Commercialising MFC Products: Compliance to Ethical Standards and Legislation

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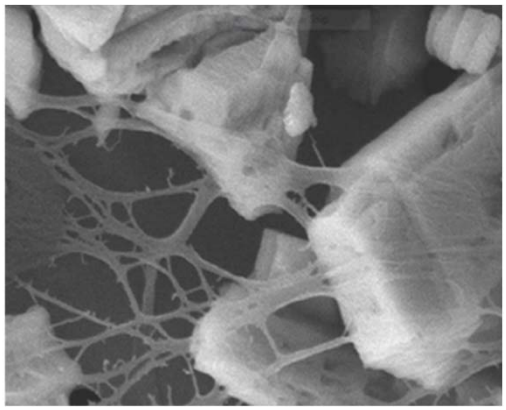
Who we are

- ✓ Established in 2016
  - Formerly a joint venture between Imerys and Omya
  - New owner Werhahn Group 2021
- ✓ 66 Employees
- ✓ One of the world's largest producers of microfibrillated cellulose (MFC)

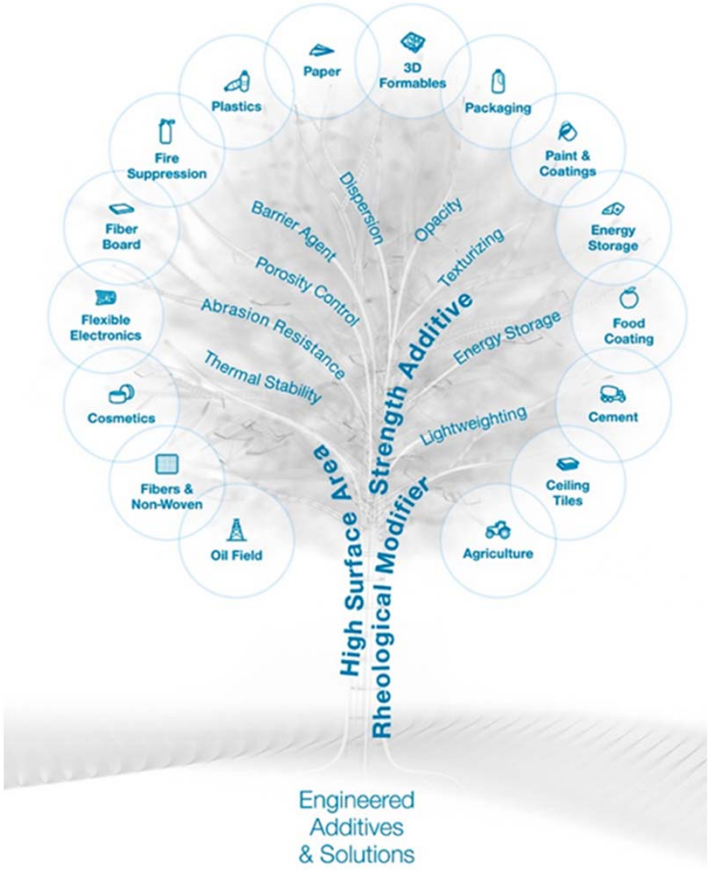
Locations:
R&D laboratory and pilot plant in Cornwall UK
Offices in UK, Belgium, Sweden and US


Expertise: Fibrillation of Cellulose Pulp for production of microfibrillated cellulose (MFC)

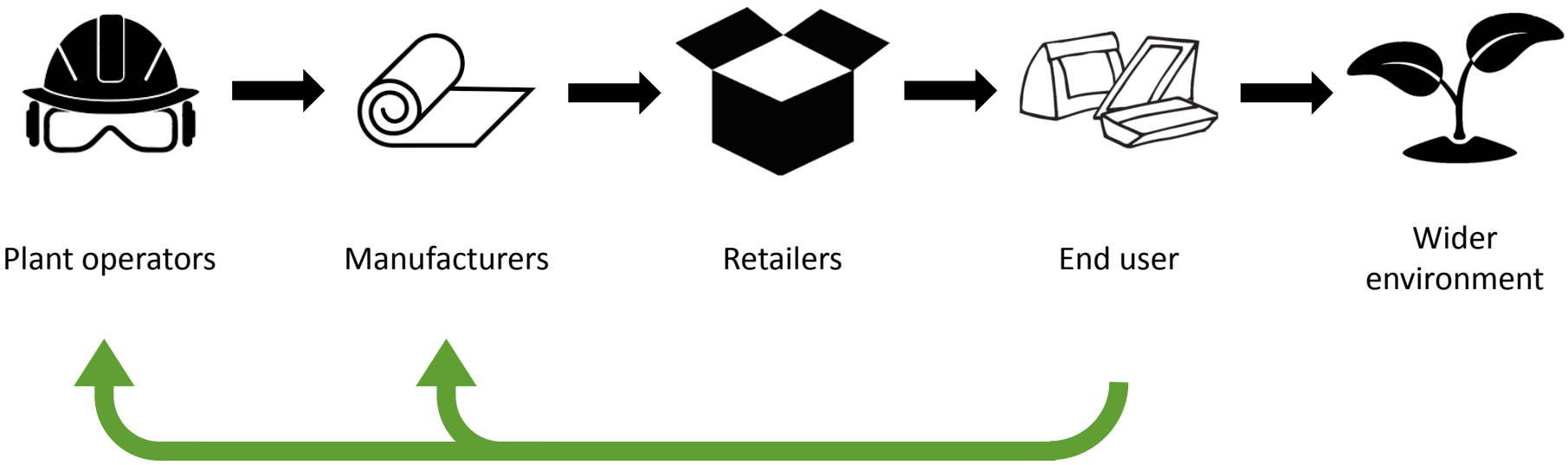
- **Cost effective**, vertical, stirred wet mill based method to produce MFC and mineral/MFC composites from cellulose and minerals
  - **Robust, industrially proven grinding equipment**
  - The **mineral particles act as micro-grinding media**, thus reducing the energy requirement
  - **Scalable**
- **Commercialised as FiberLeanMFC®**
- 12 000 dry metric tonnes pa of fibril capacity (40 000 dry metric tonnes of mineral/MFC composite) operational across three continents. Further capacity under construction
- Version with no mineral available
- Low solids (satellite) and **high solids (merchant) production capability**
- Multiple partnerships with universities, research centres, equipment suppliers, etc.
- **Established sales in paper and packaging**
- **First sales in construction**

MFC – Utility for increased bonding in fibre-based structures, viscosification, reinforcement…



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

Duty of care for the whole supply chain



Regulatory clearances are essential for many target 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

