Novel Fractionation Techniques: Fractionation of MFC suspensions in a viscoplastic fluid

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In this work we explore a novel fractionation technique for microfibrillated cellulose (MFC) particle suspensions based upon the control of the threshold for motion of the particles in a yield stress fluid. We do so in order to produce a higher quality suspension at lower production and energy costs.

Here we explore three different techniques of fractionating commercially available MFC, i.e. a pressure screen, a hydrocyclone, and a novel technique advanced by Madani et al.[1]. In the first part of the work, a small Metso FS-03 pressure screen with three different basket sizes (130, 90 and 60 μm) was used. 15 litre of sample with initially 0.1 % consistency was passed through the system through the 130 μm basket size, then through the 90 μm and finally three stages through the 60 μm basket size. In each step the accept was diluted to 15 litre again and was returned to the system; as a result in the final stages of fractionation the sample consistency was quite low.

In the second part of the work, we repeat the fraction but in this case we used a hydrocyclone. Here a 10 mm C-1201 Y Microspin polypropylene hydrocyclone was used. Since we were looking for the removal of the longer fibres, the reject was collected (As reported in [2] and [3] unlike commercial size hydrocyclones, in small size hydrocyclones short fibres are found in the reject). The fractionation was performed in 4 different stages using 0.2 % pulp consistency and in each step reject was collected and was passed through the hydrocyclone again.

Finally a novel gel fractionation technique was used and compared to the traditional methods presented in the first two stages of the work. MFC suspensions were mixed into a weak gel and subjected to a centrifugal force. The centrifuge in use was an Eppendorf 5804. The gel in use was Carbopol-940 obtained from Noveon. Due to the presence of yield stress, natural settling would not happen in this fluid and as long as the applied force is smaller than the resisting force, the particles would stay trapped in the fluid. Under the applied centrifugal force, some fibres would move and some would remain trapped. This fractionation technique is detailed in Madani et al. [1]. After applying the centrifugal force, the particles were collected in two fractions. The top fraction which included the fibres trapped in the fluid and the bottom fraction which contained the fibres which had moved to the bottom of the test tube.

The key findings from this study are presented in Figure 1. In Figure 1 the change in the average fibre length is reported as a function of the number of fractionation stages. Four stages of treatment for hydrocyclon and five stages of treatment for pressure screen were imposed; the gel fractionation occurred in one stage. The results show that all the three methods are capable of removing longer fractions of MFC. With regards to the pressure screening results, the first two steps of screening are not very effective but as soon as the 60 μm basket was used, a reduction in average fibre length was observed. In this case, the average fibre length decreased from 221 μm to 192 μm. For the hydrocyclone, after second fractionation stage the length did not change from the value 180 μm. Gel fractionation showed higher capability compared to hydrocyclone and pressure screen and without multi step fractionation the average length of 100 μm was achieved.

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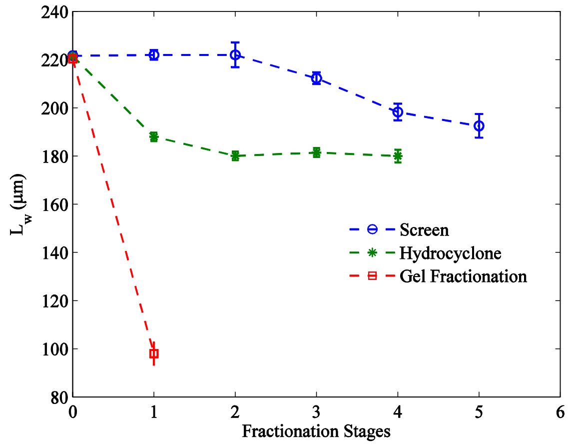

Figure 1-Fibre average length variation using different fractionation techniques.

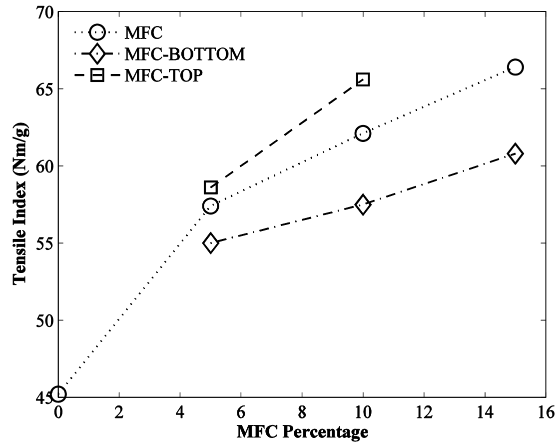

Figure 2-Improvement in tensile properties by addition of MFC before and after fractionation.

In the next set of experiments, composites were formed in a process similar to paper making. MFC was added to a chemical pulp in 3 different mass concentrations (5, 10 and 15 %). Then the pulp was passed through a handsheet former and sheets of paper were formed. Mechanical strength tests were performed on the handsheets. The results presented in Figure 2 shows a significant increase in tensile strength of the sheets. More than 30% increase in tensile index was observed by addition of 10% of MFC to the handsheet. Similar results have been reported in the literature [4]. Finally in order to see the effect of fractionation, the fractionated MFC (using gel technique) collected from top and bottom of the test tubes were added to the pulp. As expected, the top portion including more short fibres had more positive effect on the tensile index compared to the un-fractionated pulp. The bottom fraction which contains relatively longer fibres had less positive effect on the tensile index compared to the original pulp but still an increase in the tensile index was observed. Increasing the MFC content had a negative effect on the drainage time of the handsheets. Especially when 15% of top portion was added, the drainage time was too long that the experiments had to be stopped.

References

[1] Madani A., Storey S, Olson J.A, Frigaard I.A., Salmela J. & Martinez D.M. "Fractionation of non- Brownian rod-like particle suspensions in a viscoplastic fluid" *Chem. Eng Sci* 65(5), pp.1762-1772(2010).

[2] Bliss, T. "Secondary fibre fractionation using centrifugal cleaners", proceedings from the 1984 TAPPI Pulping Conference (Book2), Seattle , 217–225(1984).

[3] Paavilainen, L. "The possibility of fractionating softwood sulfate pulp according to cell wall thickness", Appita J. 45(5), 319–326(1992).

[4] Nakagaito, A.N., & Yano, H. "Novel high-strength biocomposites based on microfibrillated cellulose having nano-orderunit web-like network structure", *Appl. Phys.* A80, pp. 155–159 (2005).

Novel fractionation methods: Separation of MFC in a viscoplastic fluid

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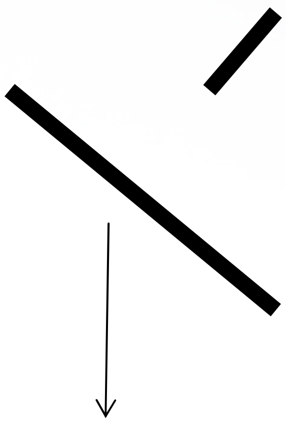
Introductory Comments

- Objective – methodology to purify commercially available MFC
- Assess enhancement in paper properties
- Motivation/efficiency

Part 1. Understanding efficiency: the toy problem

Toy Problem

Goal: Separation of rods based upon length through settling



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Ideal conditions: Stokes flow (Re=0), isolated (C=0), quiescent flow field

$$\mathbf{u} = \frac{\Delta\rho d^2}{16\mu}\left[\left(\ln 2r + 0.193\right)\mathbf{g} + \left(\ln 2r - 1.807\right)\left(\mathbf{p}\cdot\mathbf{g}\right)\mathbf{p}\right]$$

