Investigations of coagulation of cellulose solutions

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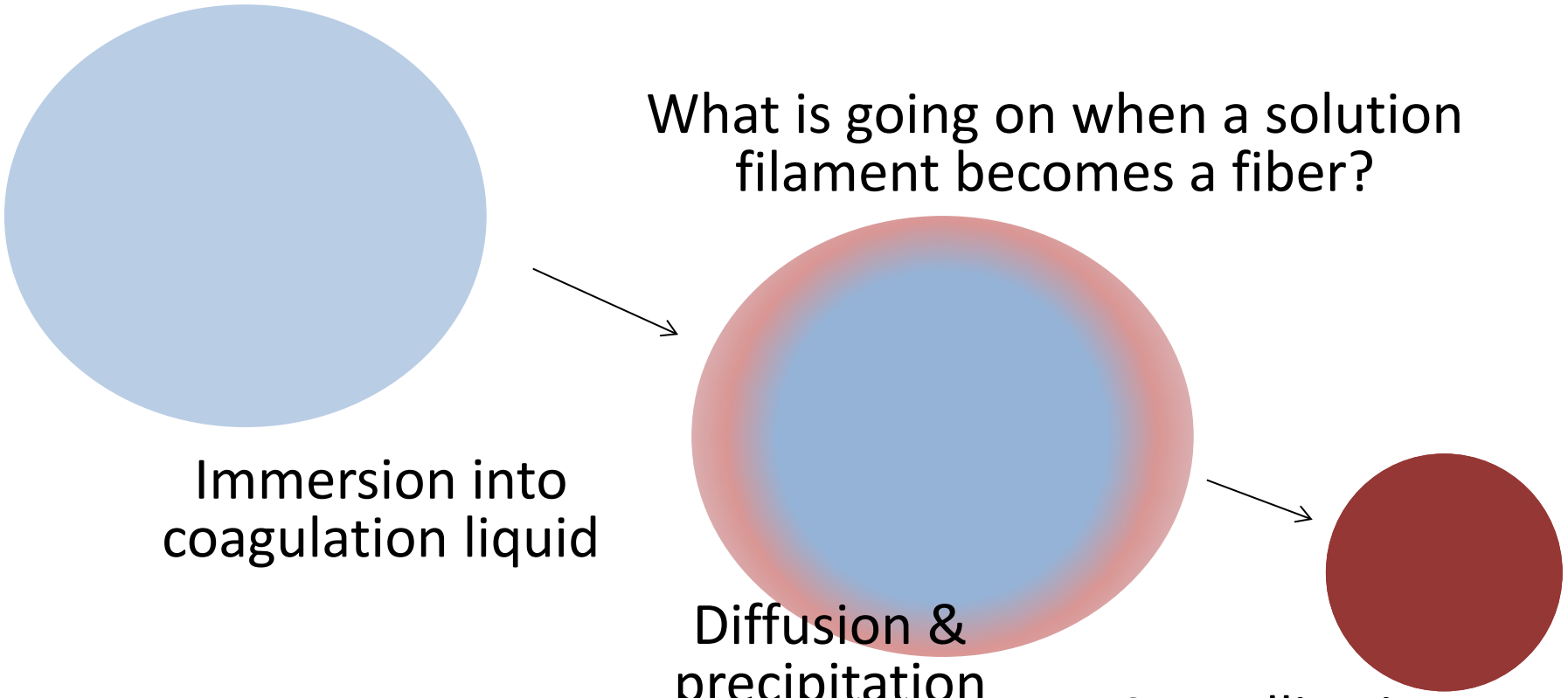
Aim of project:

•Investigating the process of coagulation of cellulose solutions for the purpose of fiber production

The question:

What is going on when a solution filament becomes a fiber?

