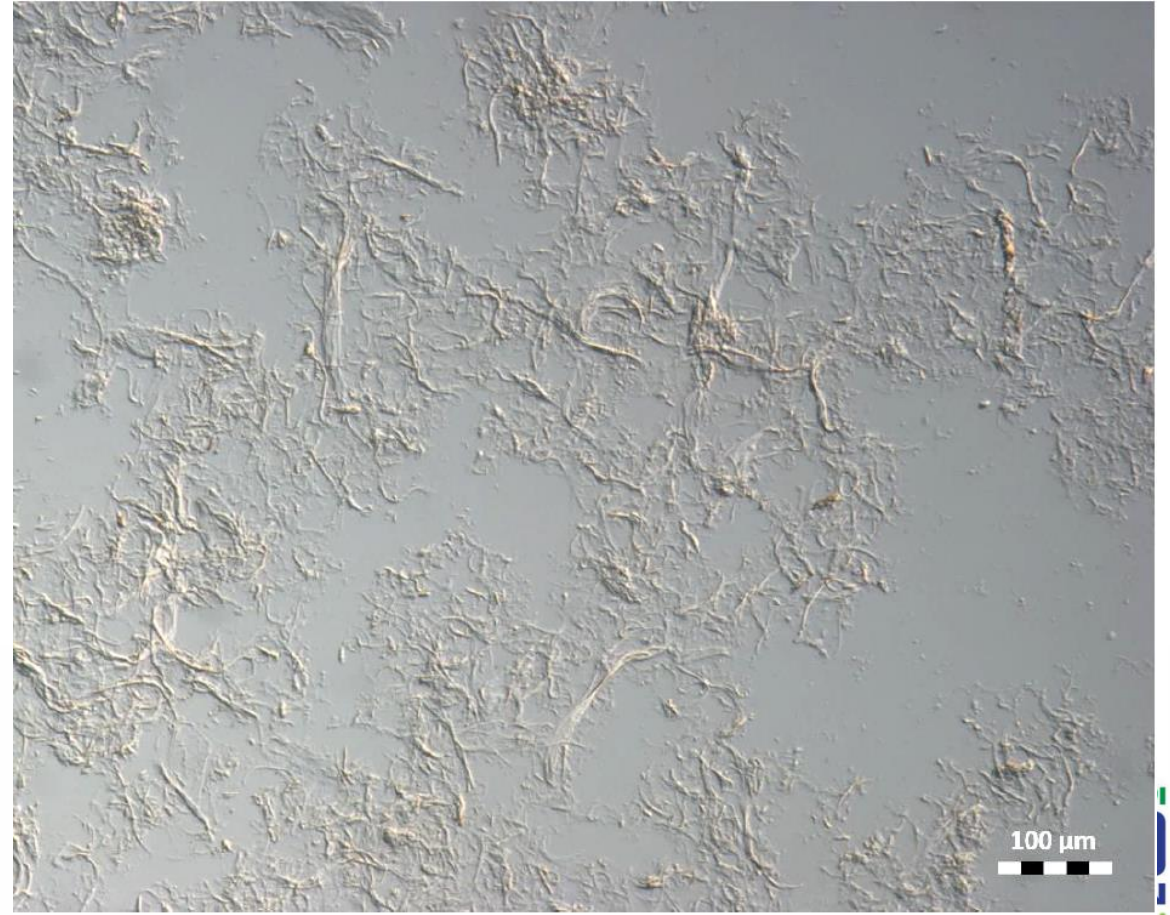
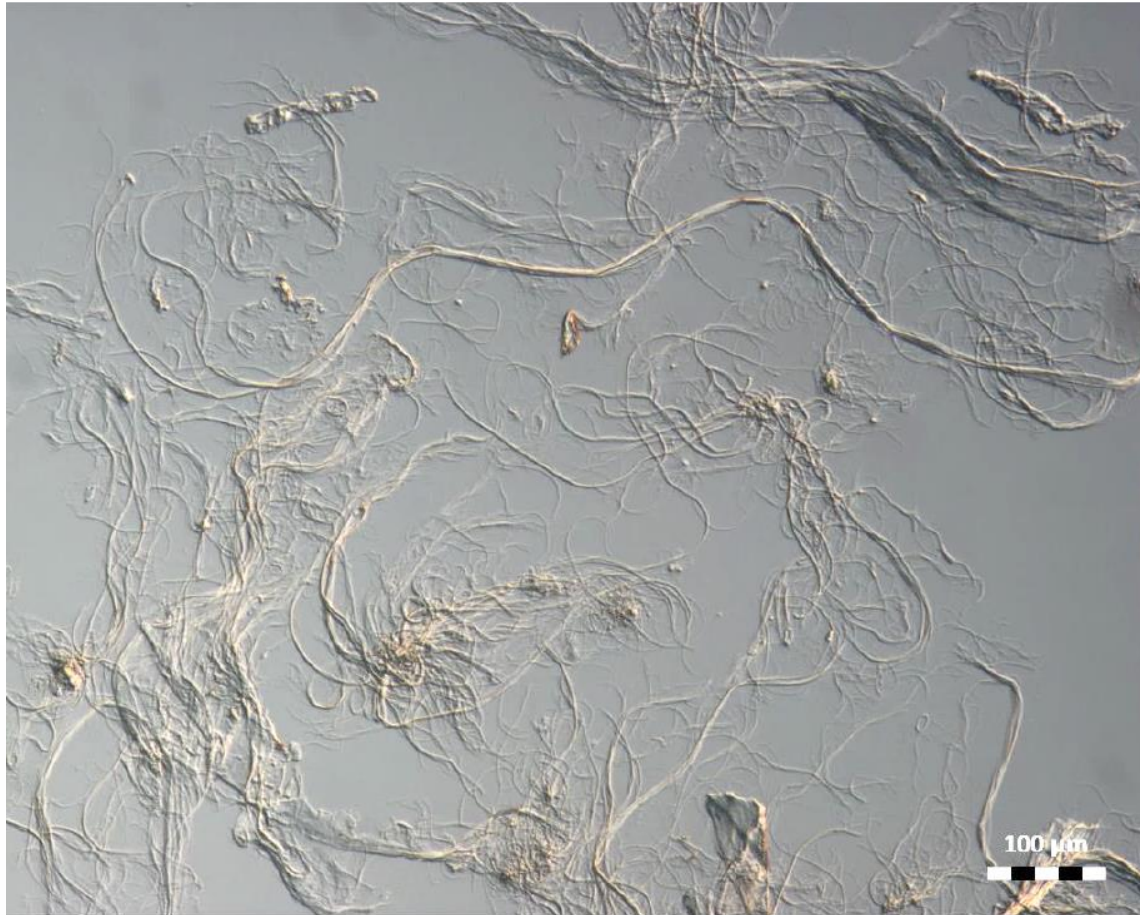
- Using a stirred media mill, parameters can be changed to **decouple fibre breakage from fibrillation**, and control them independently based upon application requirements.