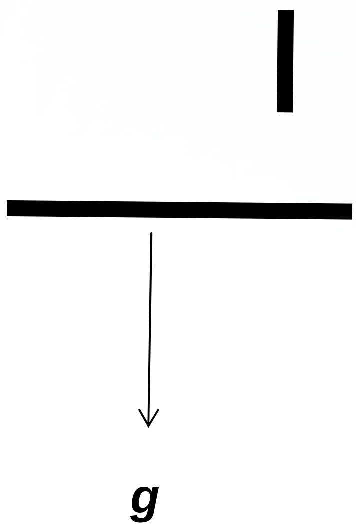
p = orientation
r = aspect ratio
g = gravity

- ***no unique settling velocity! Settling velocity dependent on orientation***

- ***drift velocity of the same order of magnitude as settling***

Toy Problem

Goal: Separation of rods based upon length through settling



Ideal conditions: Stokes flow (Re>0), isolated (C=0), quiescent flow field

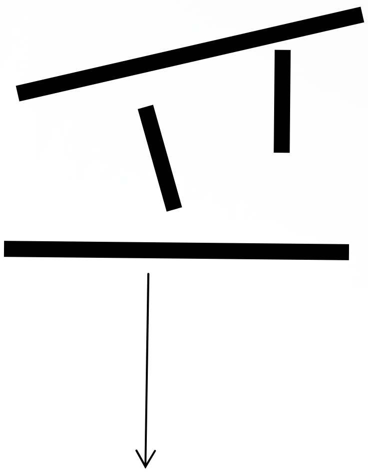
$$\rho C_d(u)u^2 = \frac{g\Delta\rho V}{A}$$

Mass/area

- ***stress acting on the particle is not symmetric***
- ***adopts preferential orientation (horizontal)***

Toy Problem

Goal: Separation of rods based upon length through settling



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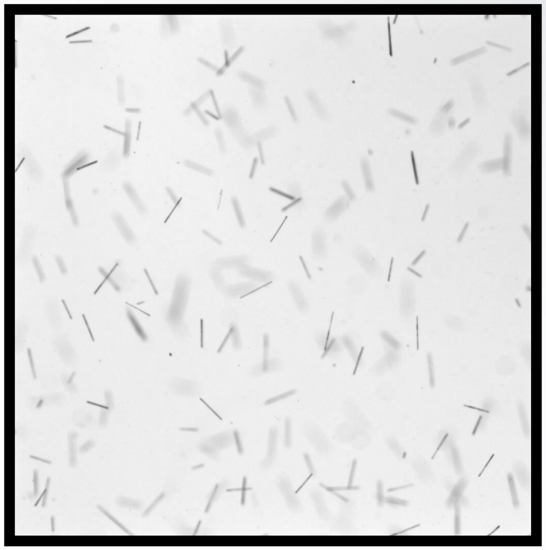
Ideal conditions: Stokes flow (Re>0), isolated (C>0), quiescent flow field

- ***stress acting on the particle is not symmetric***
- ***long range hydrodynamic disturbances (chaotic)***

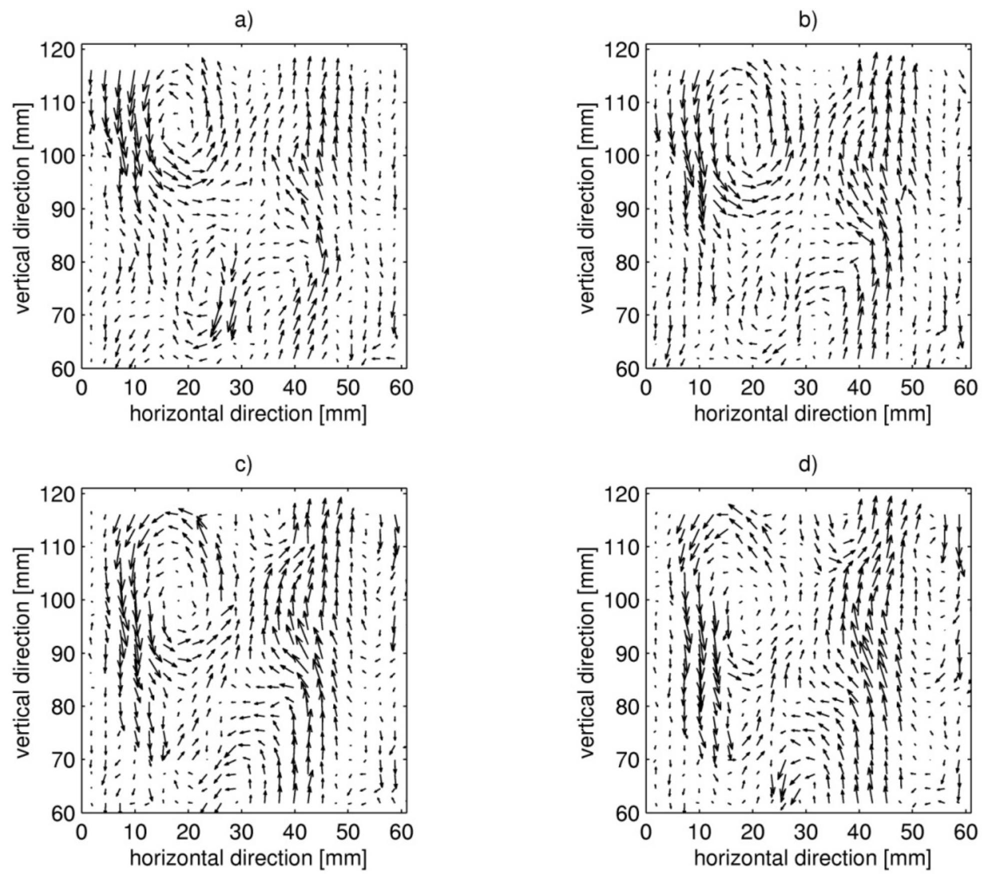
Flow Visualization: PIV Results

N = 0.01



N = 5

Salmela,Kataja, Martyinez, AIChE J. (2007)

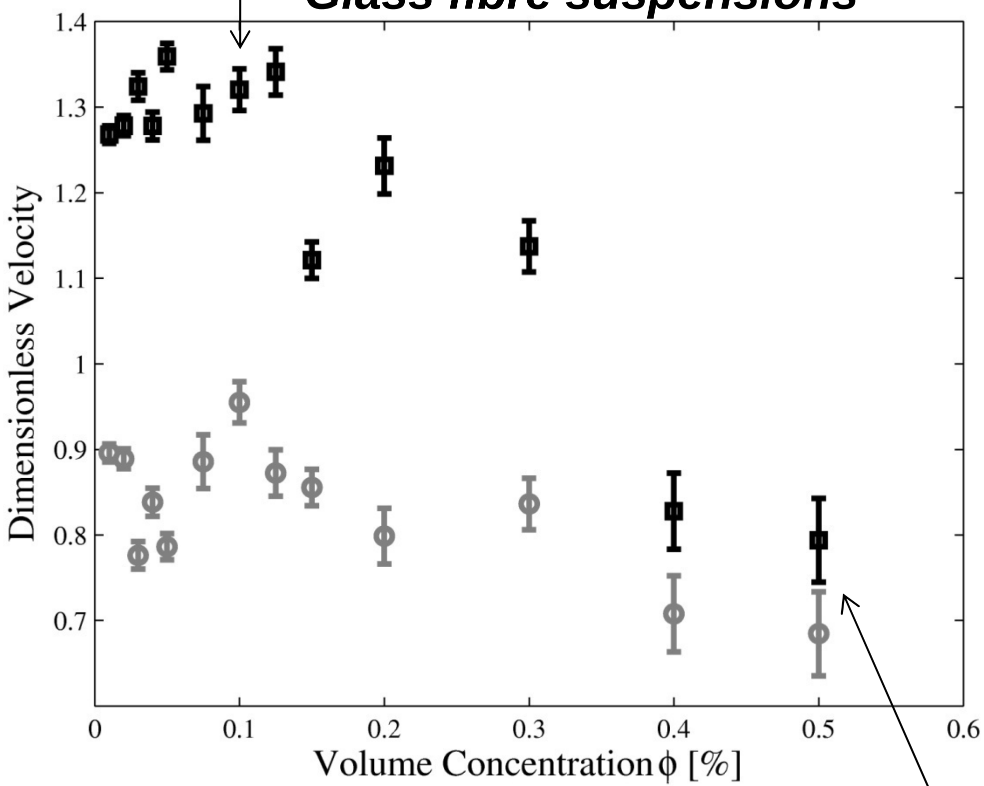


Holm, martinez et al Progress in paper Physics (2005)

Is Separation Possible under ideal conditions?