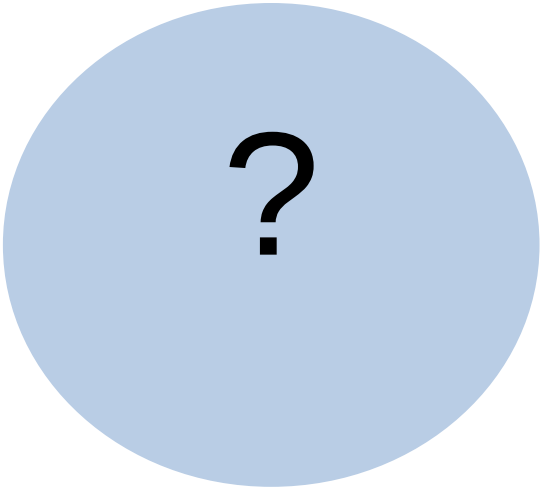


Immersion into coagulation liquid

Diffusion & precipitation

Crystallization, densification & drying

Properties of solution just before coagulation



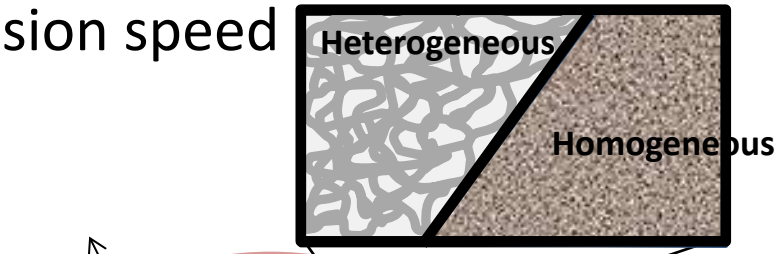
- Type of solvent
- Viscosity
- DP - MW_{η}
- Polymer content
- Macromolecular structure due to flow history

Diffusion & precipitation

Diffusion speed:

•How much chain relaxation is allowed?