Fibre Breakage and Fibrillation (ii)

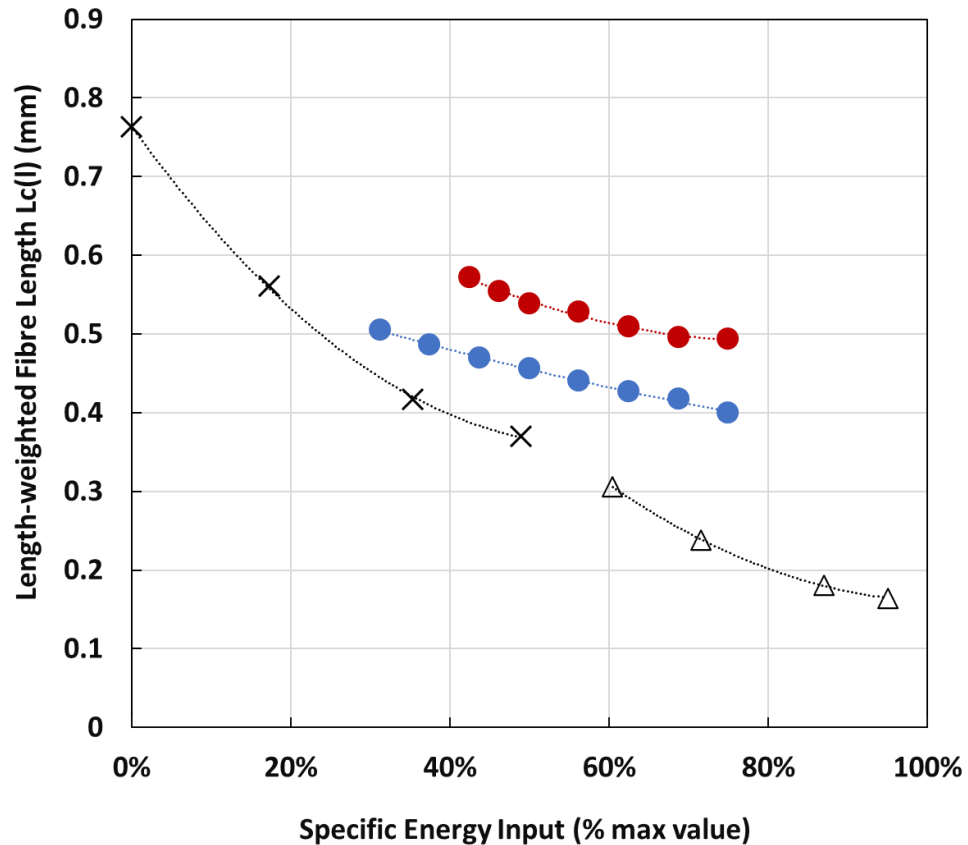
Very different product morphology possible with the same feedstock.

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

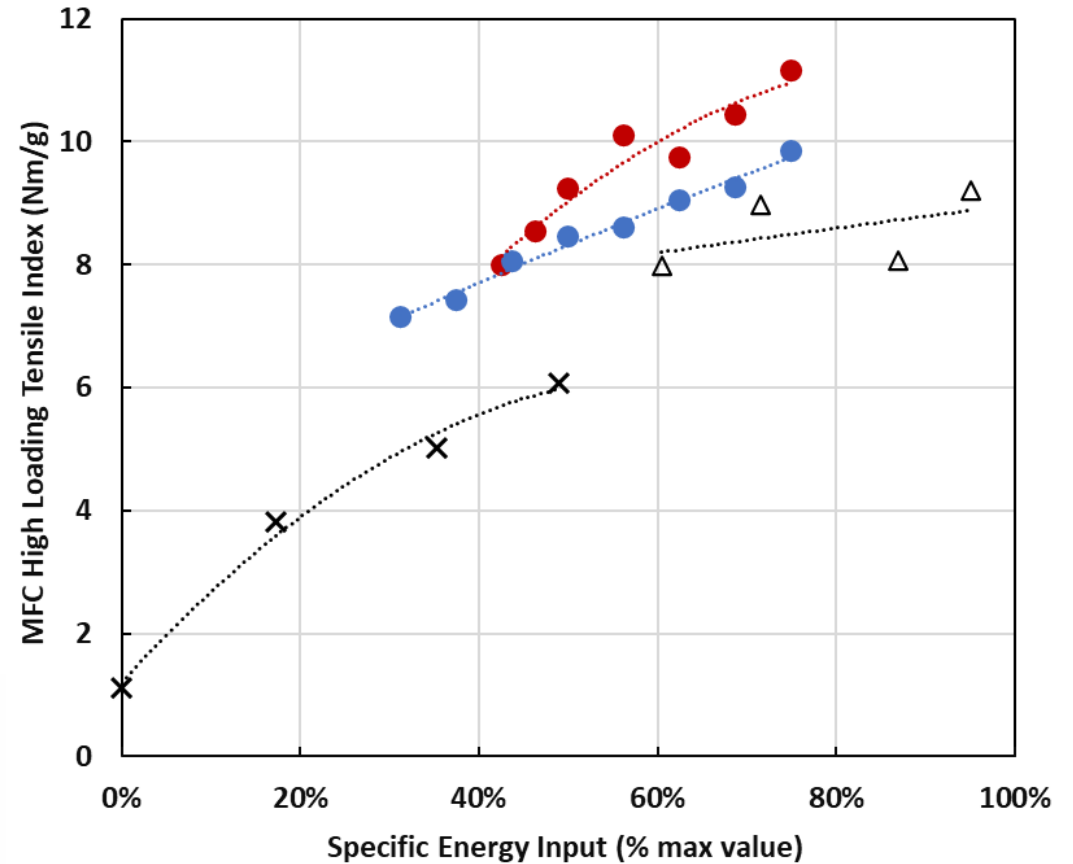


Performance / Energy Balance

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



● Stirred Media Mill Condition 1 ● Stirred Media Mill Condition 2
× Alternative technology 1st stage △ Alternative technology 2nd stage