FDA GRAS – applied for as part of Vireo led consortium

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"

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

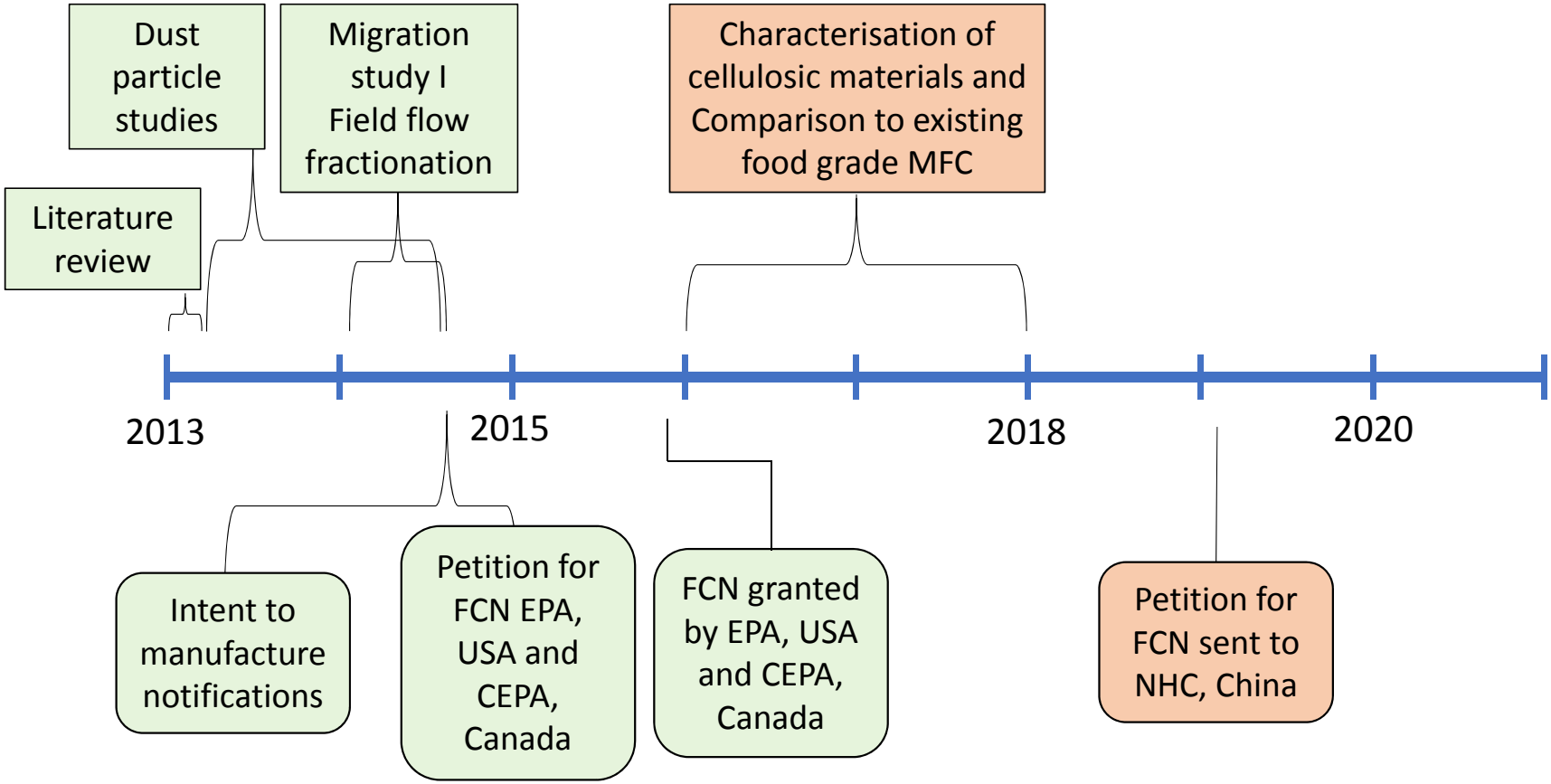
Germany

Acceptance has recently been confirmed for BfR XXXVI and XXXVI/2 at up to 5 wt.% fibrils

Netherlands

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

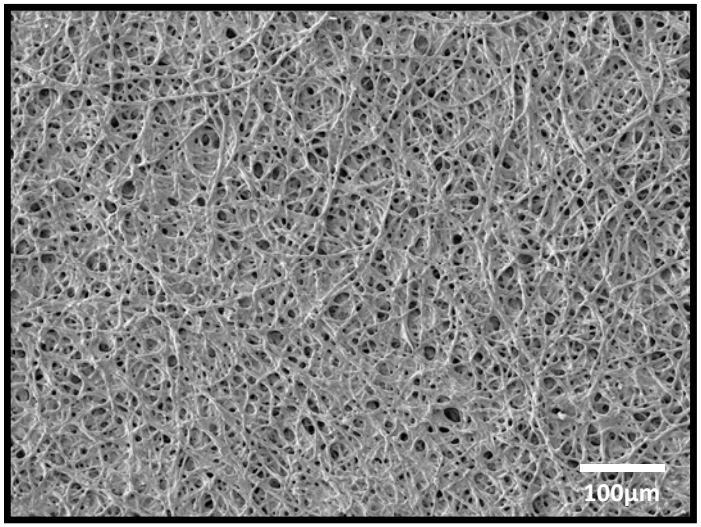
Regulatory timeline

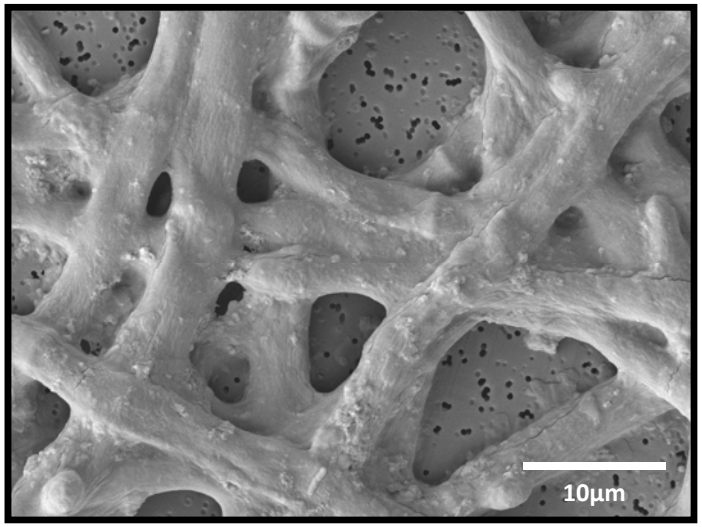


Comparison with existing food grade MFC

Ketchup

Dilution and centrifugation technique





Conducted a range of characterisation test including SEM, fibre analysis, laser diffraction and IR- spectroscopy