Structure: affects diffusion speed

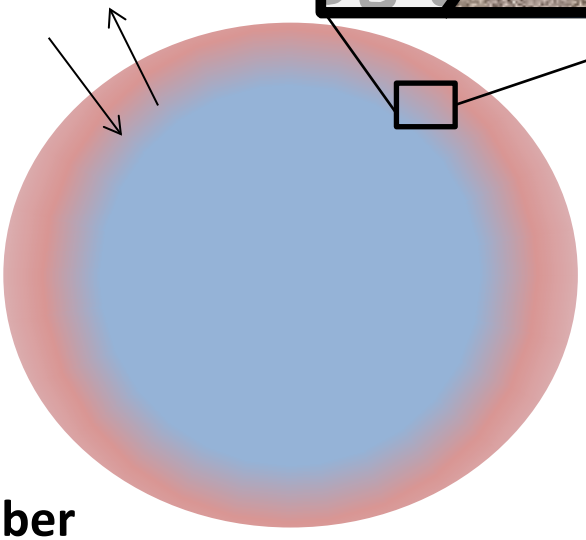


Oriented polymer solution

Oriented polymer fiber

or

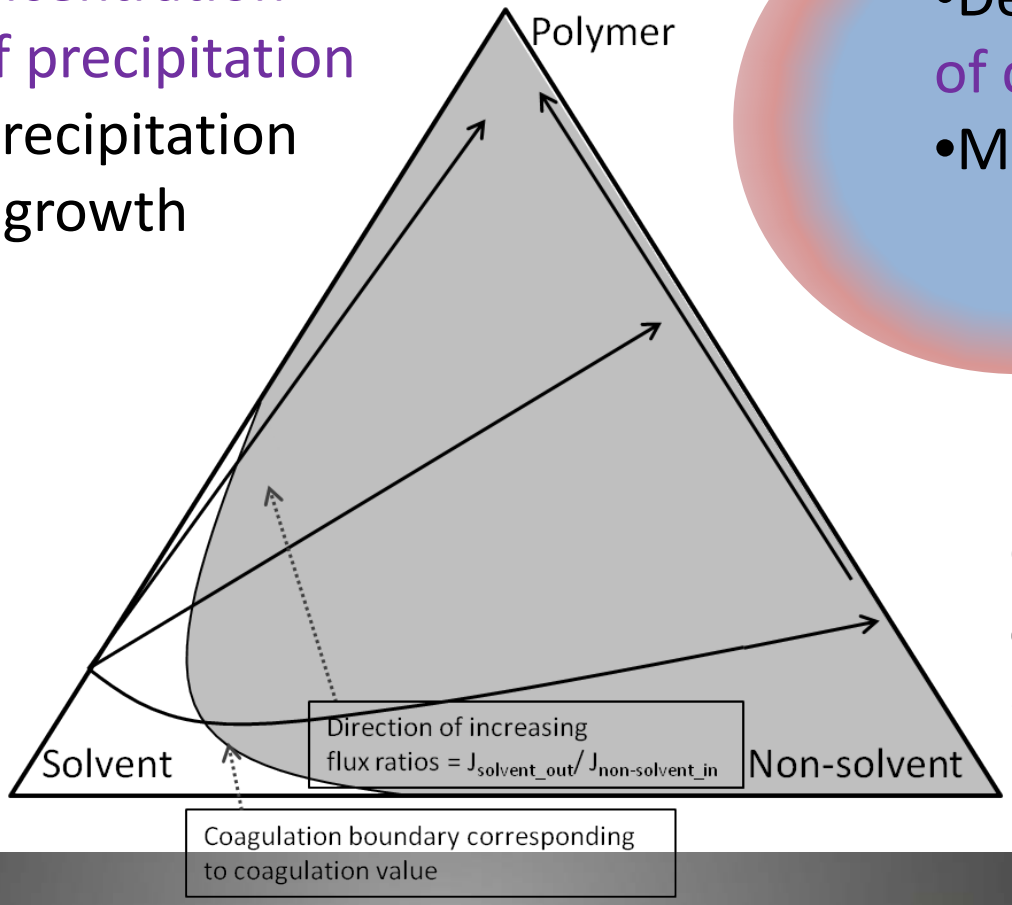
Isotropic polymer fiber



Diffusion & precipitation

Precipitation - Crystallization:

- Critical concentration for onset of precipitation
- Mode of precipitation
- Crystallite growth



Flow ratios out/in:

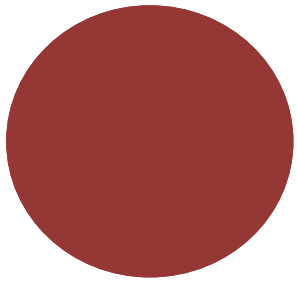
- Determines diluteness of coagulating dope.
- Multiple components

Changing solution chemistry:

- Changed viscosity

Properties of final fiber

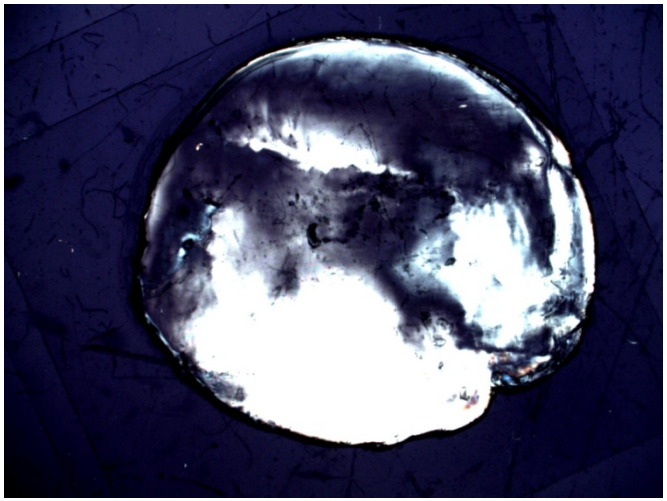
- Macromolecular orientation
- Cross section
- Porosity (size and volume ratio)
- Density
- Crystallinity
- Crystallite dimensions
- Wet cohesion



