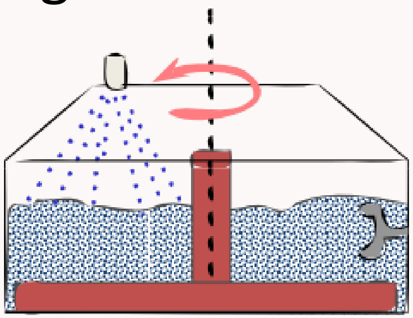
Linking granulation performance with residence time & liquid distributions in twin-screw granulation

Ashish Kumar

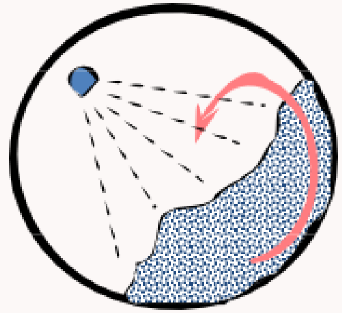
LPPAT-lab meeting

Traditional to new granulation method

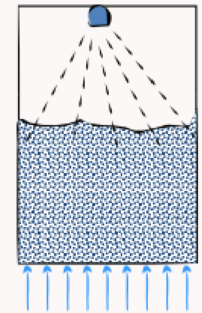
High-shear mixer



Drum



Fluidised-bed

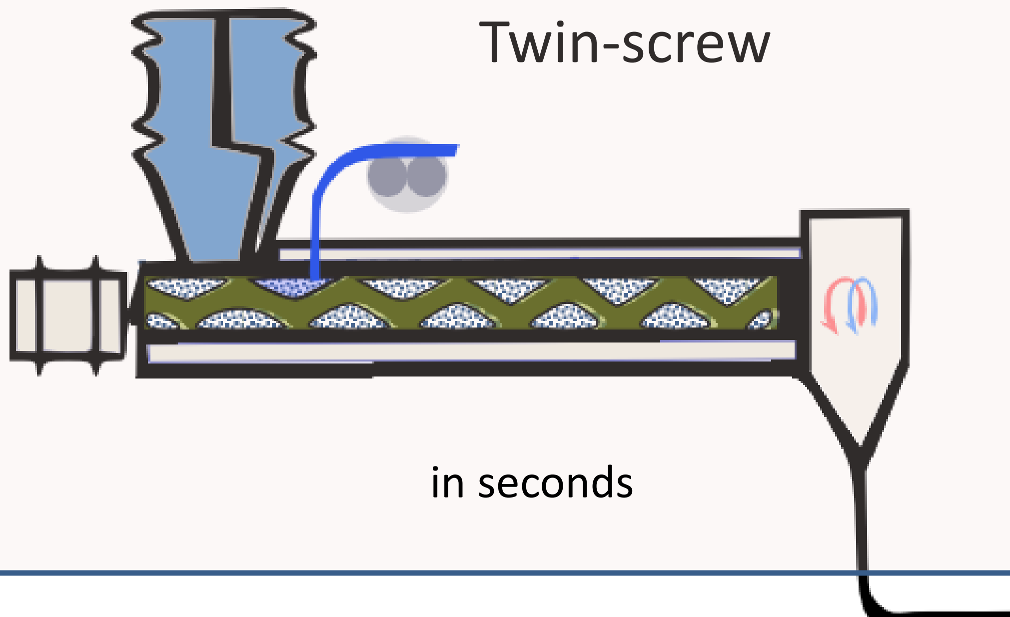


From minutes to hours

Batch

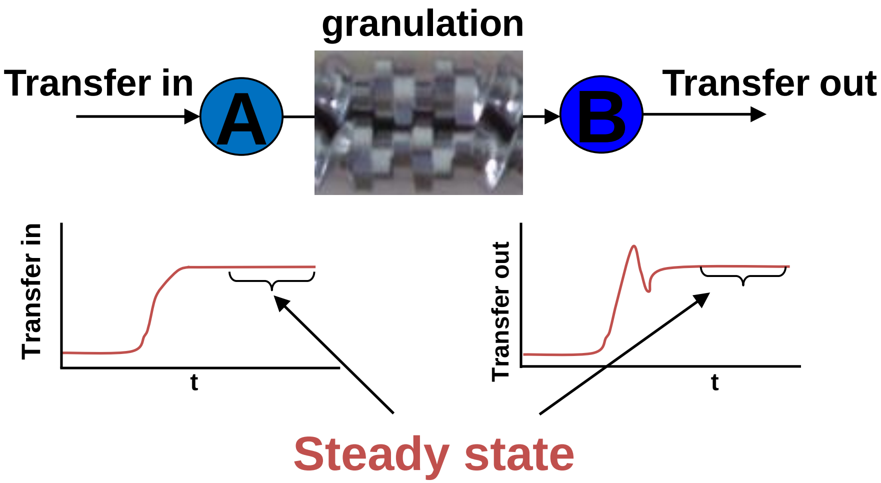
Continuous

Twin-screw



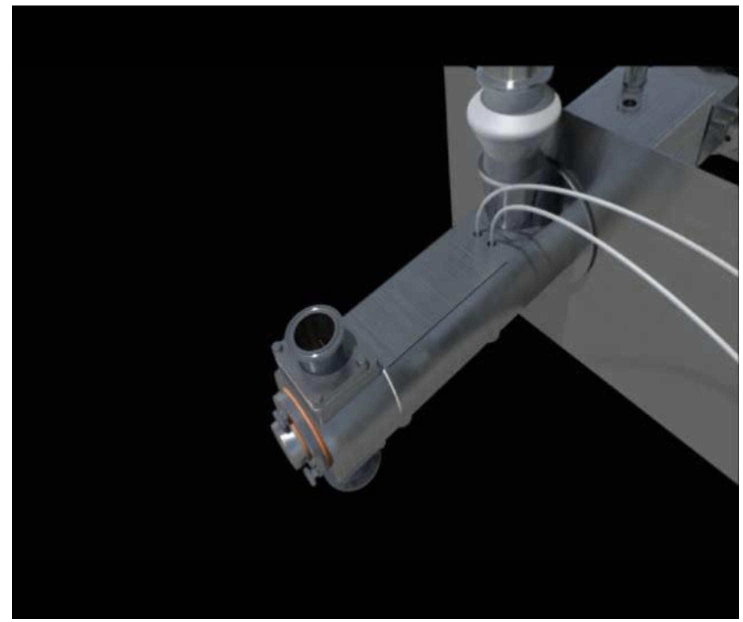
in seconds

At appropriate conditions, granulation is in steady state



$$\textit{transfer in} \approx \text{constant} \approx \textit{transfer out}$$