Separation under dilute conditions

Glass fibre suspensions



Salmela et al, FRC, Oxford, 2009

No separation at Higher concentrations (N~5)

Papermaking fibre suspensions

PET results indicate no significant differences in settling velocity of different fractions in mechanical pulp suspension

Martinez et al FRC Oxford 2001

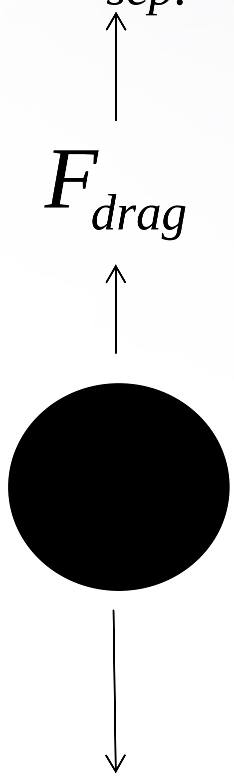
- **Part 2. The Novel Principle**

Goal:
•dampen long range interaction (isolated particle)
•separation based upon physical property

Toy Problem

A new threshold for motion

$F_{sep.}$



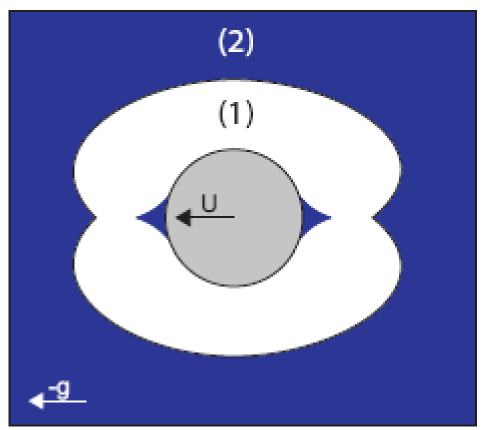
F_{drag}

$F_{driving}$

Separating force has two functions:

- ***dampen ALL disturbances***
- ***create the separation principle***

Solution: Change the rheology !



Beris et al., JFM 1985

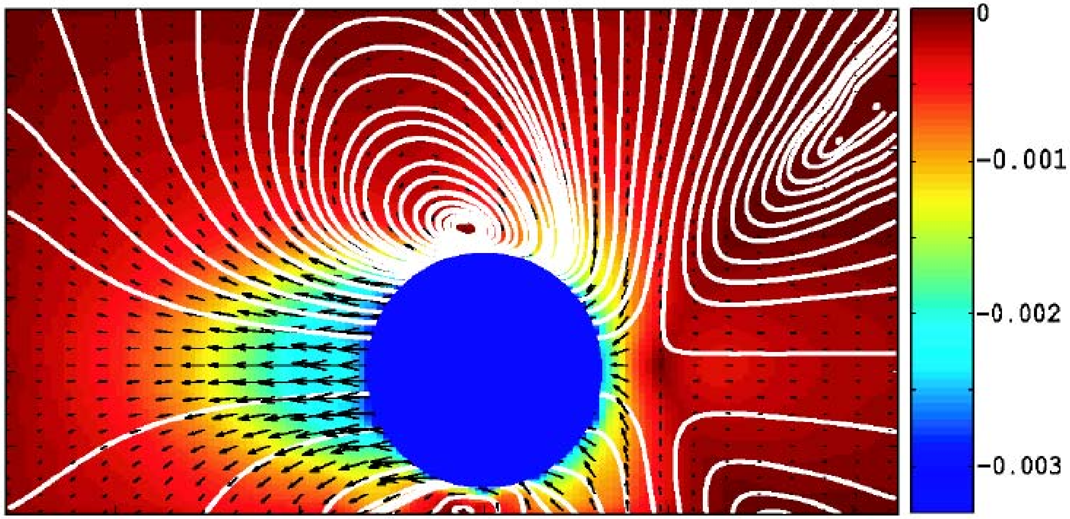
Dampen the forces

Separation Principle

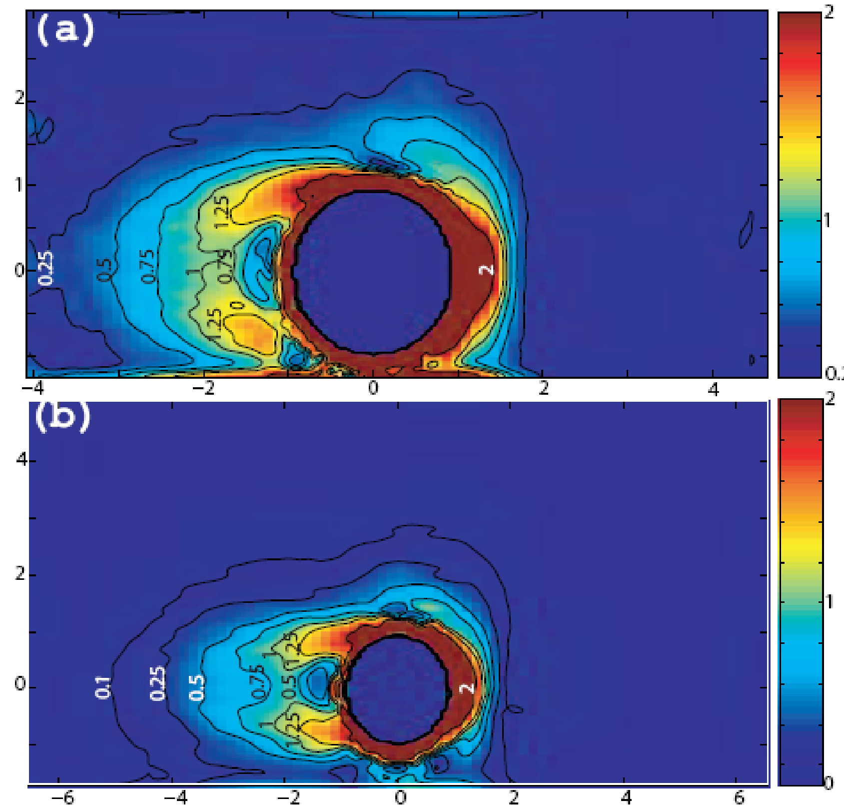
$$\frac{F_{driving}}{F_{sep}} = \frac{g\Delta\rho V}{\tau_y A} > 1$$

