International Conference on Nanotechnology for Renewable Materials

Microfibrillated Cellulose Products: Environmental and Regulatory Aspects

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TAPPI NANO DIVISION

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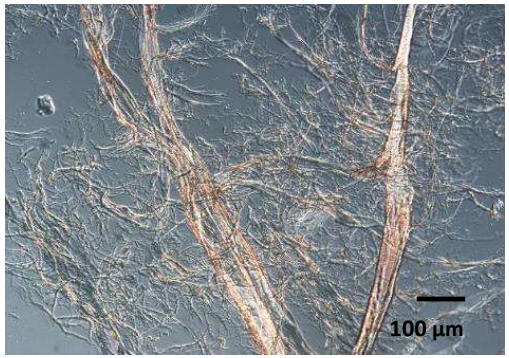
Introduction

- Microfibrillated Cellulose (MFC) – produced by mechanical treatment of cellulose
- Highly viscous suspension in water
- Typically 1-2% solids content
- Satellite production adjacent to final use location
- Produced using stirred media mills

Product families

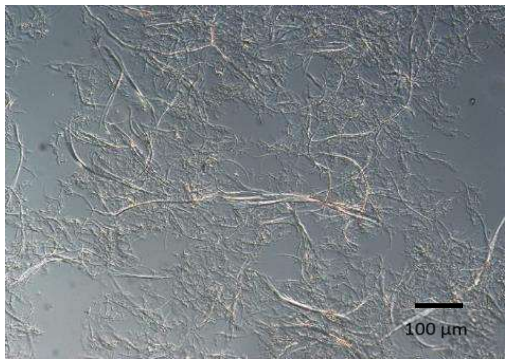
- MFC from 100% virgin pulp
- MFC from recycled fibres
- MFC mineral composites

- NB Two of these families have no added minerals. MFC only



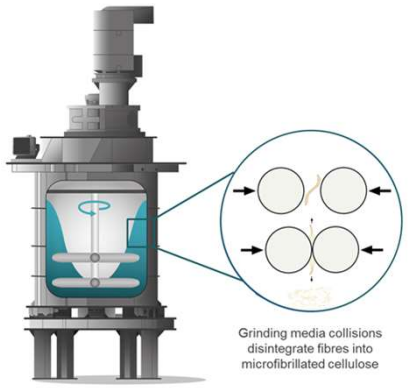
Coarse MFC

Medium MFC



Fine MFC

Stirred Media Mills



Stirred vessel containing small grinding media beads, which are agitated by an impeller

- Grinding media collide with each other, breaking and fibrillating fibres that are caught in the interstices
- High media surface area enhances fibrillation
- Highly tuneable

Advantages

- No close tolerances or precision engineered components
- Robust proven technology
- Continuous single stage process
- Availability > 95%
- Low Capex and Opex
- High throughput
- Small footprint
- Modular easily-scalable design
- No additives or pre-treatments

Applications

Microfibrillated Cellulose (MFC)

- Increased bonding in fibre-based structures
- Viscosification, highly shear thinning
- Reinforcement (of green polymers)
- Formation of barrier layers