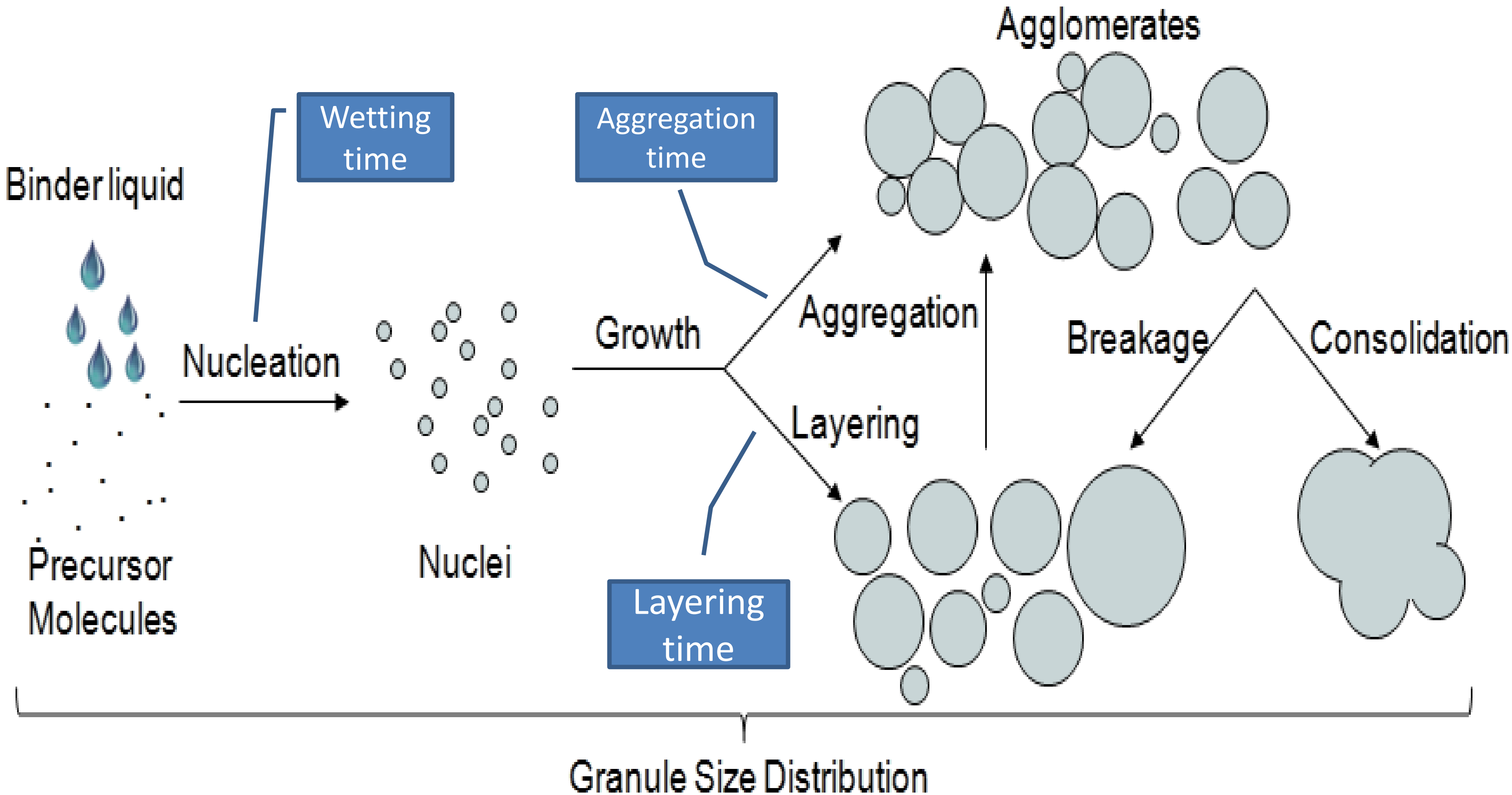
$$\frac{d[P_m]}{dt} \approx 0 \approx \frac{d[G_m]}{dt}$$



Two key implications

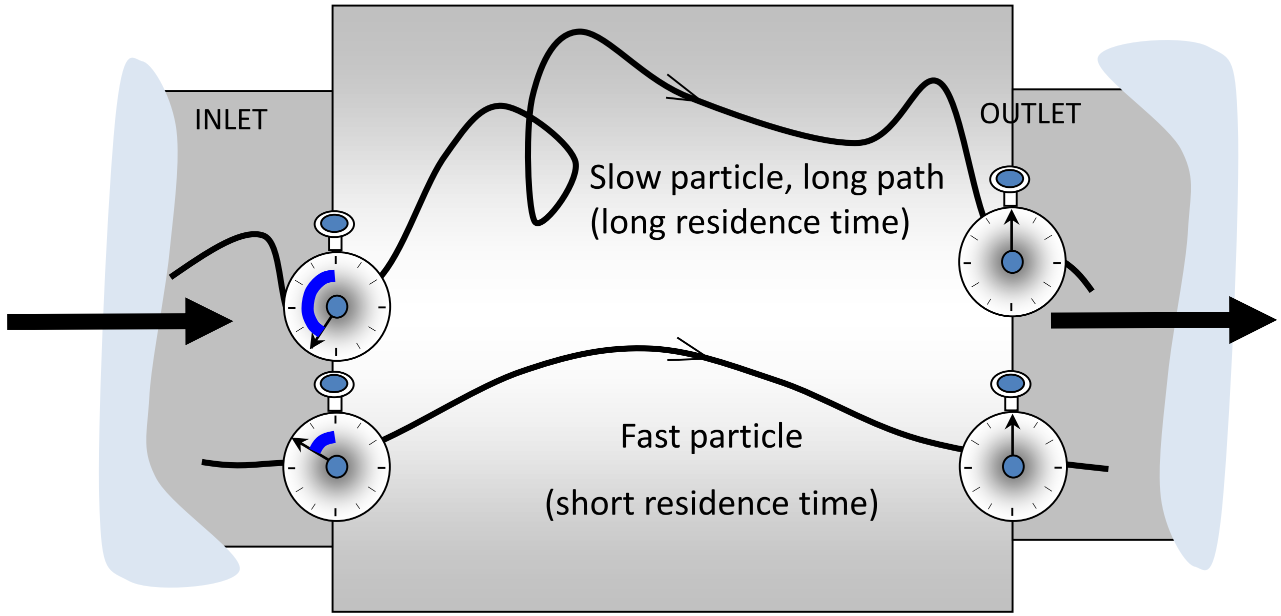
1. Fluxes are roughly constant (Dynamics are transient)
2. Same amount of time is to complete all sub-processes