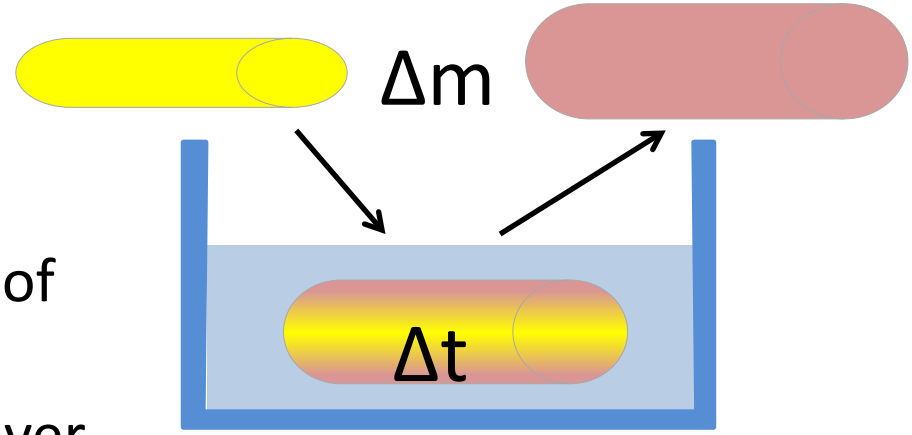
Initial experimental work

•Dissolving pulp and 1,3-ethyl-methyl-imidazoleum acetate (EmimAc) are mixed to a 25wt%, clear solution

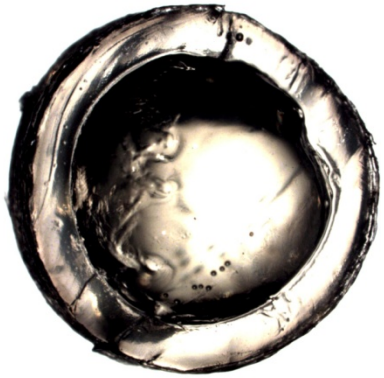
•It is then diluted by methyl-imidazole (MIM) to achieve compositions of (cellulose:EmimAc:MIM): (5:15:85), (10:30:60), (15:45:40)