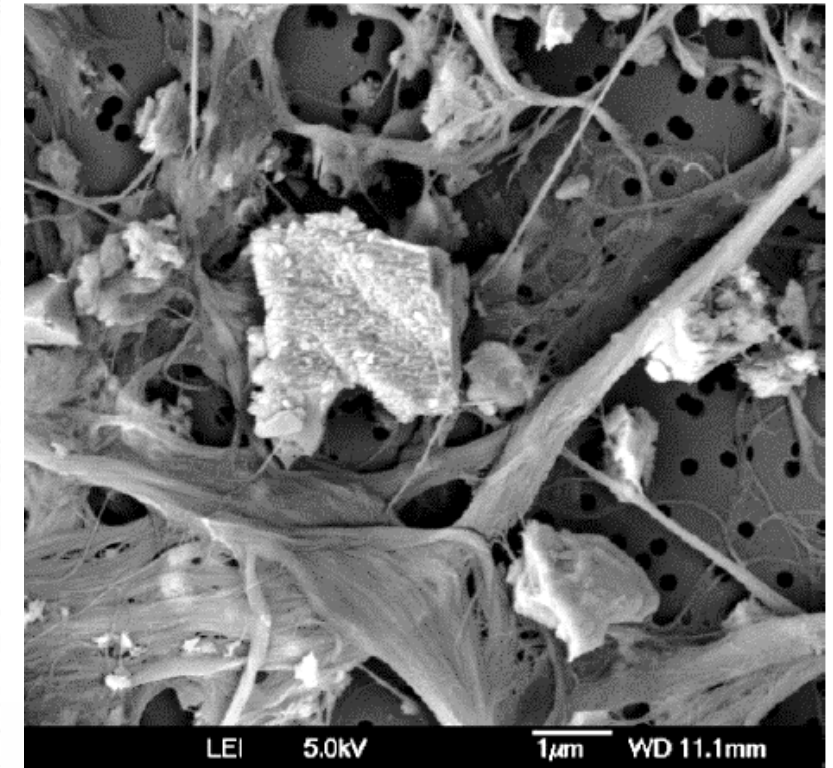
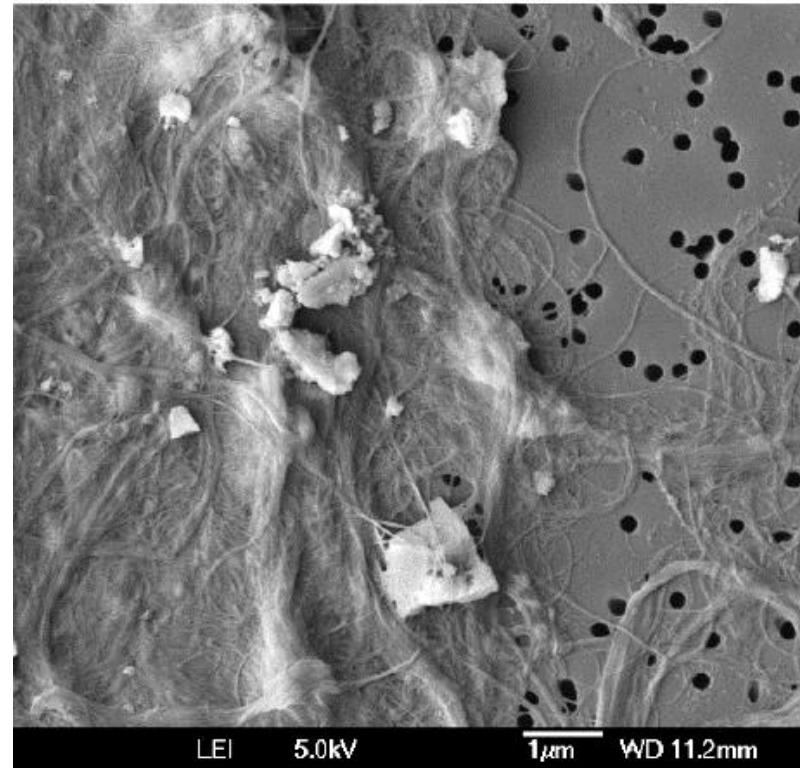