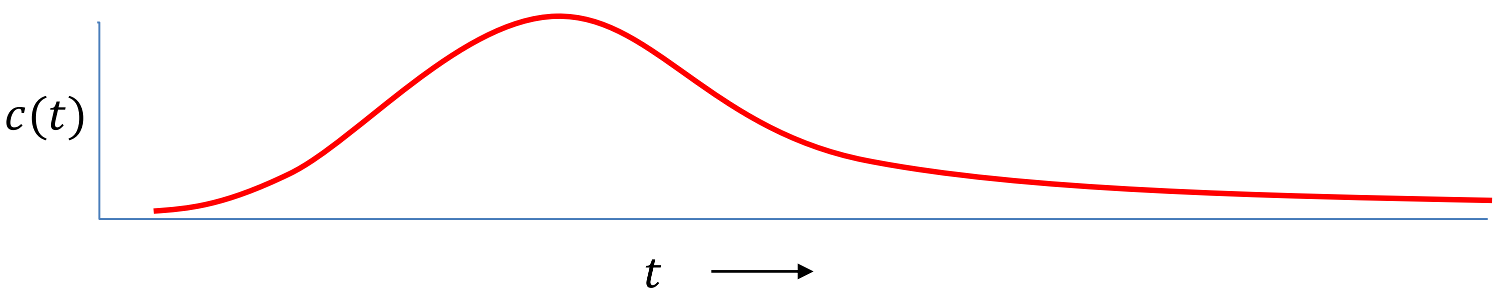
High Shear Wet Granulation involves different rate processes