Measurements

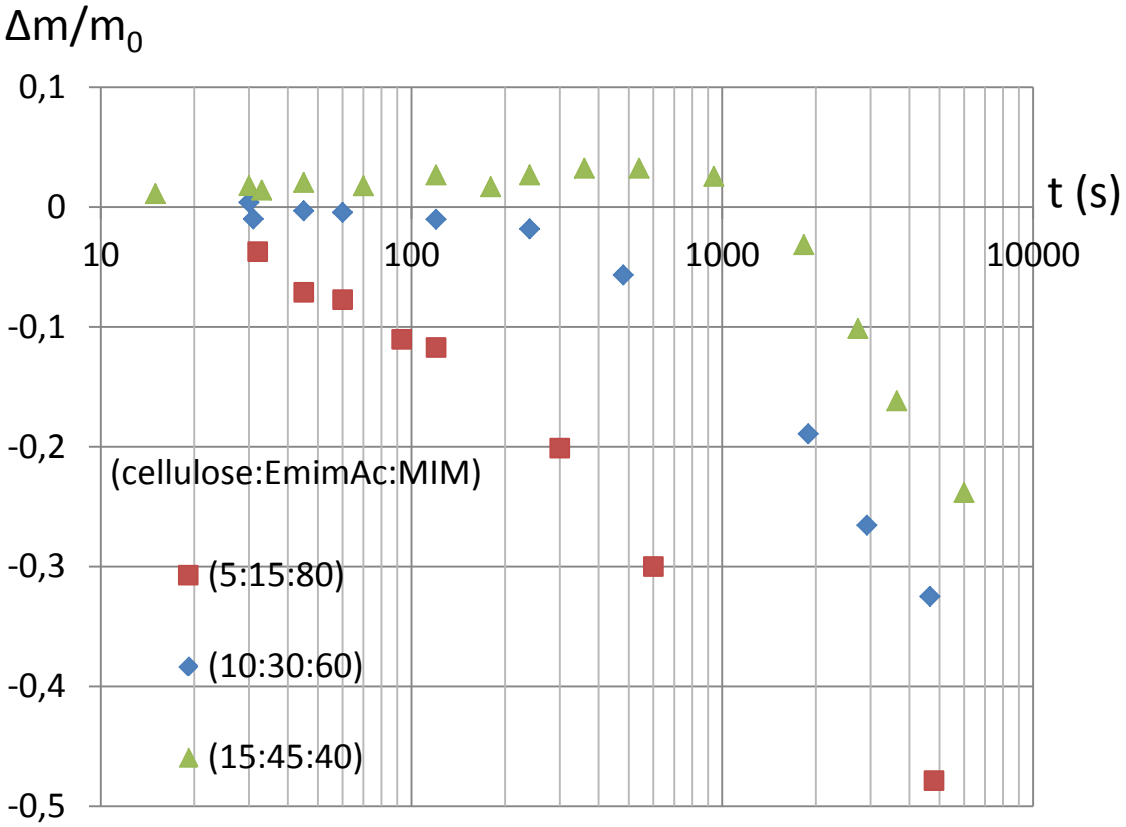


- Measurements of mass uptake/loss during coagulation of rods in water
- Measurement of coagulated layer thickness
- Measurements of solvent components in the bath (by conductivity and UV-absorption techniques)



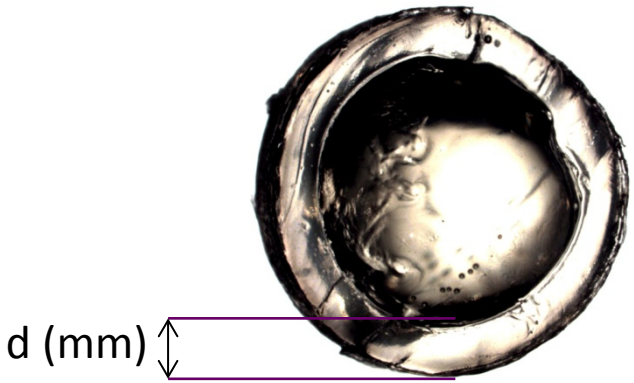
Relative mass gain over time

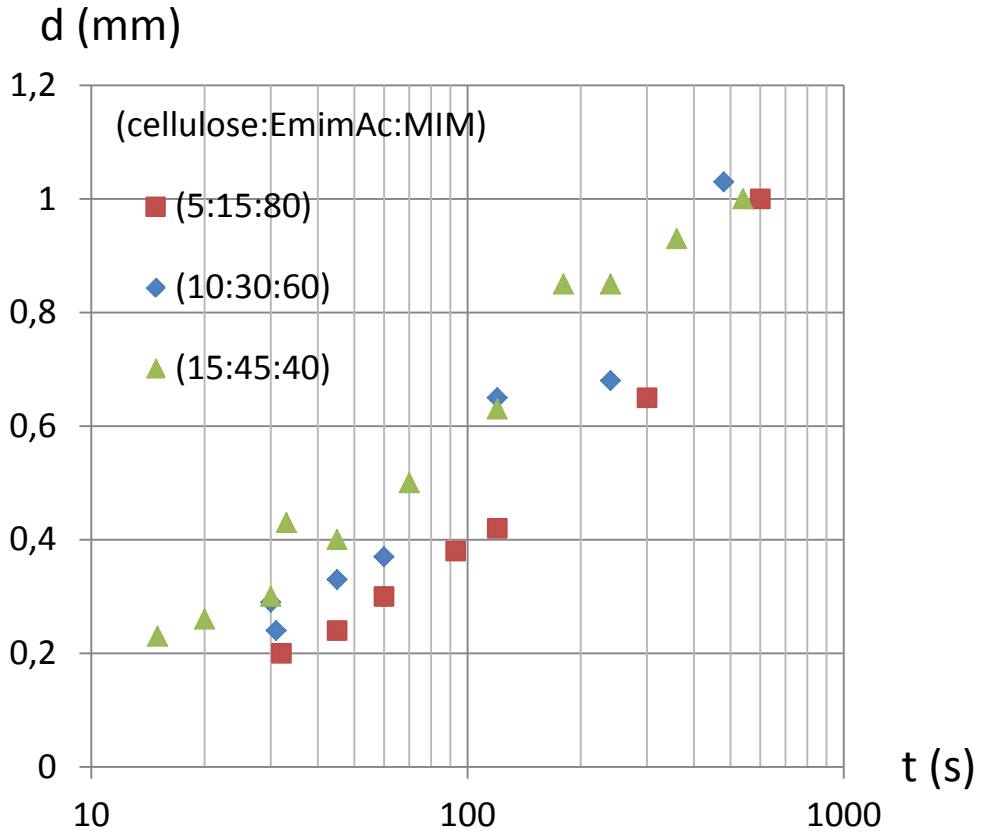
- Large differences in the initial stage
- But once rods are coagulated throughout, all lose mass at comparable pace