● Stirred Media Mill Condition 1 ● Stirred Media Mill Condition 2
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Optimisation for Fibre Substrate (i)

Fibre Hemicellulose Content

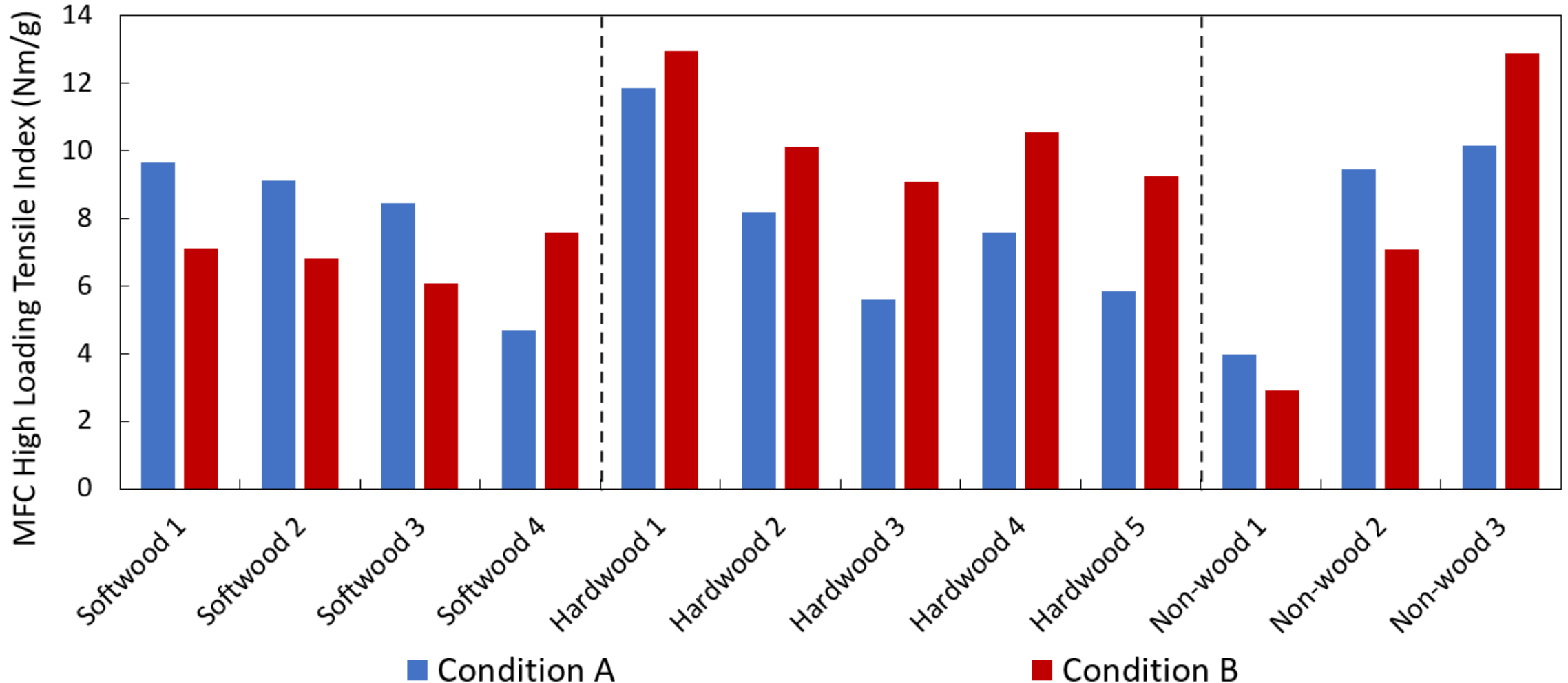
- Hemicellulose is a polysaccharide that forms a weak, amorphous layer **spacing apart microfibrils**.
- All else being equal, a fibre with a **high amount of hemicellulose** more readily **generates finer microfibrils**, improving bonding ability / high loading tensile index.
- Such fibre chemistry advises on suitable grinder operating conditions.