Having many time-scales is challenging

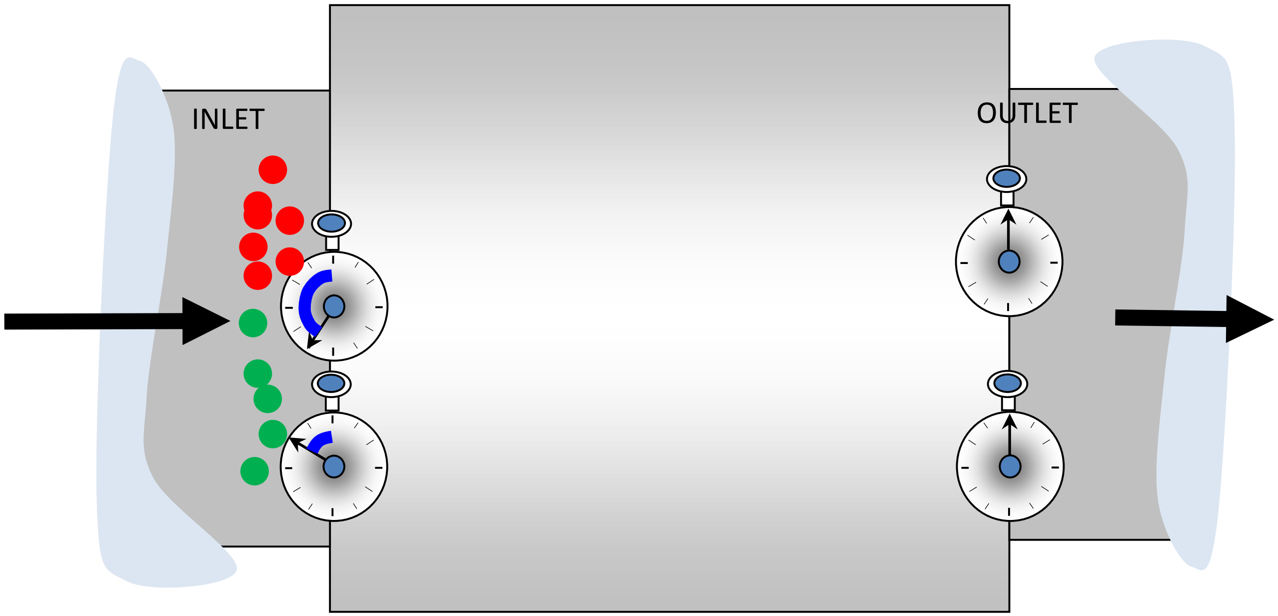
Residence time-scale

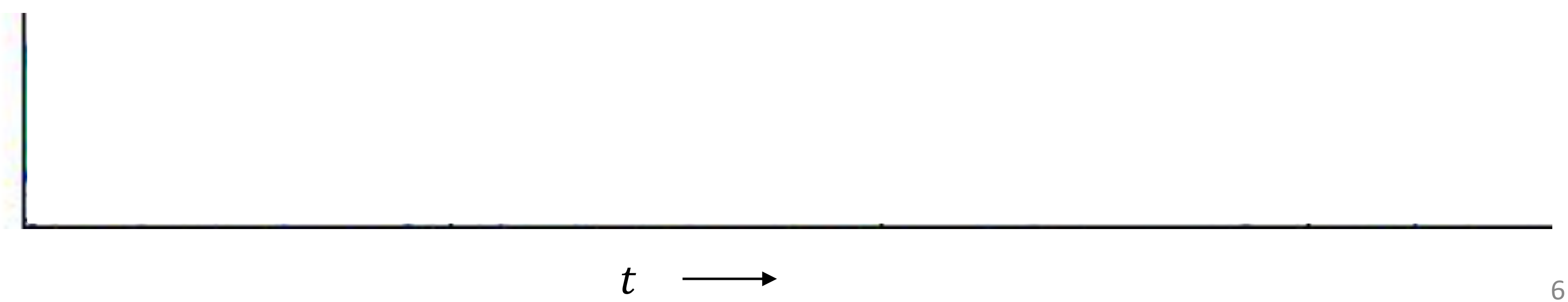




Having many time-scales is challenging

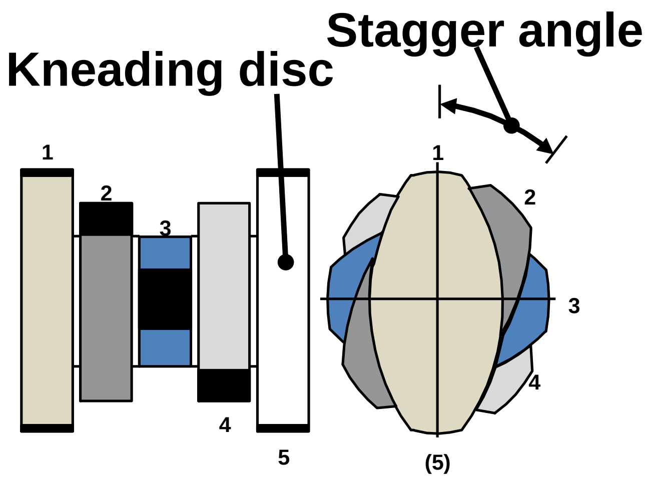
Mixing time scale





Residence time and moisture distributions effect on the granulation performance

Screw Configuration
- Number of kneading discs (4, 6, 2x6)
- Stagger angle (30°, 60°, 120°)



Process parameters
- Material throughput (10-25 kg/h)
- Screw speed (500-900 rpm)
- Liquid-to-solid ratio (6-8%)