Coagulated skin thickness d(t)

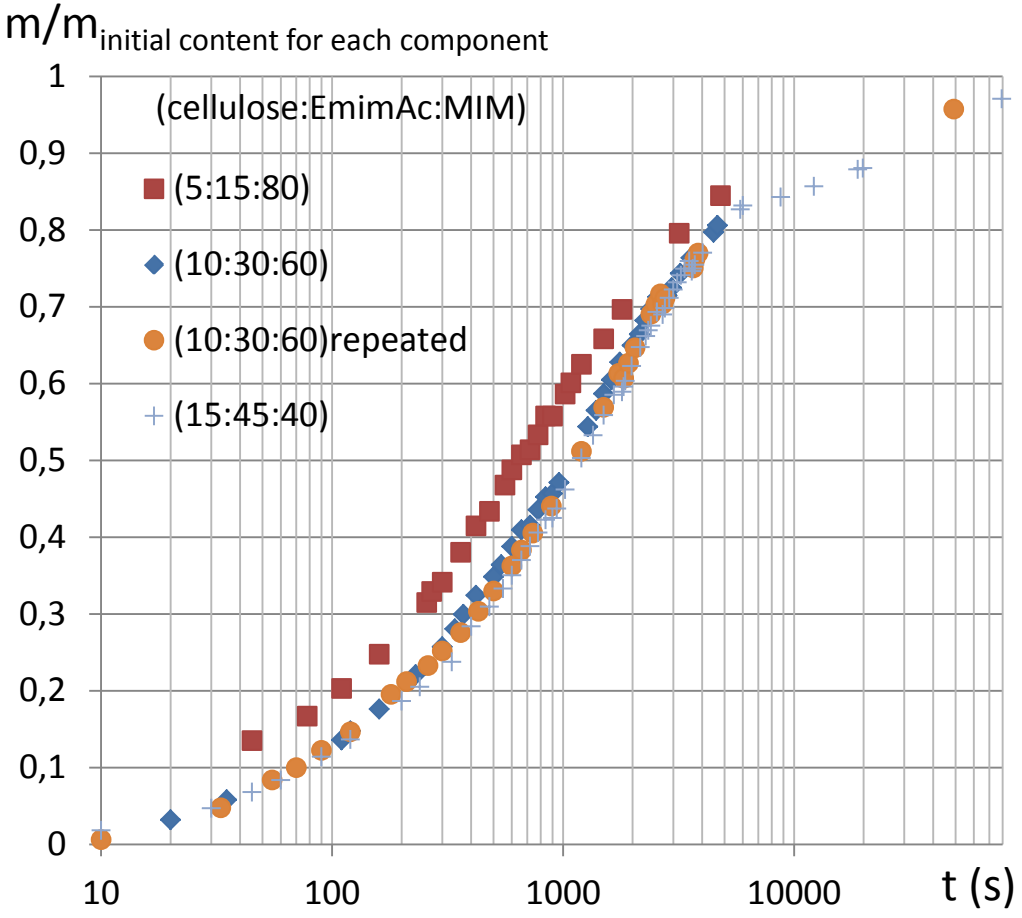
- Dilute solutions coagulate more slowly
- For long times the difference is smaller