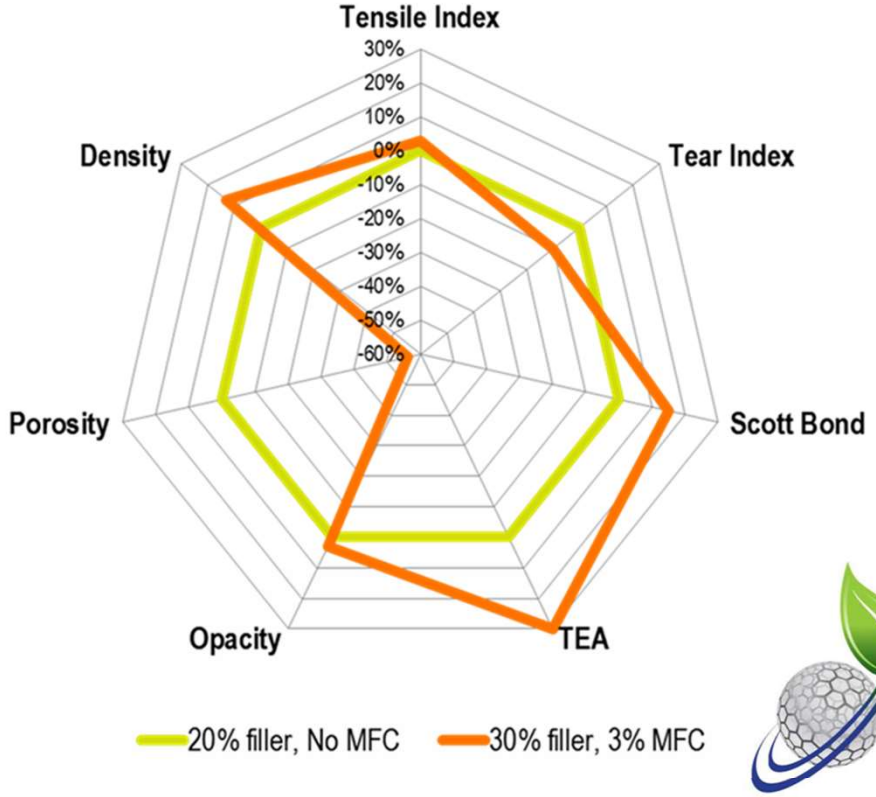
Many applications

- Printing and writing paper
- Packaging
- Paints and coatings
- Adhesives
- Food
- Construction materials
- Nonwovens
- Medical materials

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



Regulatory considerations

- Moral/ ethical responsibility to produce safe materials and comply with legislation
- Legislative requirements vary around the world
- Typically aspects include:
  - Manufacturing
  - Food contact packaging
  - Food use
  - Environmental impact
- Extensive technical programme around workplace/ environmental exposure, physical chemistry, toxicity, migration behaviour from packaging etc is required
  - Mostly out-sourced from contract laboratories (Fraunhofer etc)
- Don't underestimate the work/ cost
- Start as soon as the product is defined

Initial analysis

- Third party regulatory law firm opinion:
  - Starting materials are either cellulose or cellulose and mineral
  - Starting materials are REACH exempt and have GRAS status
  - Only aspect that needs attention is that the raw materials have been processed and the particle size distribution has got finer
  - Need to consider:
    - Manufacturing - Work place exposure and discharge to the environment
    - Whether chemistry has changed
    - Food contact packaging status
    - Food status

Manufacturing, work place exposure and discharge considerations

Chemistry

- Extensive testing required by various regulatory agencies
- No changes detected compared to feed pulp other than size

Fibre dimensions (Fiber Analyser)
Particle size (light scattering)
SEM Fibril width

Molecular mass/ range
Morphology
Aggregation
Chemical composition
Thermal decomposition profile
Solubility

Shape
Surface charge
Viscosity
pH
Hydrolysis
Zeta potential
.....

Dusting testing at MFC manufacturing, papermaking and paper shredding locations

- Utilized PM10 nanoparticle detecting technology
- Testing at UK MFC manufacturing facility, US pilot and full scale paper machines and paper shredding simulation
- No nano dust beyond background detected