SEM Characterisation

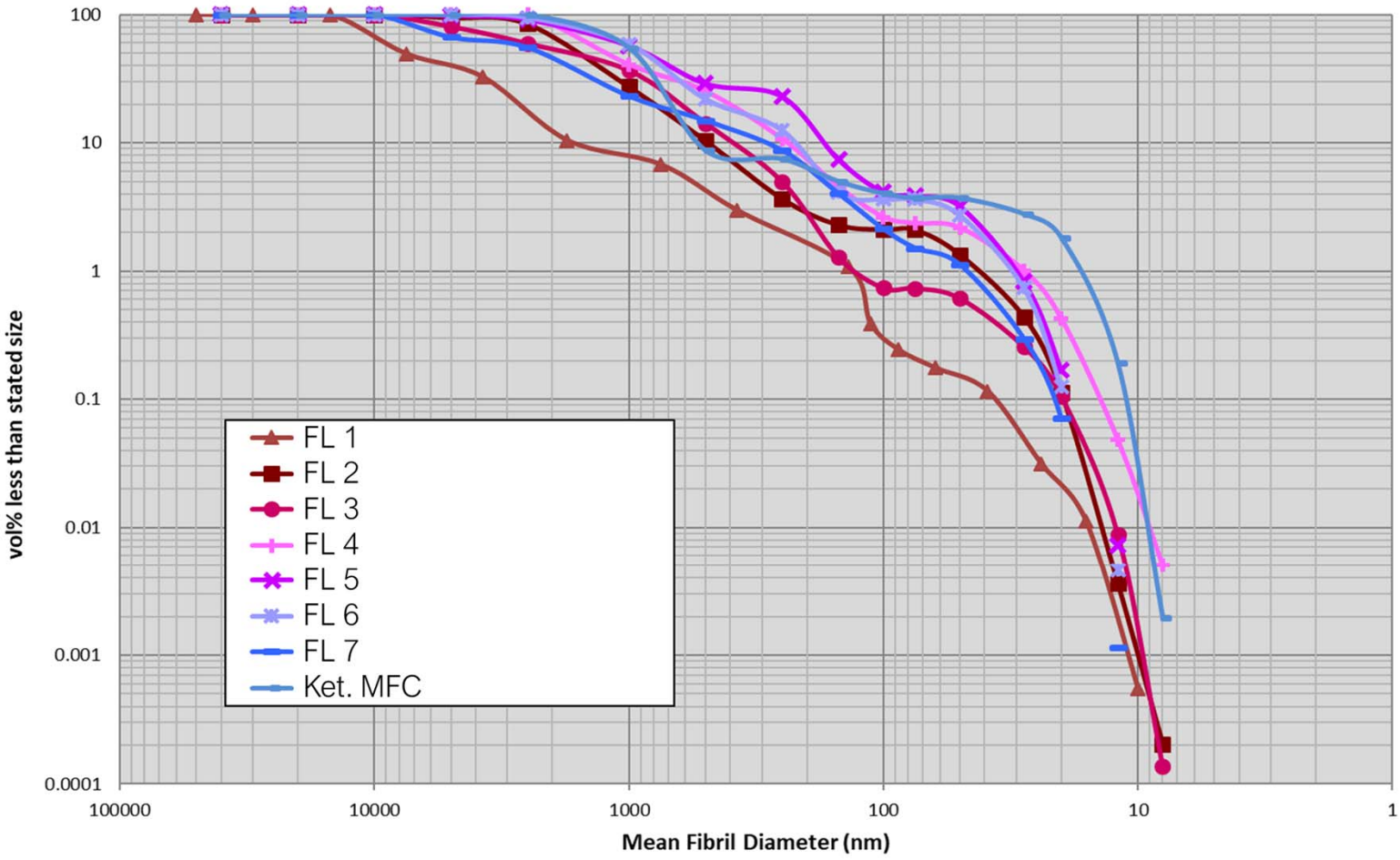
- **SEM images collected at 4 different magnifications**
  - 20 micrographs at x500, x2000 and x5000, 25 micrographs at x150 000
  - Fibril thicknesses measured manually using image analysis software

- **Duplications reduced by defining a size range for a given magnification**
  - 150 000x 0-50 nm
  - 5000x 51-250 nm
  - 2000x 251-2500 nm
  - 500x >2500 nm

- **Cumulative number distribution derived by calculating the number distributions at all four magnifications**