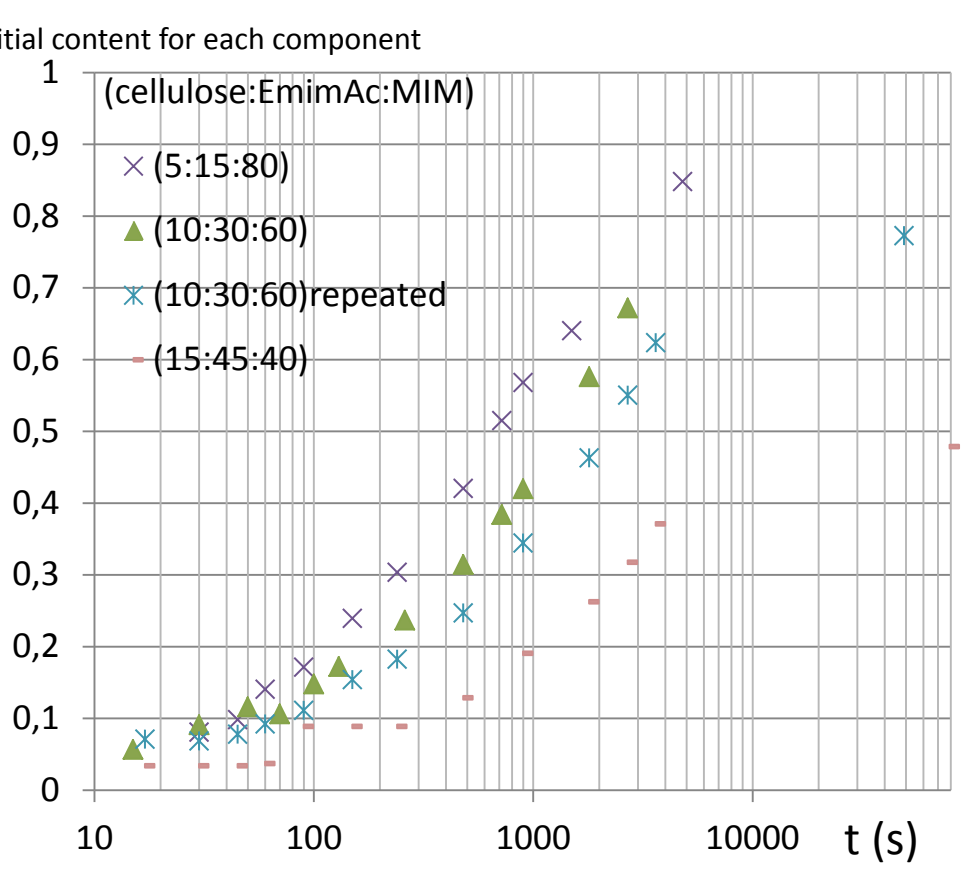
EmimAc diffused out

- Small variations in rate of diffusion as a function of composition
- Relatively complete wash-out from samples although thick (4-5mm)



MIM diffused out (UV-absorption)

- The UV-absorption measurement of MIM is not very reliable (large difference between repeated trials!)
- Surprisingly low total wash-out!
- An EmimAc signal partly superposes with the MIM-signal, particularly disturbing for low concentrations.
- Method must be revised



Future work

- Widen matrix (20:60:20)&(25:75:0)
- Spin fibers to compare fiber properties against data from coagulation experiments
- Compare different coagulation liquids (alcohols, acetone, water) in addition to solution compositions
- Determine critical concentrations of non-solvent for coagulation onset
- Other solvents

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You

for kind attention


Vi arbetar på vetenskaplig grund för att
skapa industrinytta.
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