Discharge

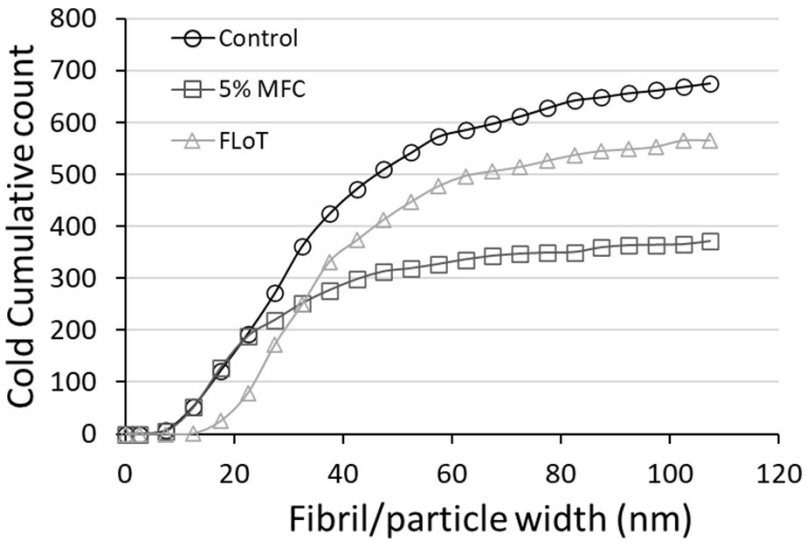
- 10 kg cellulosic residue/ tonne MFC
- Either processed through mill effluent system or used for soil remediation

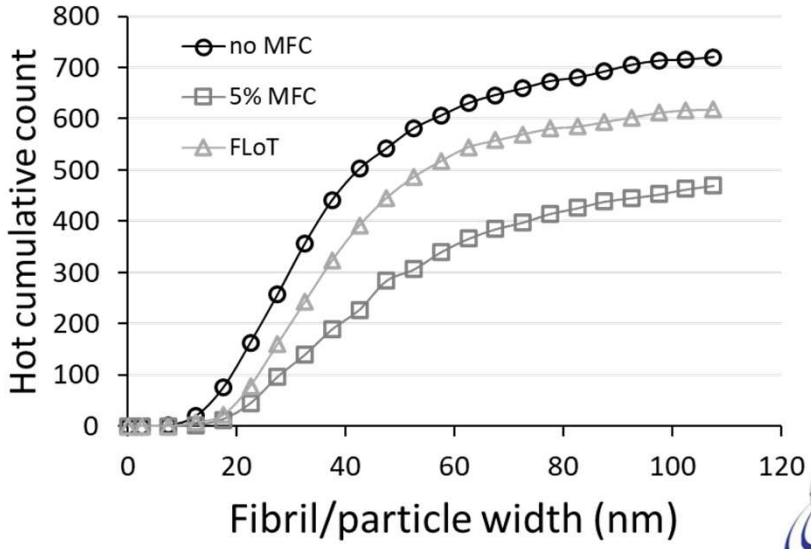
Food contact clearances

- Detailed chemical analysis, comparison with existing GRAS cellulosics and migration studies
- Comparison with existing GRAS cellulosics
  - Baking paper
  - Tomato ketchup MFC
- Migration studies

Migration Study Results

- Cold (left) and hot (right) water migration sample cumulative count curves
- MFC containing sheets migrated less than control
- Hot migration samples migrated more than their cold migration counterparts
- Show some higher counts in the finer region for MFC containing samples