MFC – mineral composite produced from fibres with high hemicellulose content (left) and low hemicellulose content (right)

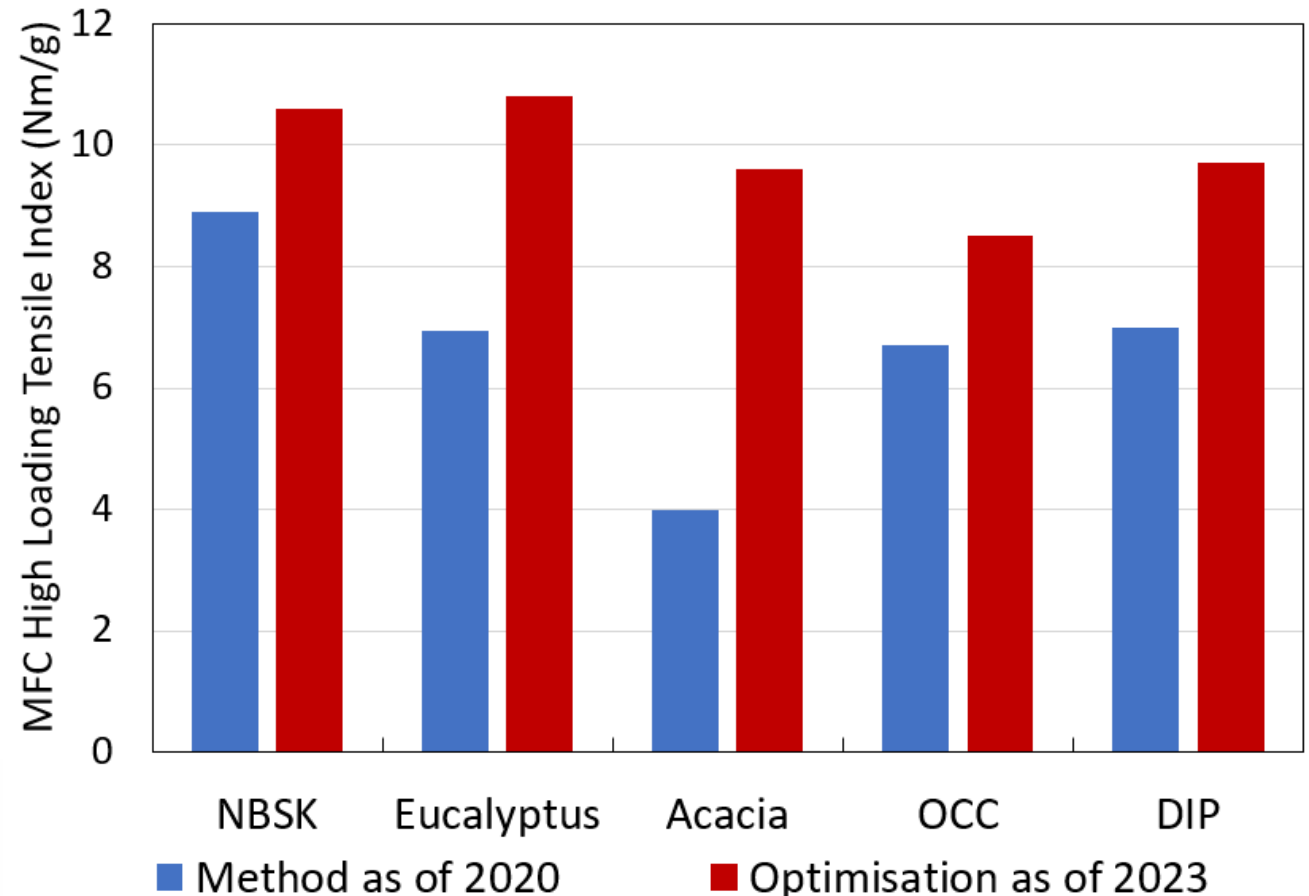
Optimisation for Fibre Substrate (ii)

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.

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.



Thanks for your attention

Any Questions / Comments?

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