SEM results

SEM fibril analysis:
cumulative volume %



Comparison to existing food grade MFC

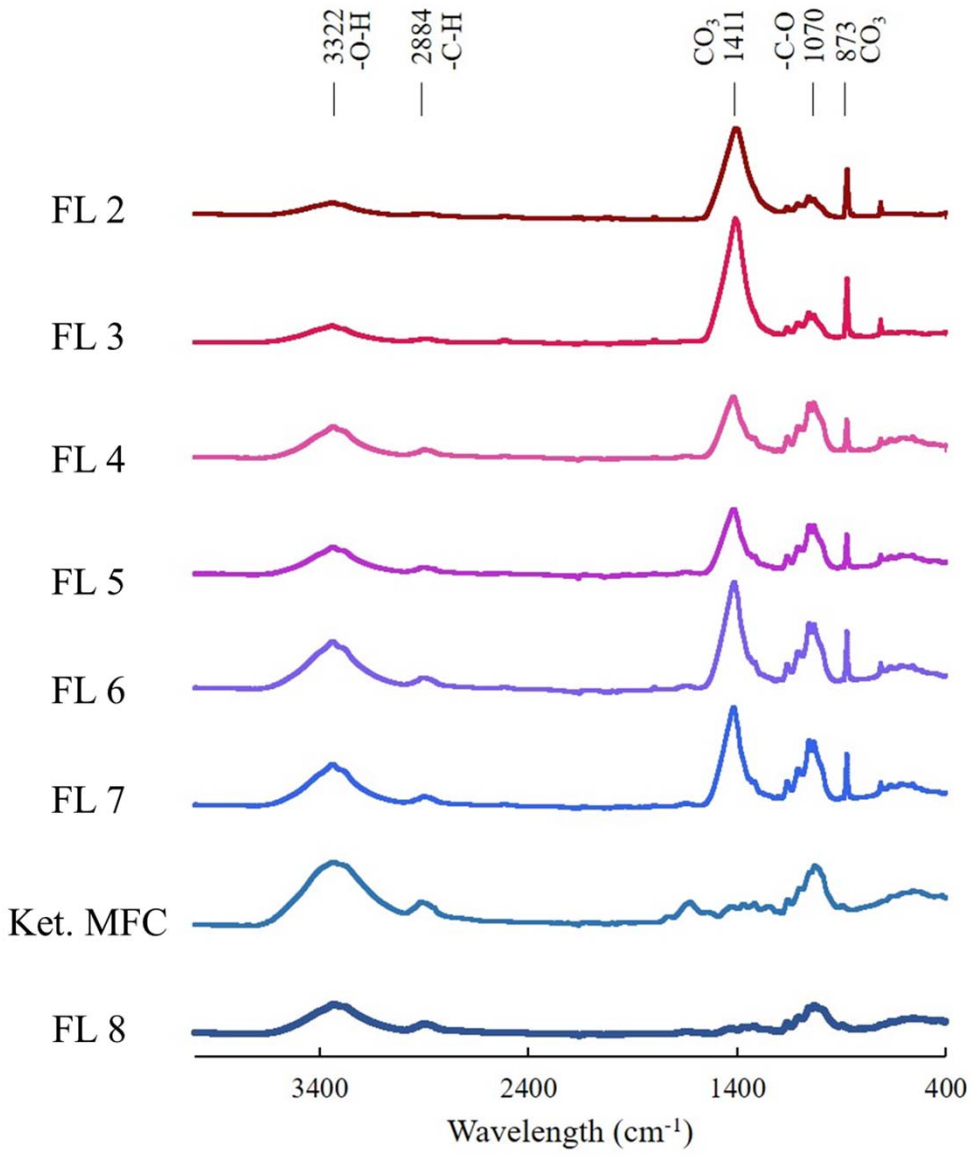
Valmet fibre analyser results

Sample	Arithmetic av. Fibre length (mm)	Length weighted av. Fibre length (mm)	Weight weighted av. Fibre length (mm)
FL 2	0.15	0.27	0.46
FL 3	0.15	0.32	0.87
FL 4	0.14	0.23	0.39
FL 5	0.87	0.11	0.16
FL 6	0.83	0.17	1.63
FL 7	0.90	0.21	0.78
Ket. MFC	0.15	0.24	0.41