Mass/area

Estimates of the Interaction Area



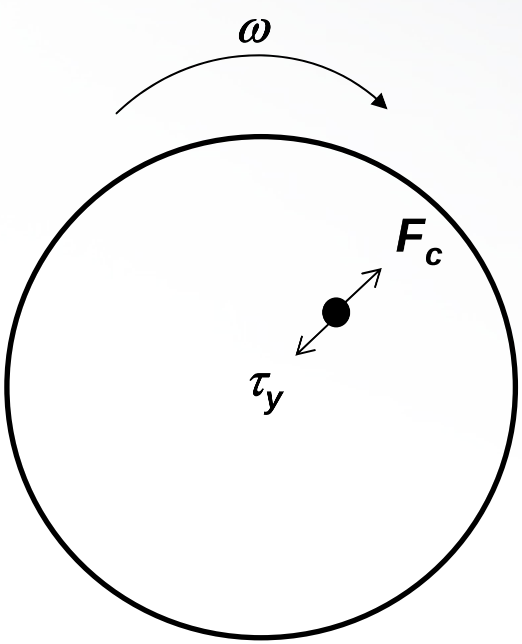
Flow Field



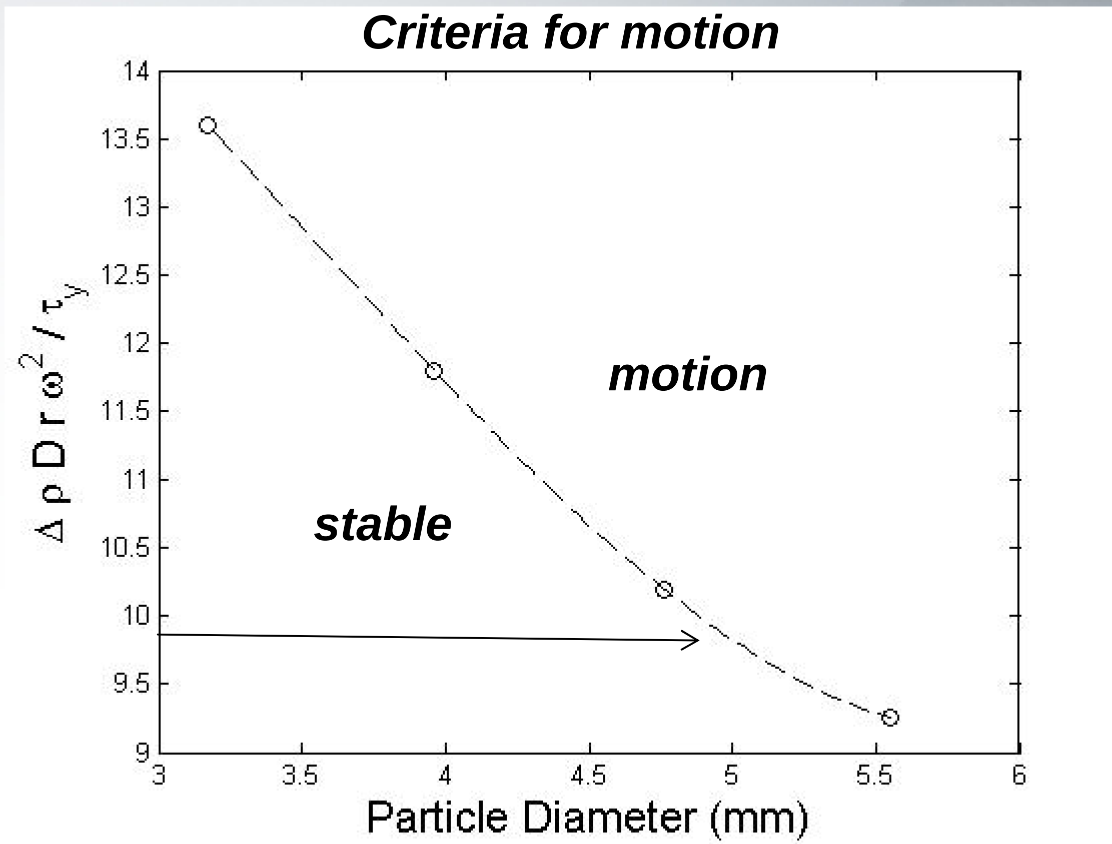
Second Invariant of the strain rate tensor