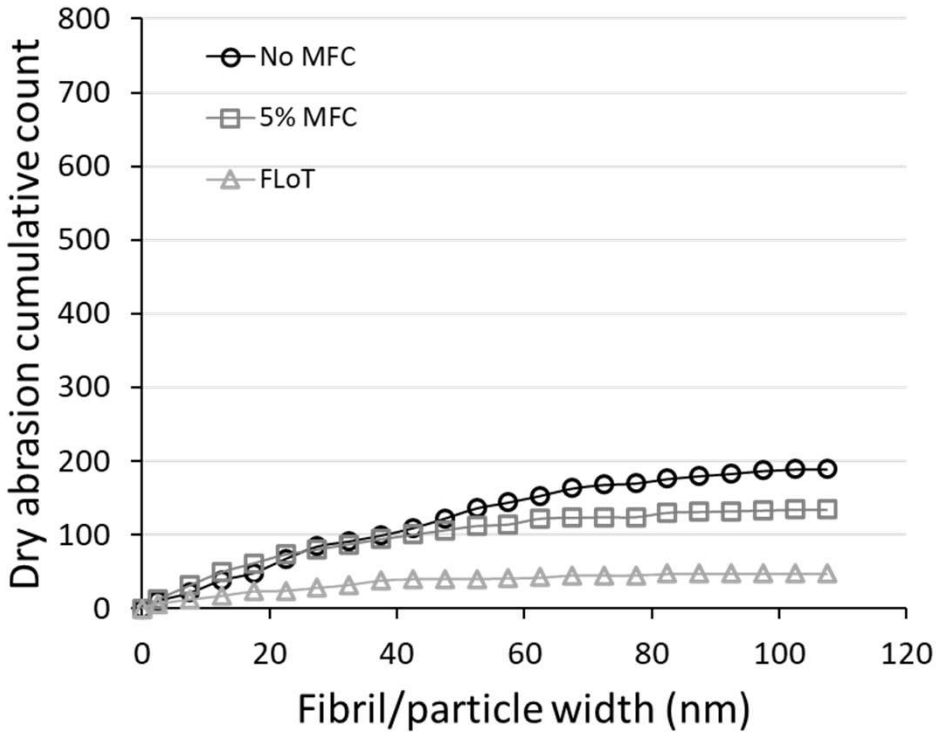
Migration Study Results

Conclusions

Developed a method for comparative analysis of migrated nano-content using SEM

Fewer counts from sheets made with MFC than from sheets made with standard furnish

- 16-83% less migration from cold water tested samples
- 14-58% less from hot water migration samples
- 29-75% less from dry abrasion tested samples



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

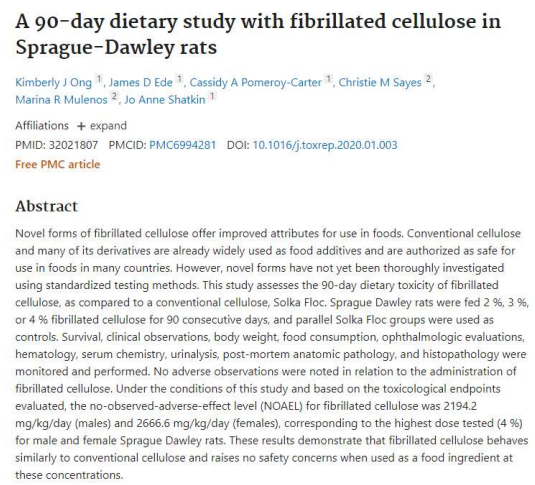
Mineral-free application has been filed with BfR

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

Novel food and food additive allowances

- Vireo Advisors led consortium working to demonstrate the safety of a range of MFC products in food contact and food applications
- (migration studies) + feeding studies + physical chemistry, toxicological characterisation using *in vitro* gastrointestinal digestion and co culture models



A 90-day dietary study with fibrillated cellulose in Sprague–Dawley rats

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Affiliations + expand

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Free PMC article

Abstract

Novel forms of fibrillated cellulose offer improved attributes for use in foods. Conventional cellulose and many of its derivatives are already widely used as food additives and are authorized as safe for use in foods in many countries. However, novel forms have not yet been thoroughly investigated using standardized testing methods. This study assesses the 90-day dietary toxicity of fibrillated cellulose, as compared to a conventional cellulose, Solka Floc. Sprague Dawley rats were fed 2 %, 3 %, or 4 % fibrillated cellulose for 90 consecutive days, and parallel Solka Floc groups were used as controls. Survival, clinical observations, body weight, food consumption, ophthalmologic evaluations, hematology, serum chemistry, urinalysis, post-mortem anatomic pathology, and histopathology were monitored and performed. No adverse observations were noted in relation to the administration of fibrillated cellulose. Under the conditions of this study and based on the toxicological endpoints evaluated, the no-observed-adverse-effect level (NOAEL) for fibrillated cellulose was 2194.2 mg/kg/day (males) and 2666.6 mg/kg/day (females), corresponding to the highest dose tested (4 %) for male and female Sprague Dawley rats. These results demonstrate that fibrillated cellulose behaves similarly to conventional cellulose and raises no safety concerns when used as a food ingredient at these concentrations.