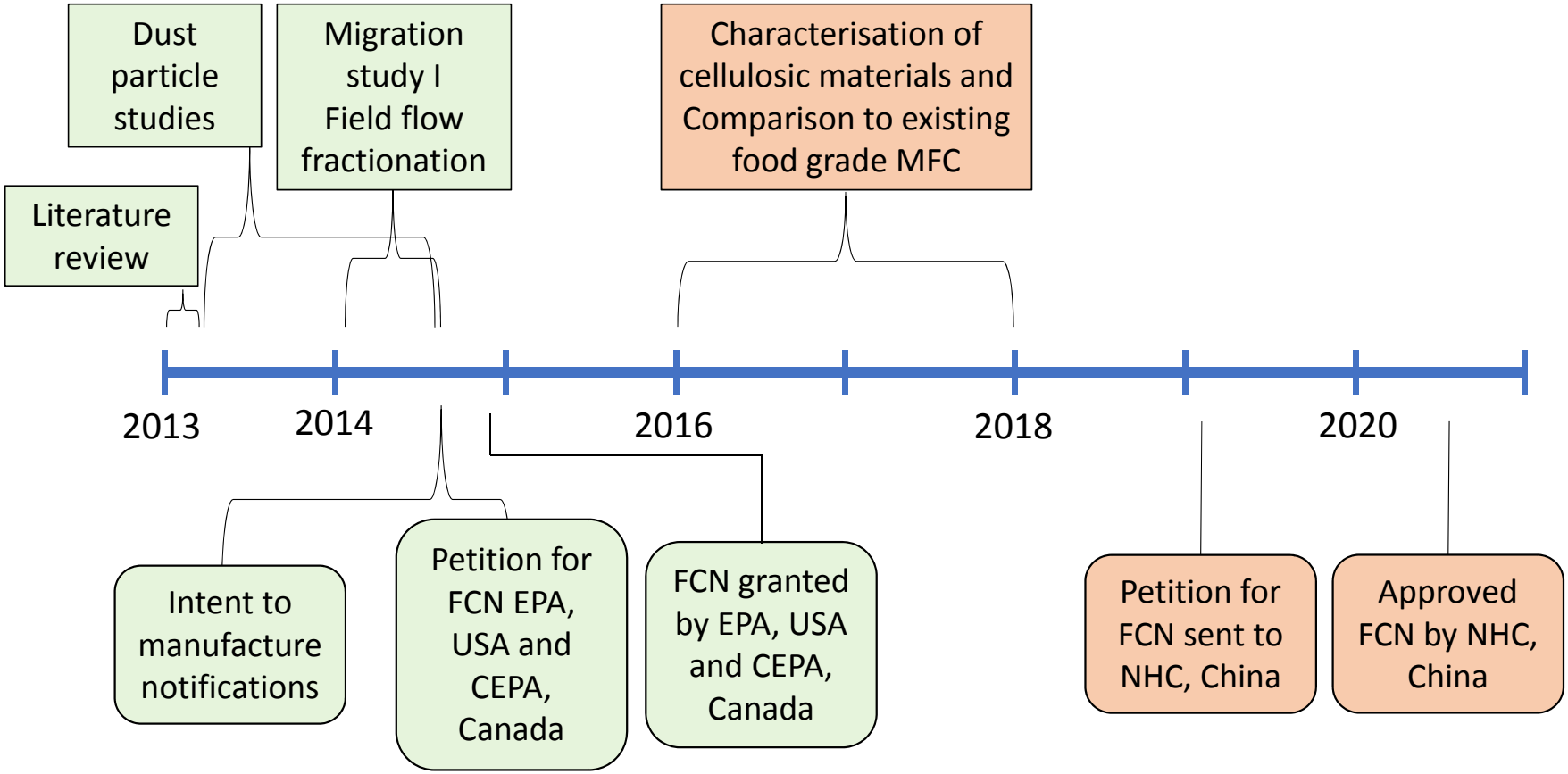
Comparison to existing food grade MFC

- IR spectroscopy

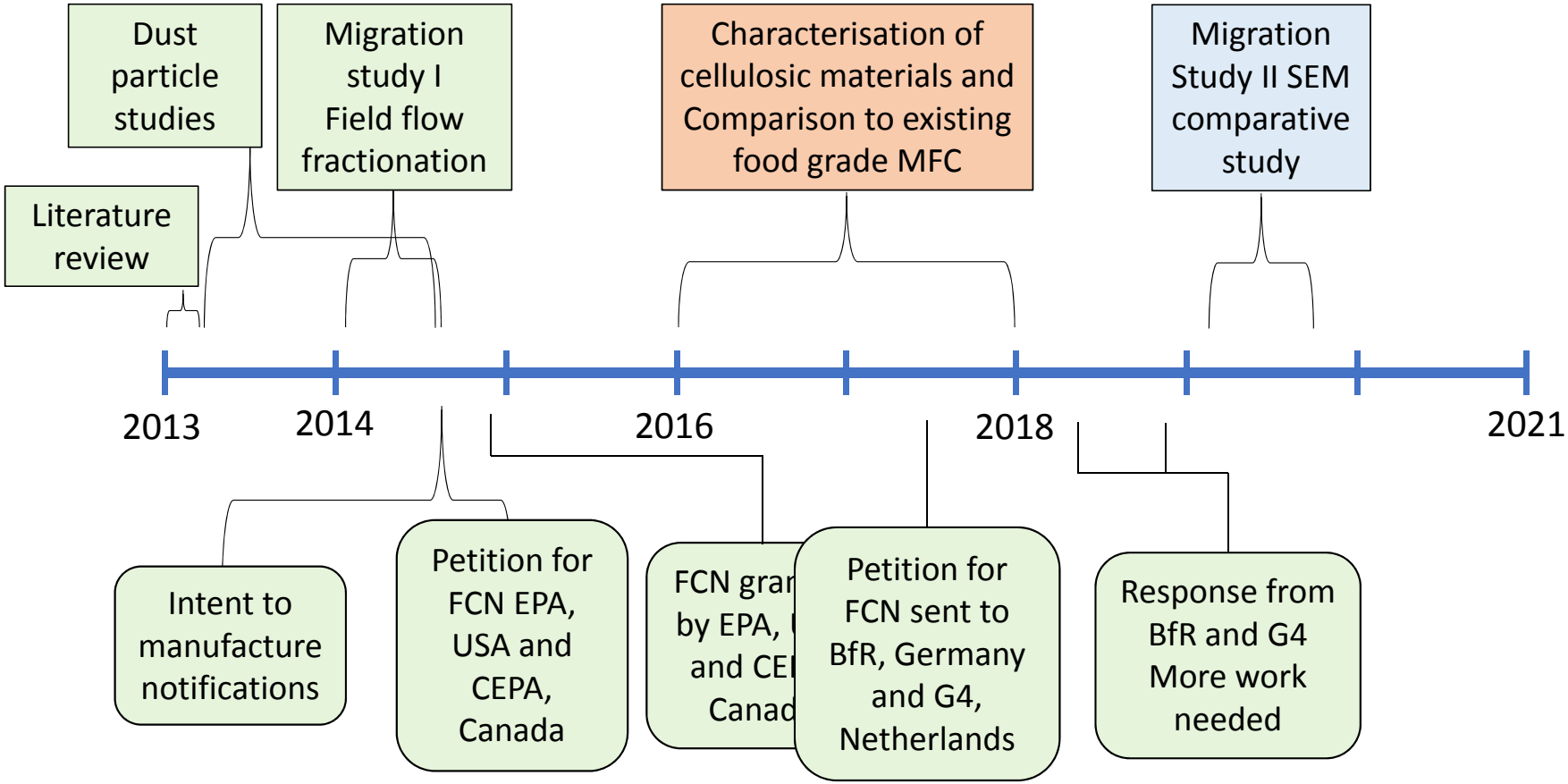
- Overall the work demonstrated strong similarities between the morphological and chemical characteristics of FiberLean MFC products and MFC found in ketchup.



Regulatory timeline



Regulatory timeline



Migration Study II

Sheet making

- 100 gsm sheet
- 70/30 Eucalyptus/Pine bleached kraft pulp
- 20% calcium carbonate mineral
- Pulp refined to 550 CSF
- With and without MFC

Wet and dry migration testing

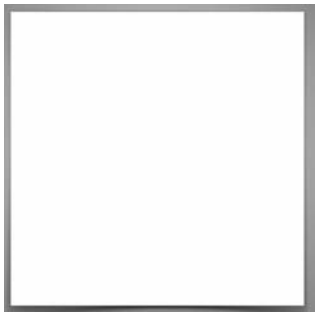
- EN645 Cold water extract
- EN647 Hot water extract
- Dry abrasion test
- Sheets didn't dissolve
- Supernatant decanted
- *Wet MFC added to extraction sample as a control. (placed into non-MFC sample)*

SEM analysis of migration water content

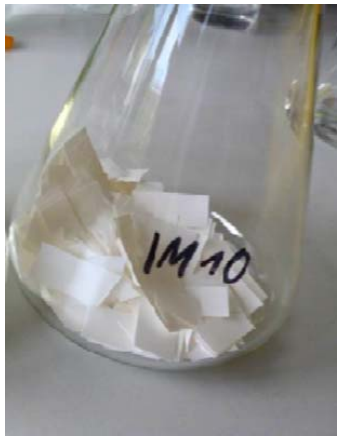
- No standard method for MFC
- Method requires development and calibration