Putz, Burgehela, Martinez & Frigaard, Phys Fluids 2008

Initial Experiments



centrifuge



Proof of Principle

Centrifuge Test with a Yield Stress Fluid

Suspension of black and red particles

Migration of black particles

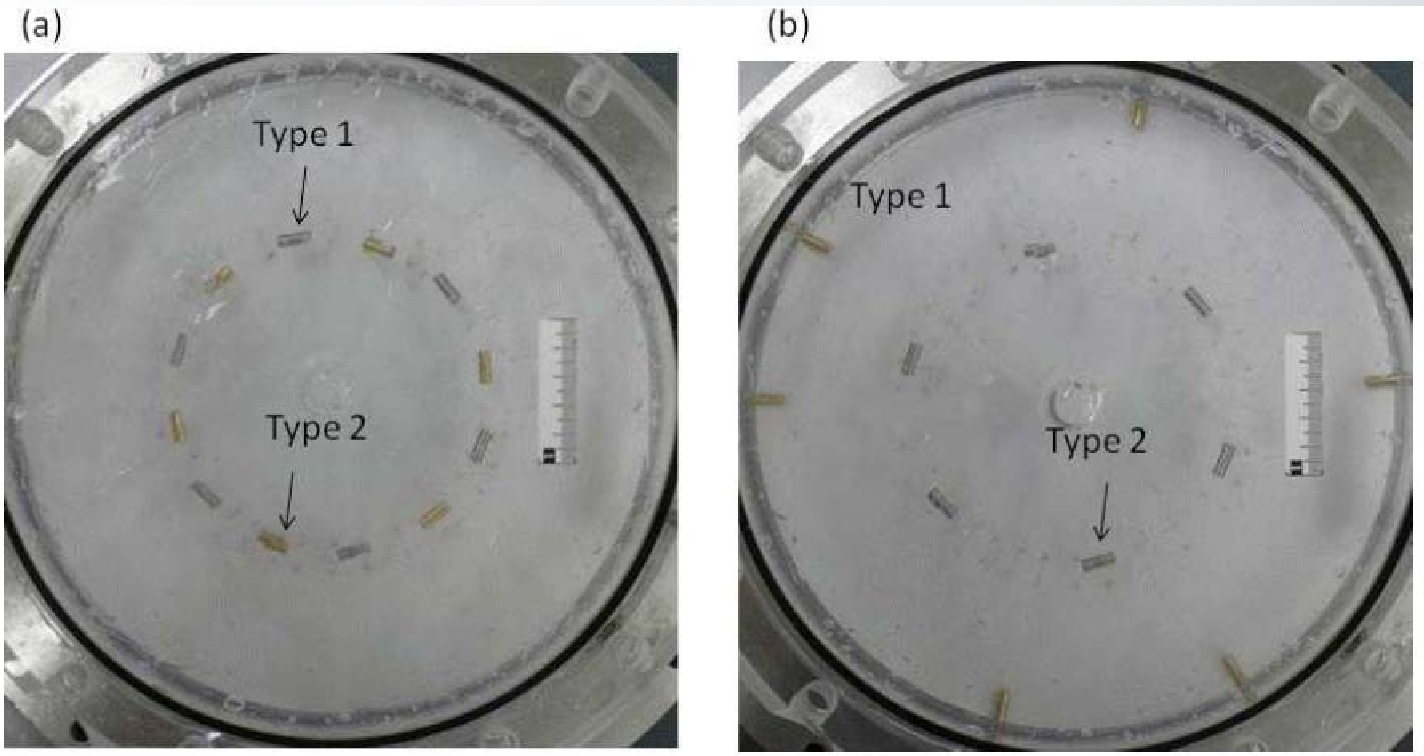
start

Finish

Proof of Principle

Centrifuge Test with a Yield Stress Fluid

Different density rods

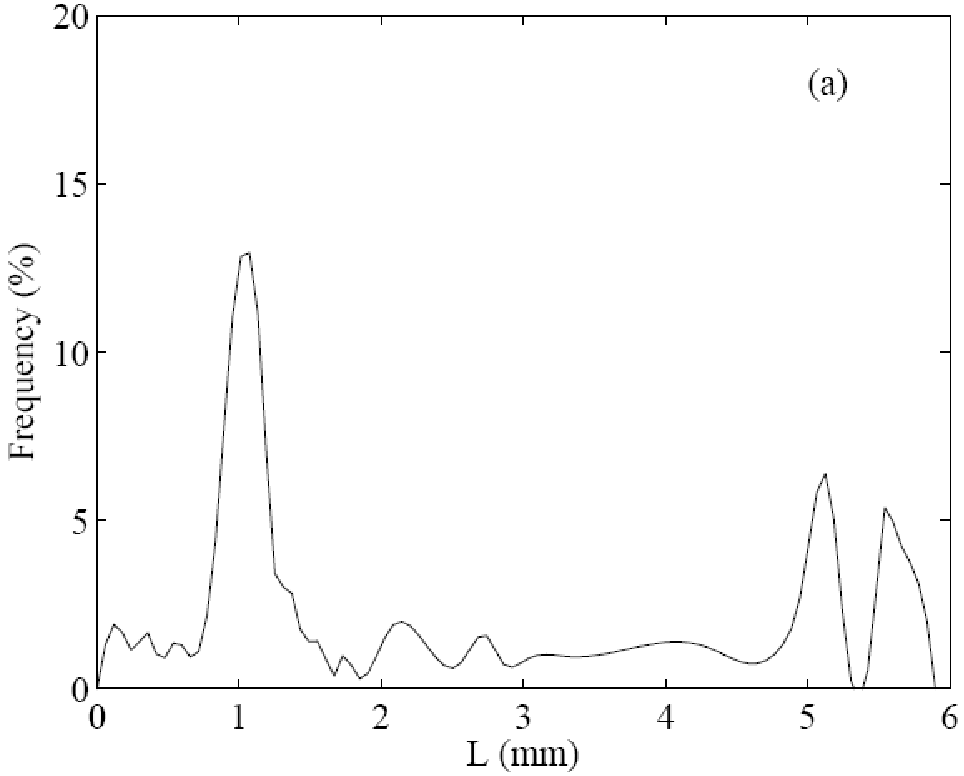


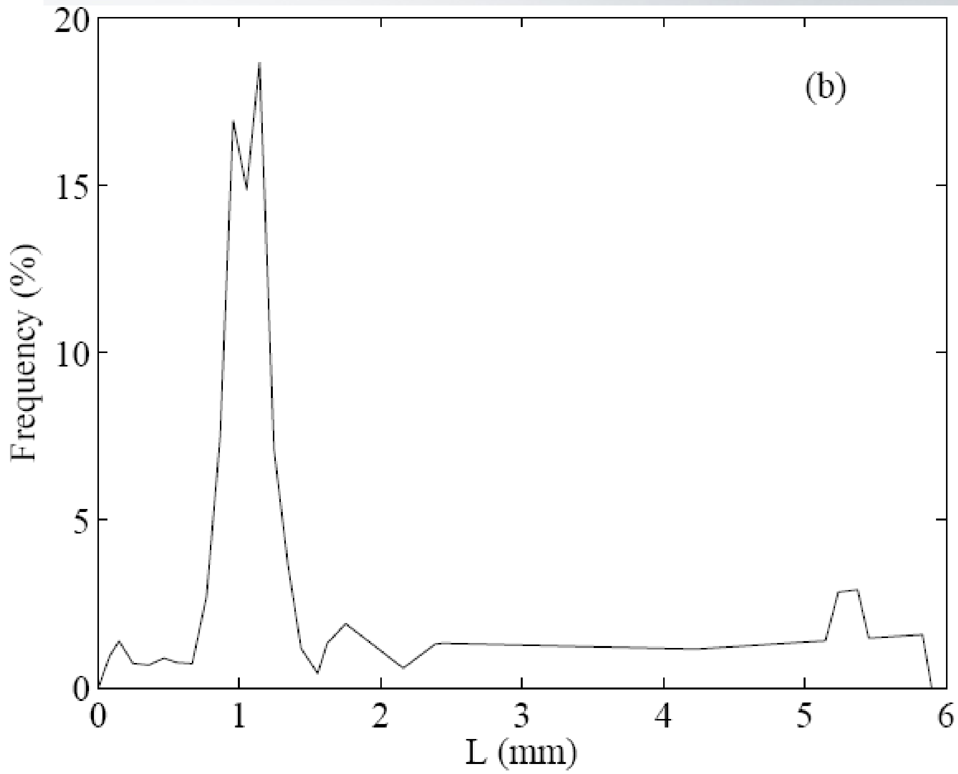
Start ***Finish***

Proof of Principle

Centrifuge Test with a Yield Stress Fluid

Bidisperse Nylon Particle Suspension (separation based upon length)



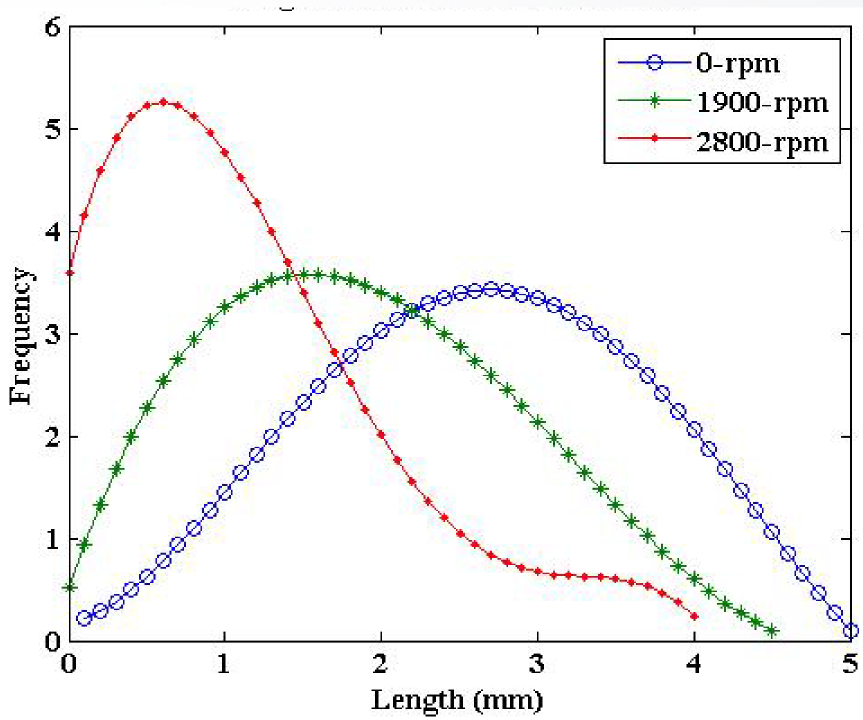


Start

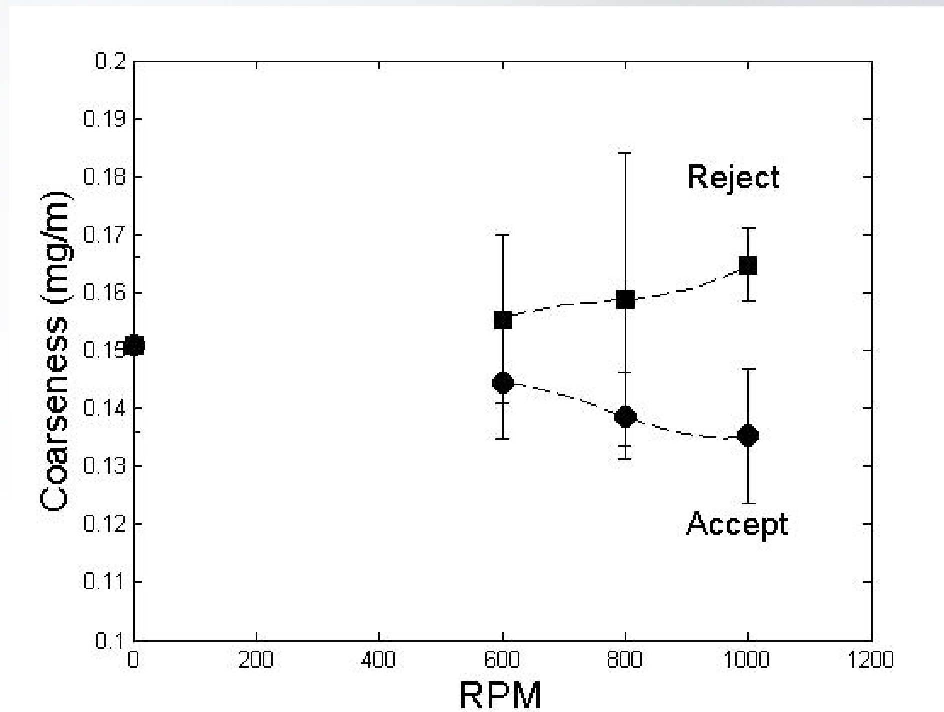
Finish

Papermaking Fibres

0.1 % SBK pulp
0.1% Carbopol 940 (pH 7)