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Physical, chemical, and toxicological characterization of fibrillated forms of cellulose using an *in vitro* gastrointestinal digestion and co-culture model

Sahar H Pradhan, Marina R Mulenos, London R Steele, Matthew Gibb, James D Ede, Kimberly J Ong, Jo Anne Shatkin, Christie M Sayes

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Abstract

Fibrillated cellulose is a next-generation material in development for a variety of applications, including use in food and food-contact materials. An alternative testing strategy including simulated digestion was developed to

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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

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

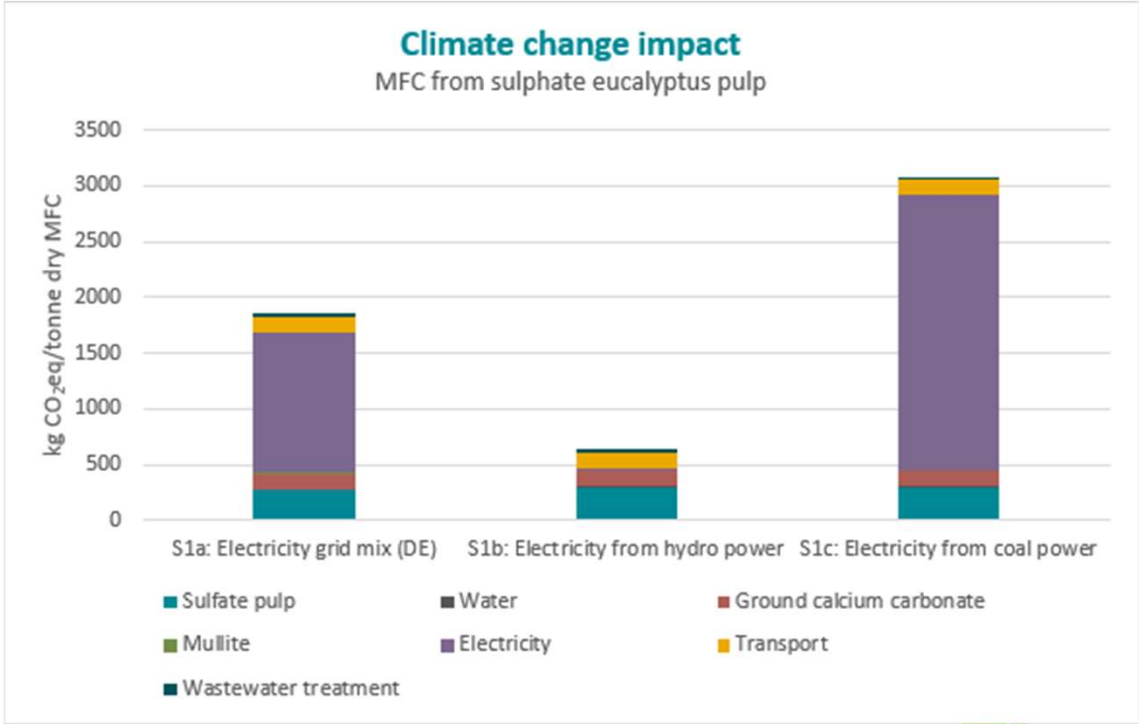
Environmental Impact

- 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

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



Conclusions

- Necessary regulatory clearances are in place covering the use of FiberLean MFC in food contact applications at up to 5% by weight MFC in the sheet. Further clearances are in progress.
- Further work to obtain food status for MFC is in progress.
- MFC coated paper is recyclable and biodegradable and is likely compostable.
- MFC slurry is biodegradable.
- MFC LCA is dominated by electricity consumption and sourcing.

Thank you for your attention

FiberLean Technologies

We are grateful to TAPPI for the opportunity to present this work

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