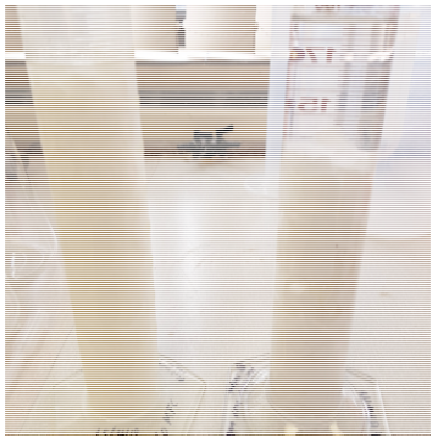
Migration study II

Sample sheet



Wet test

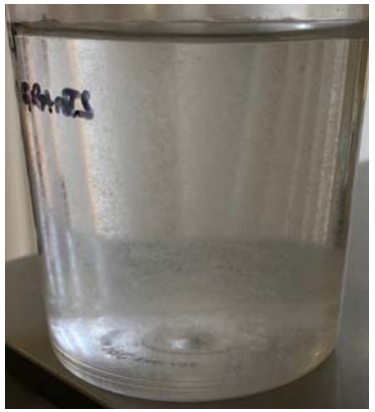




Decant supernatant

Dry test

Suspend debris



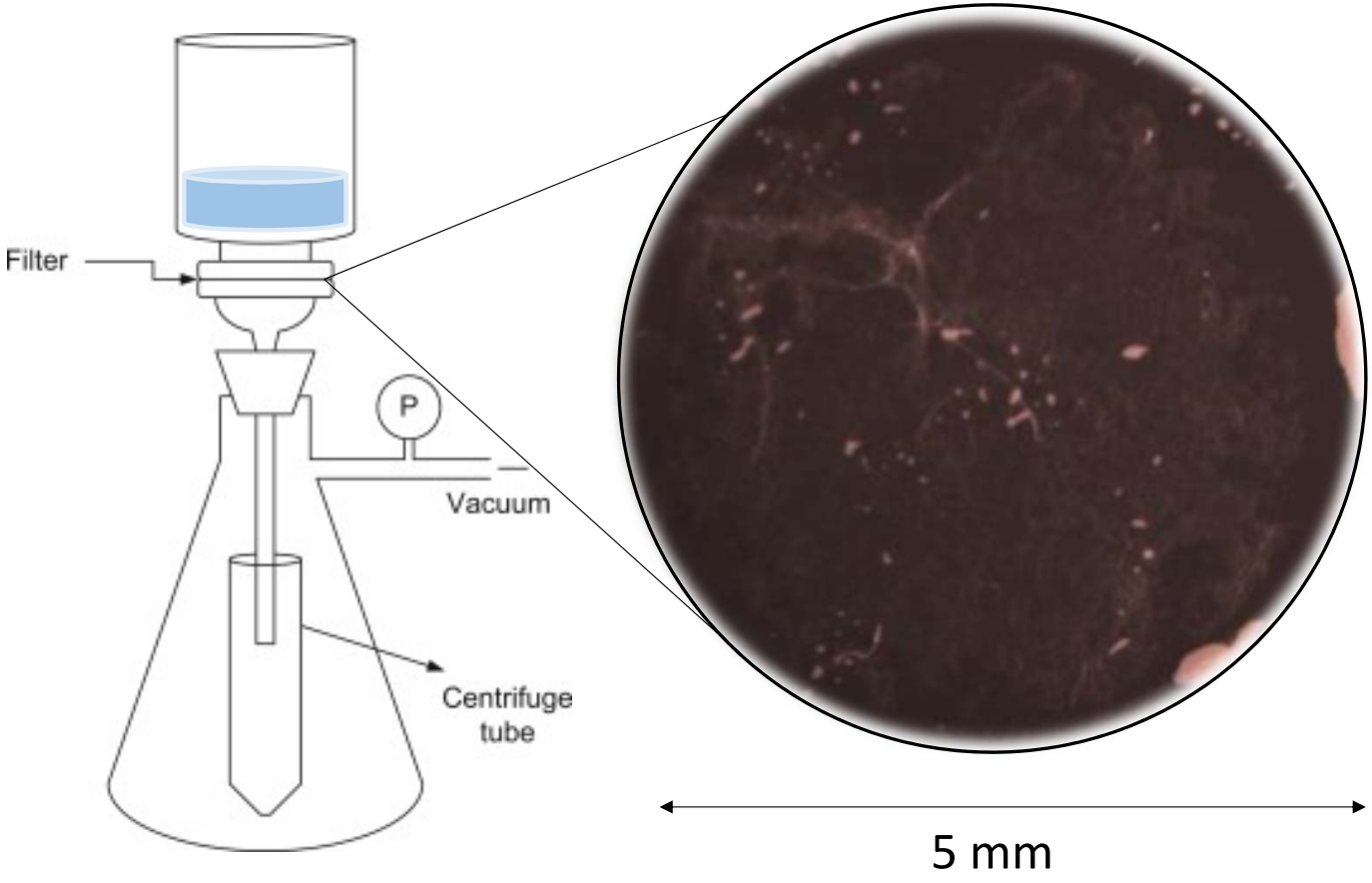
Quantify and compare nano content using SEM

SEM sample preparation

Dilute concentrations are dispersed across a substrate with a 5 mm diameter.

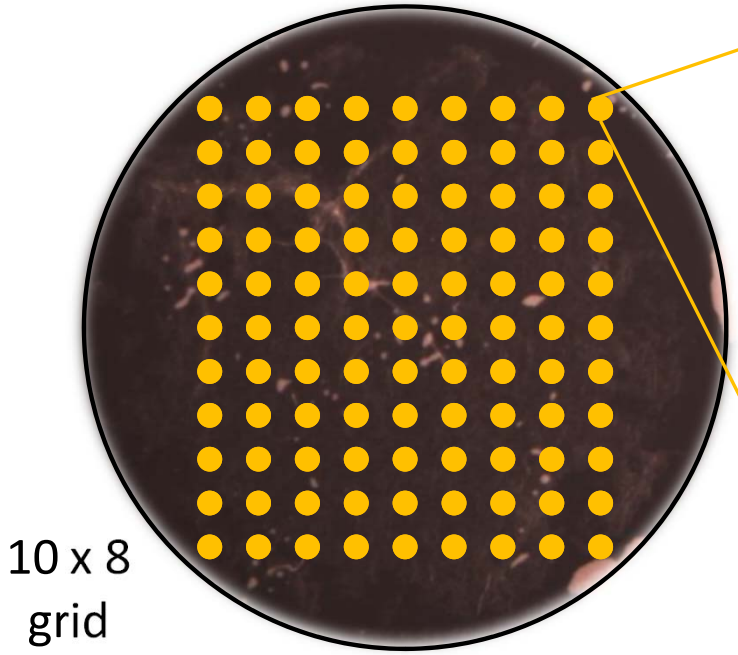
The fibres/fibrils are deposited via filtration over a 0.2 um nucleopore filter.

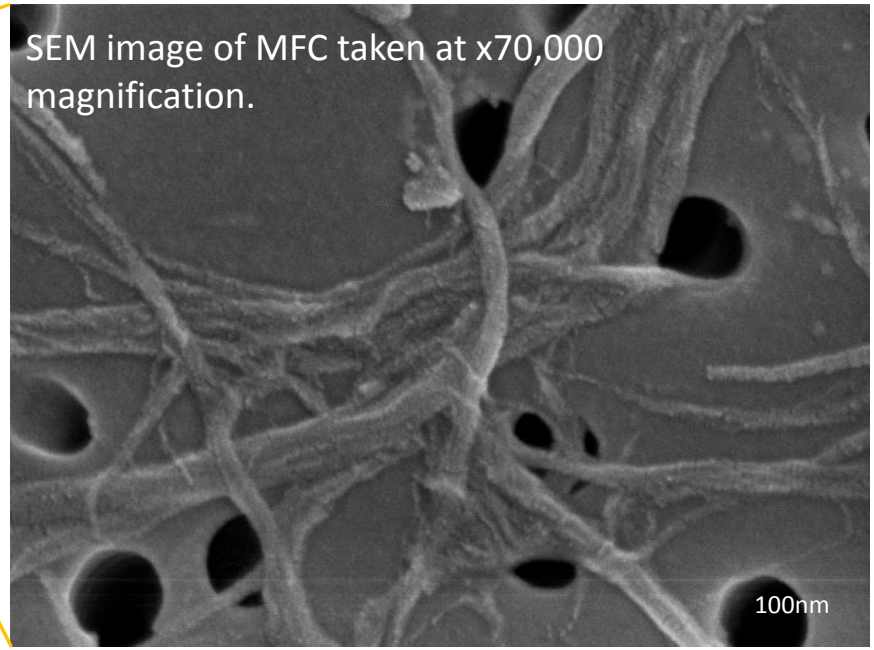
The nucleopore filter is then placed directly onto an SEM stub.