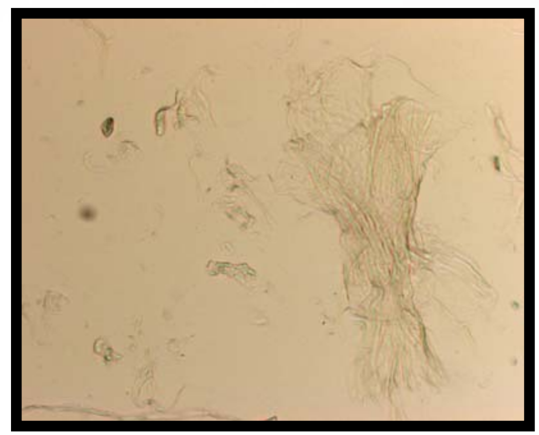
Length Separation



Coarseness Separation

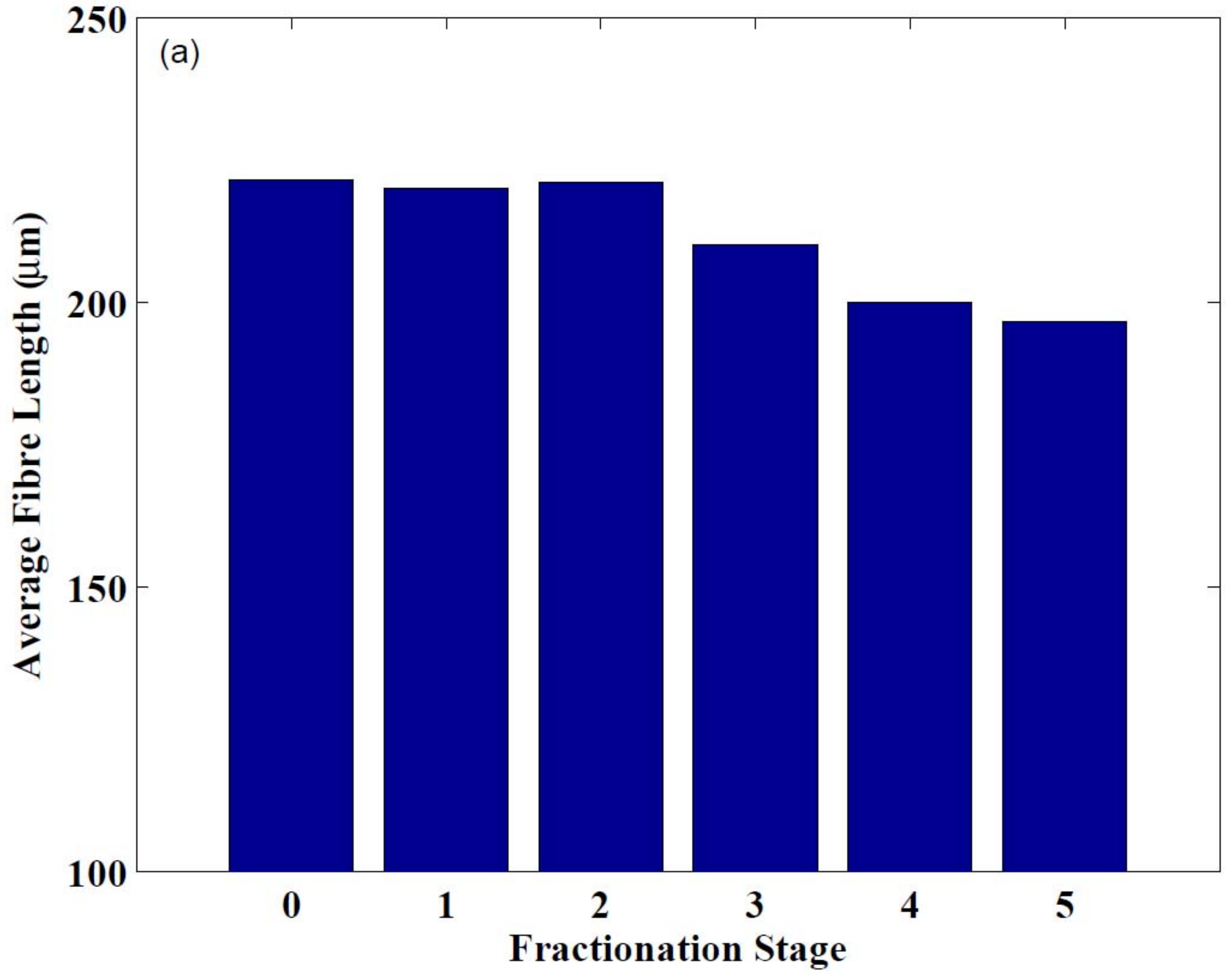
- **Part 3. Demonstration Separations**

Goal:
•Purification of commercially available MFC
•Reliable manufacturing method



Comparison of Methods

Pressure Screen

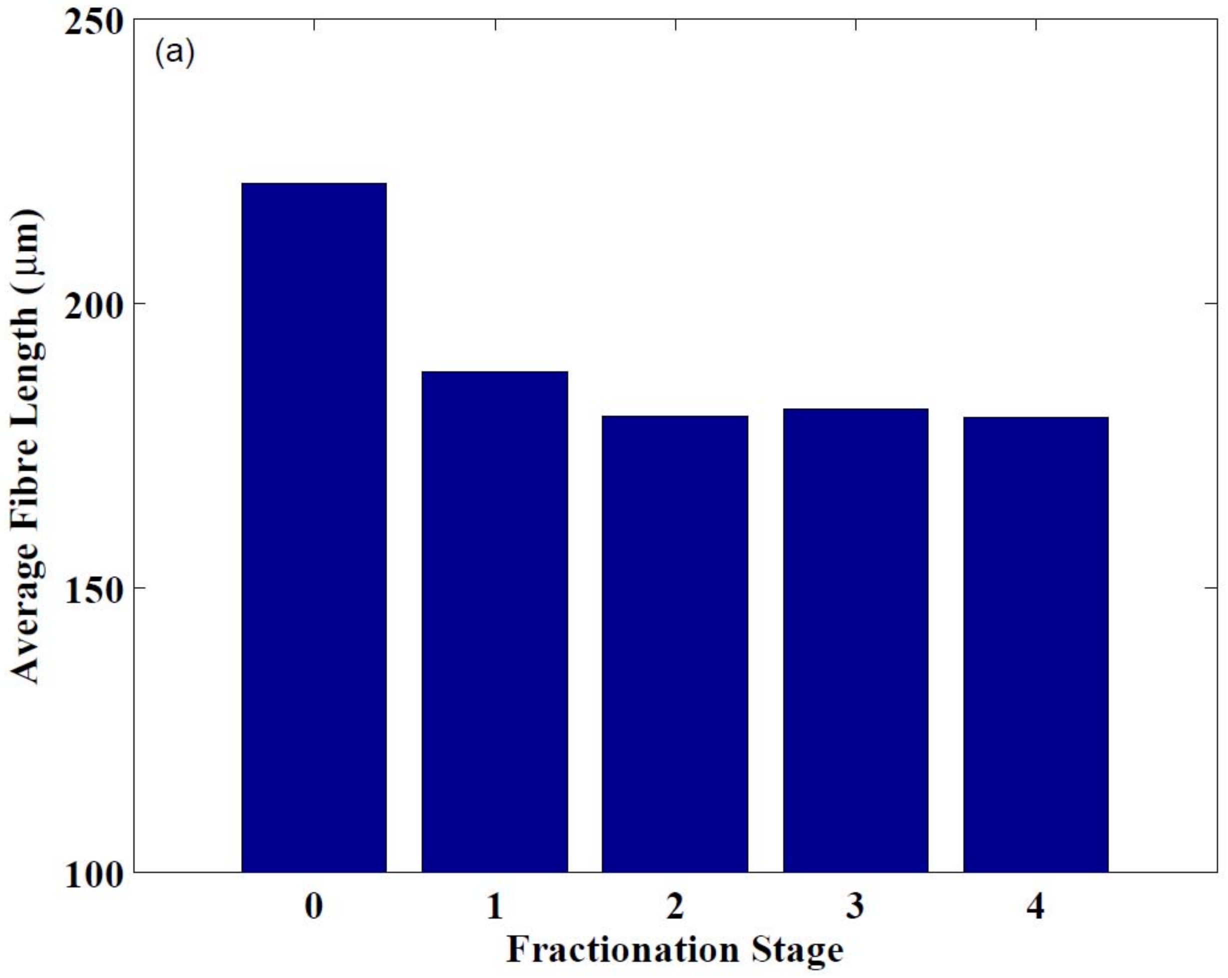


Conditions

- *0.1% MFC shown*
- *Tested at : 0.1,0.3,0.5,0.6 and 1%*