SEM image acquisition method

- Images were acquired from preselected regions to remove operator bias
- 80 images were taken of each sample using a 3x3 mm grid system.





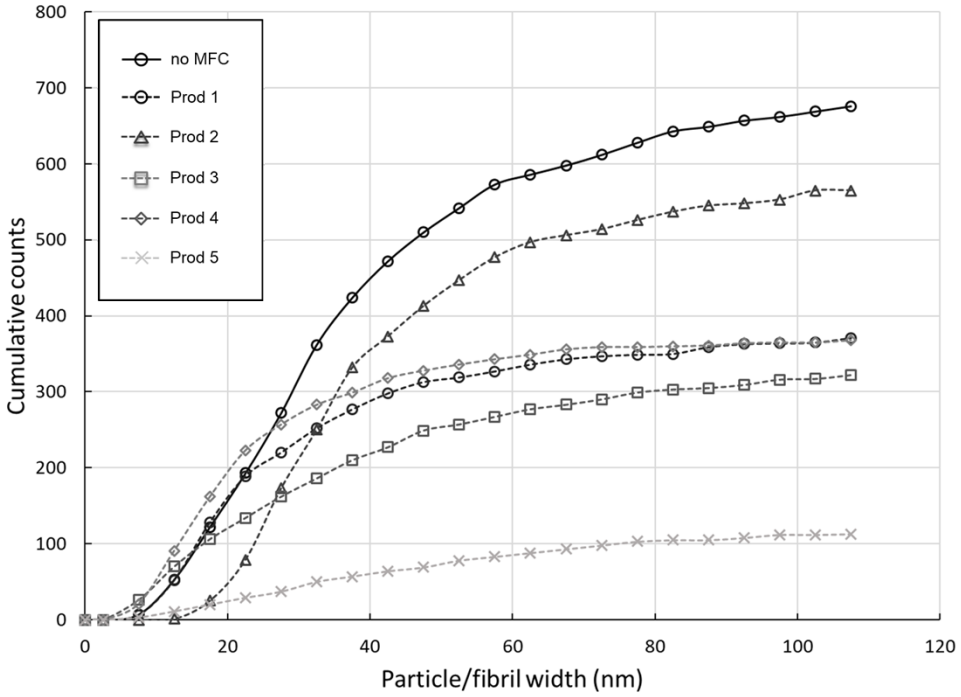
Migration Study Results

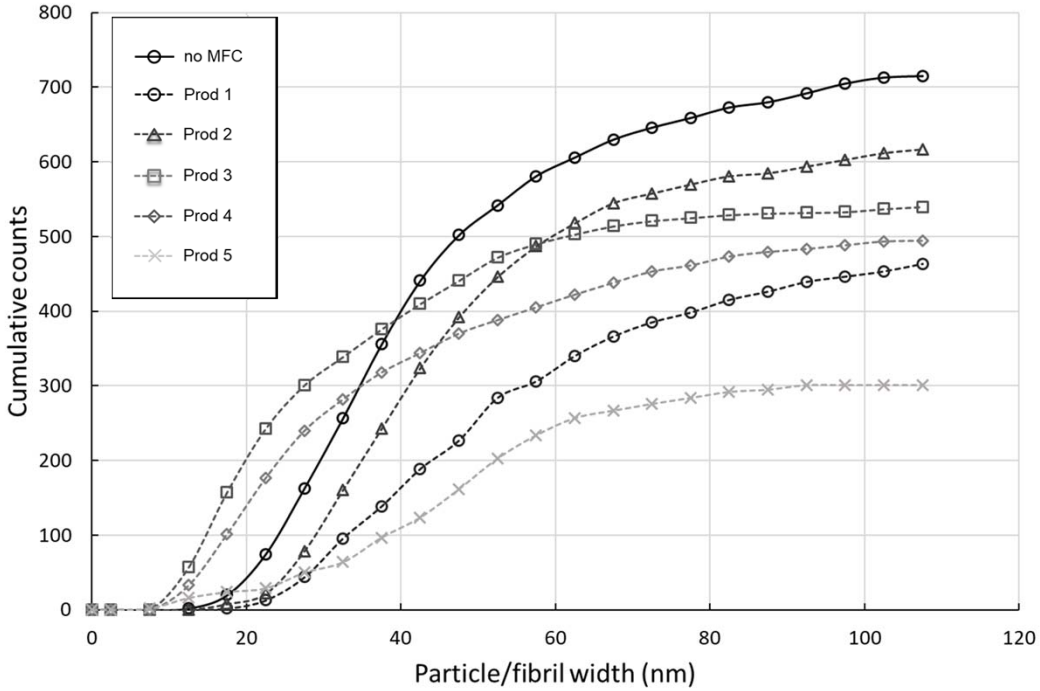
- MFC containing sheets migrated less than control
- Hot migration samples migrated more than their cold migration conterparts.
- Dry abrasion migration is very low

Test and sample		Total number of nanoparticles/fibrils	Normalised Area
Cold migration	No MFC	676	1
	Prod 1	371	0.55
	Prod 2	568	0.84
	Prod 3	322	0.48
	Prod 4	368	0.54
	Prod 5	113	0.17
	Doped	3237	4.8
Hot migration	No MFC	723	1
	Prod 1	470	0.65
	Prod 2	618	0.86
	Prod 3	541	0.75
	Prod 4	499	0.69
	Prod 5	301	0.42
Abrasion	No MFC	188	1
	Prod 1	134	0.71
	Prod 2	47	0.25
	Prod 3	109	0.52
	Prod 4	75	0.39
	Prod 5	61	0.32

Migration Study Results

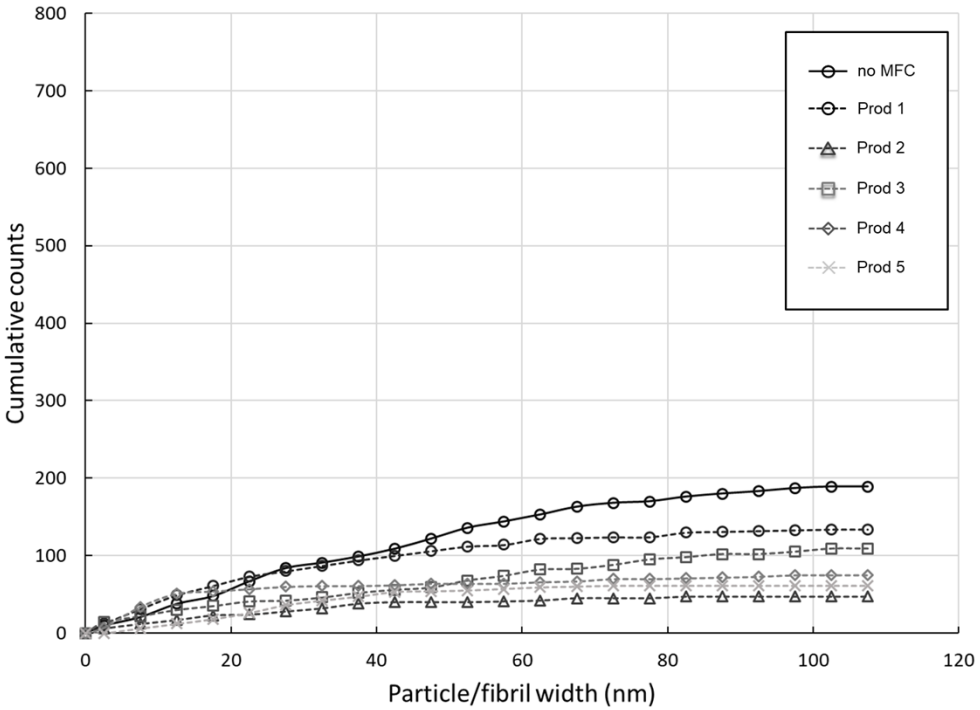
Cold (left) and hot (right) water migration sample cumulative count curves show some higher counts in the finer region for MFC containing samples.





Migration Study Results

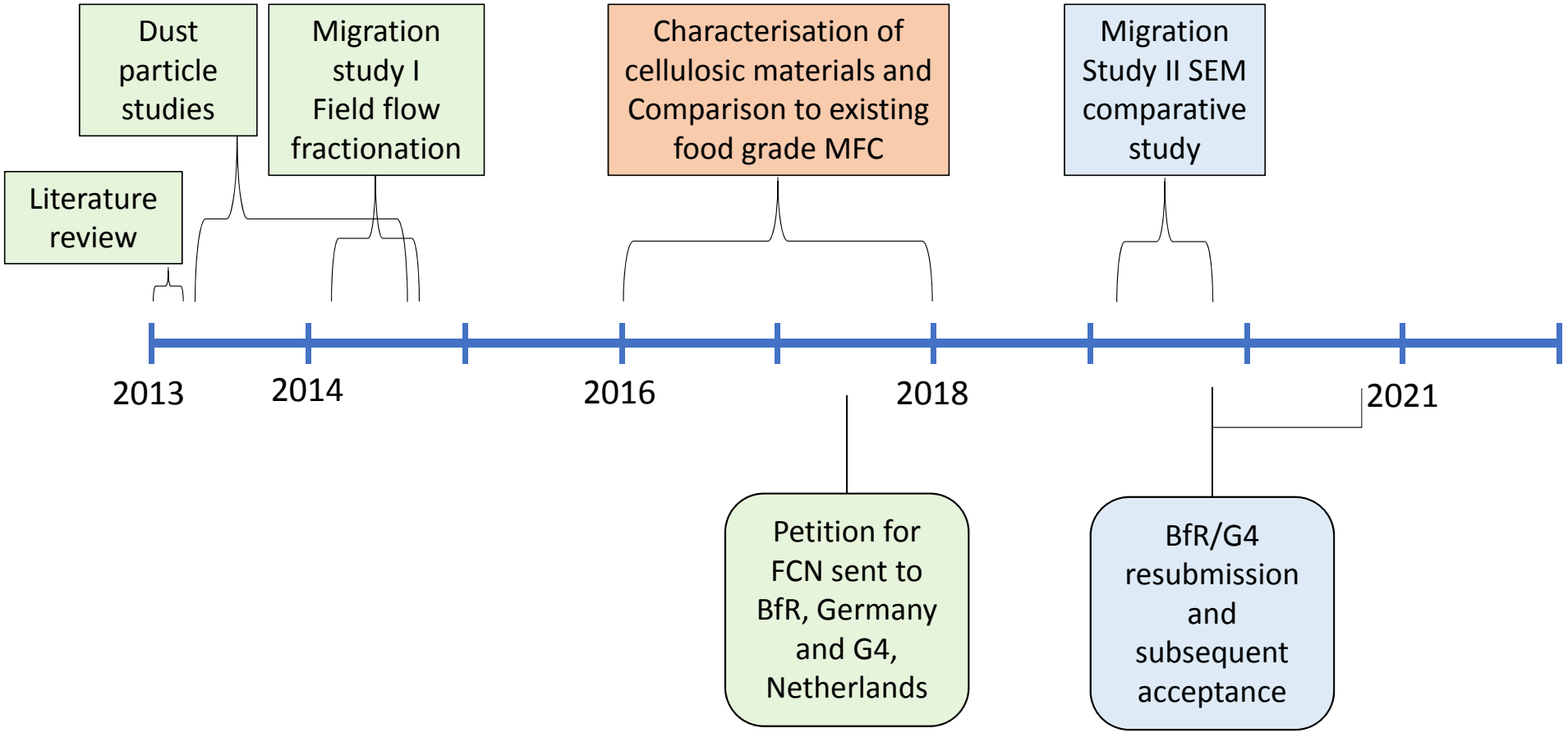
Dry abrasion migration test sample cumulative count curves



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 timeline



Consortium

- Vireo Advisors leading work to demonstrate the safety of a range of cellulose based products

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

Kimberly J Ong [1], James D Ede [1], Cassidy A Pomeroy-Carter [1], Christie M Sayes [2], Marina R Mulenos [2], Jo Anne Shatkin [1]

Affiliations + expand

PMID: 32021807 PMCID: PMC6994281 DOI: 10.1016/j.toxrep.2020.01.003

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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