- *Metso FS-03*
- *rotor speed 3500 rpm*
- *Reject ratio 60%*
- *Screen sizes:*
  - *Stage 1 : 0.13 mm*
  - *Stage 2 : 0.09 mm*
  - *Stage 3-5: 0.06 mm*

Comparison of Methods

Hydrocyclone

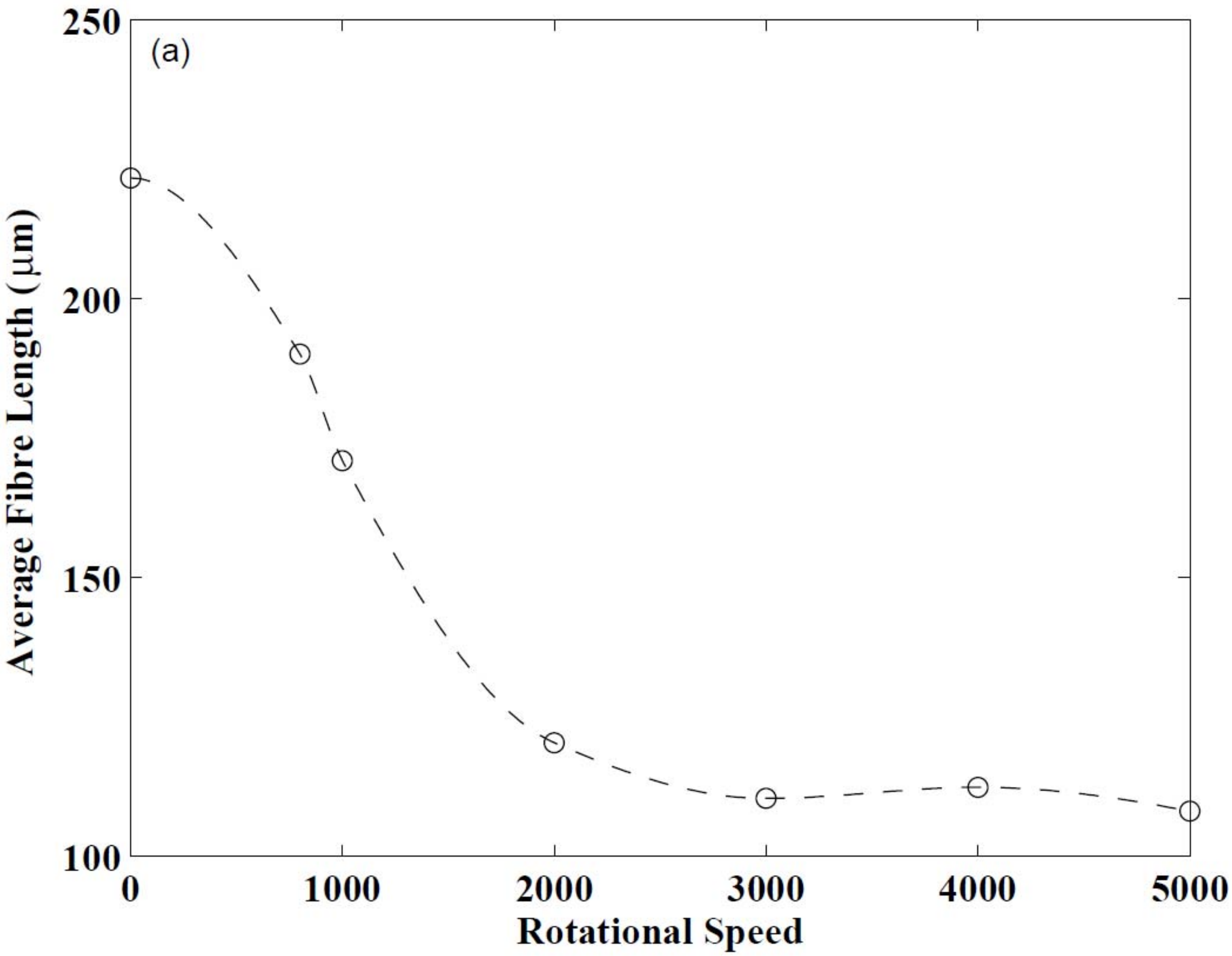


Conditions

- ***0.2% MFC shown***
- ***C-1201 Microspin polypropolyene hydorcyclone***
- ***10 mm diameter***
- ***5 bar, 4.2 lpm***
- ***``accept" refractionated***

Comparison of Methods

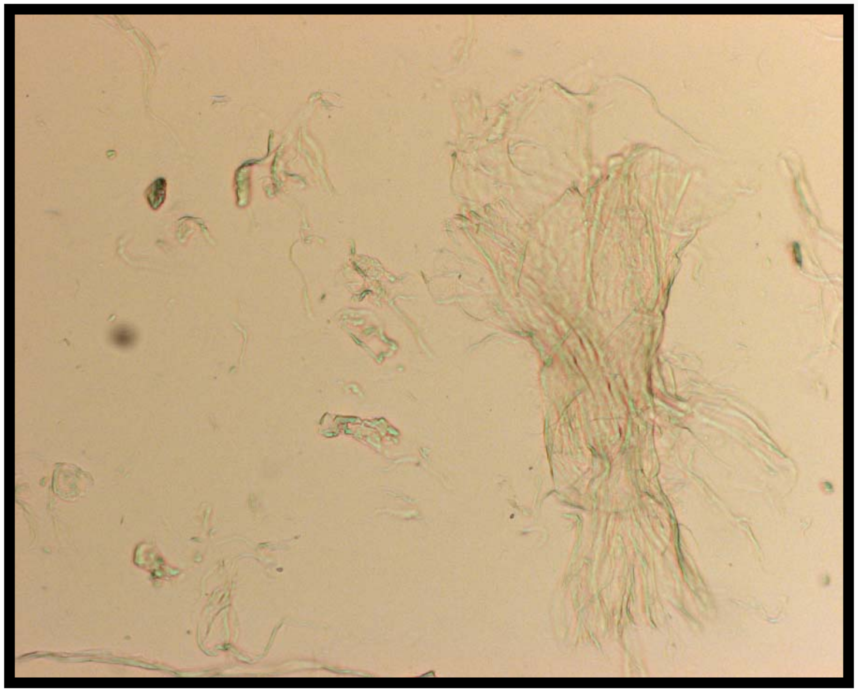
Gel Technique

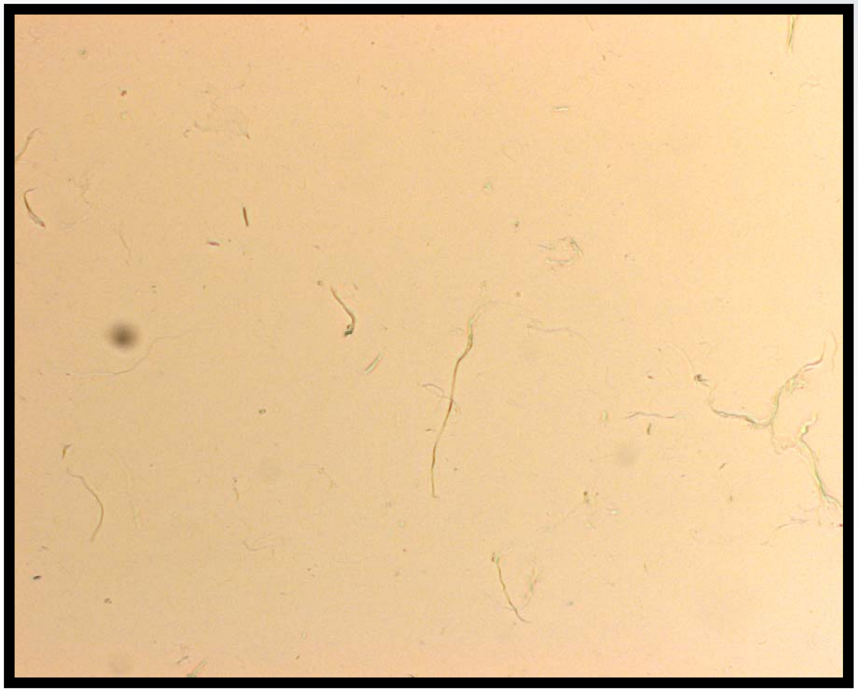


Conditions

- ***0.2% MFC shown***
- ***0.16% Carbopol 940 (pH 7)***
- ***MFC***
    - ***0.1, 0.2 0.4 and 0.6%***
- ***Eppendorf 5804 centrifuge***

Optical Microscopy

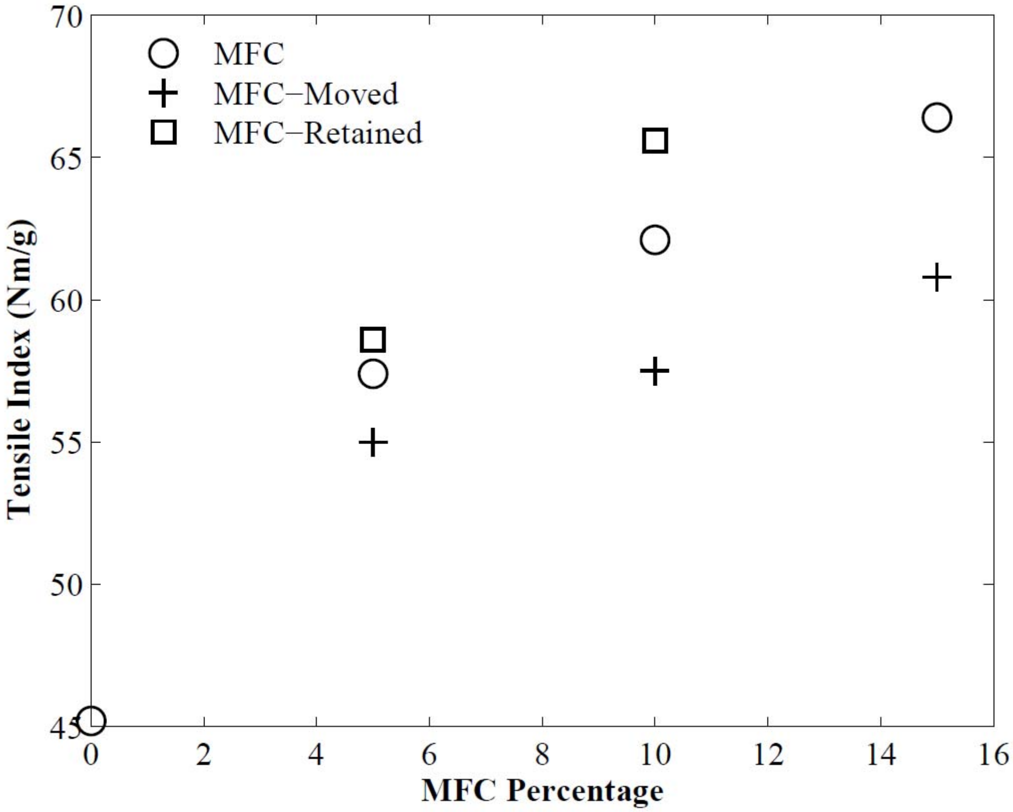


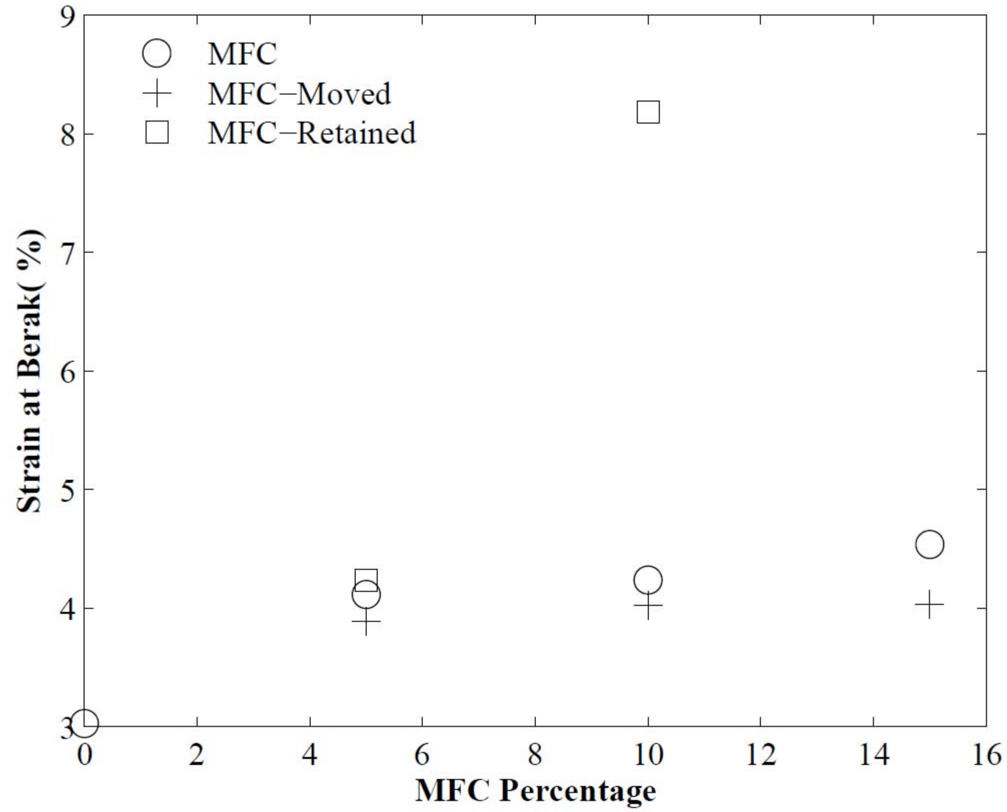


Before

After

Paper Properties





Standard handsheet, 60 g/m², bleached hardwood

Summary

- MFC – purification much different than traditional papermaking suspension
- Novel fluid: yield stress to determine separation based upon specific surface (mass/area)
- Number of demonstration separations outline utility
- Enhanced tensile and strain to break over commercially available material