Fines
< 150 µm

Yield fraction
> 150 to <1400 µm

Oversized
> 1400 µm

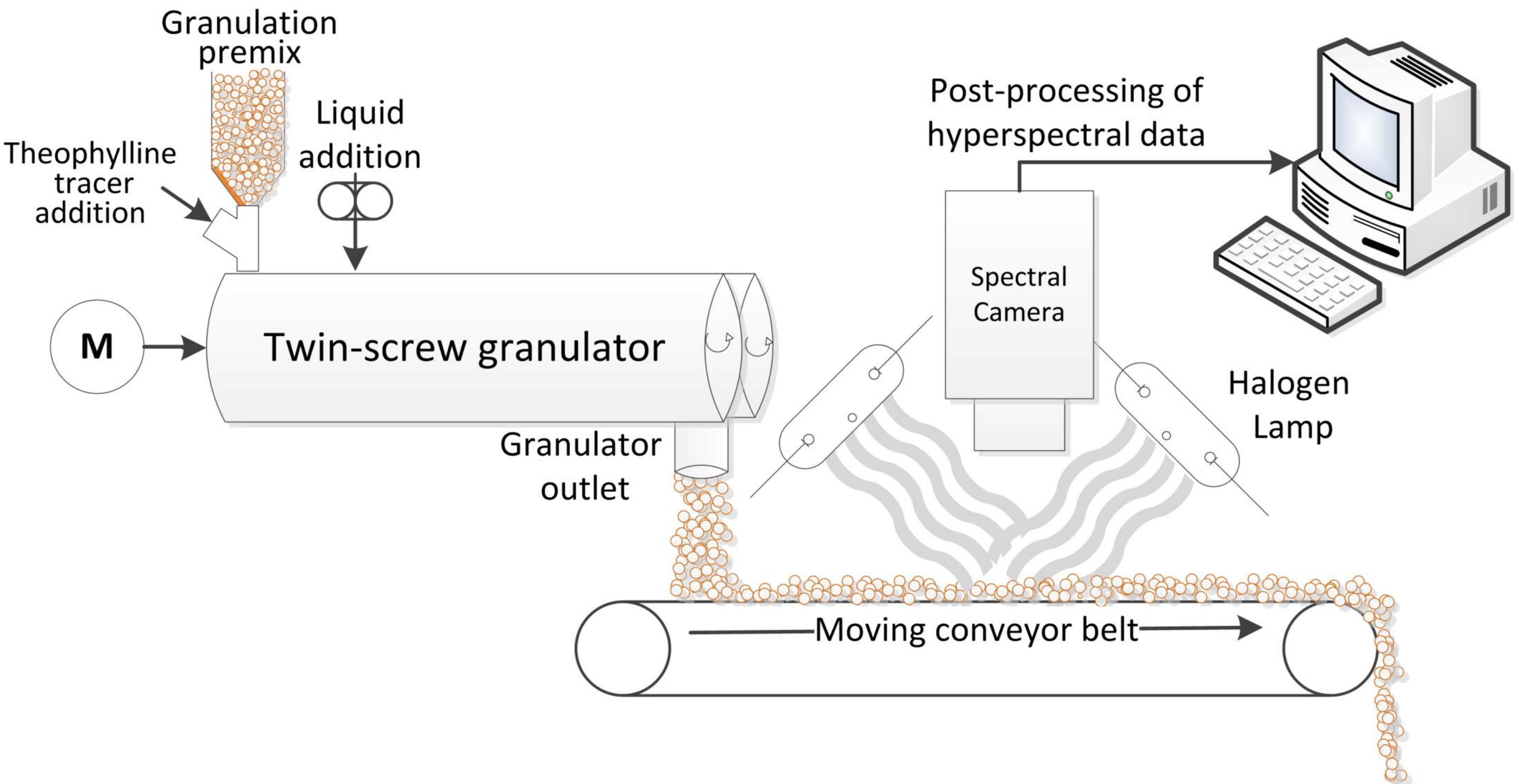
Analysis of distributions in twin-screw granulation

Measurement by distributions

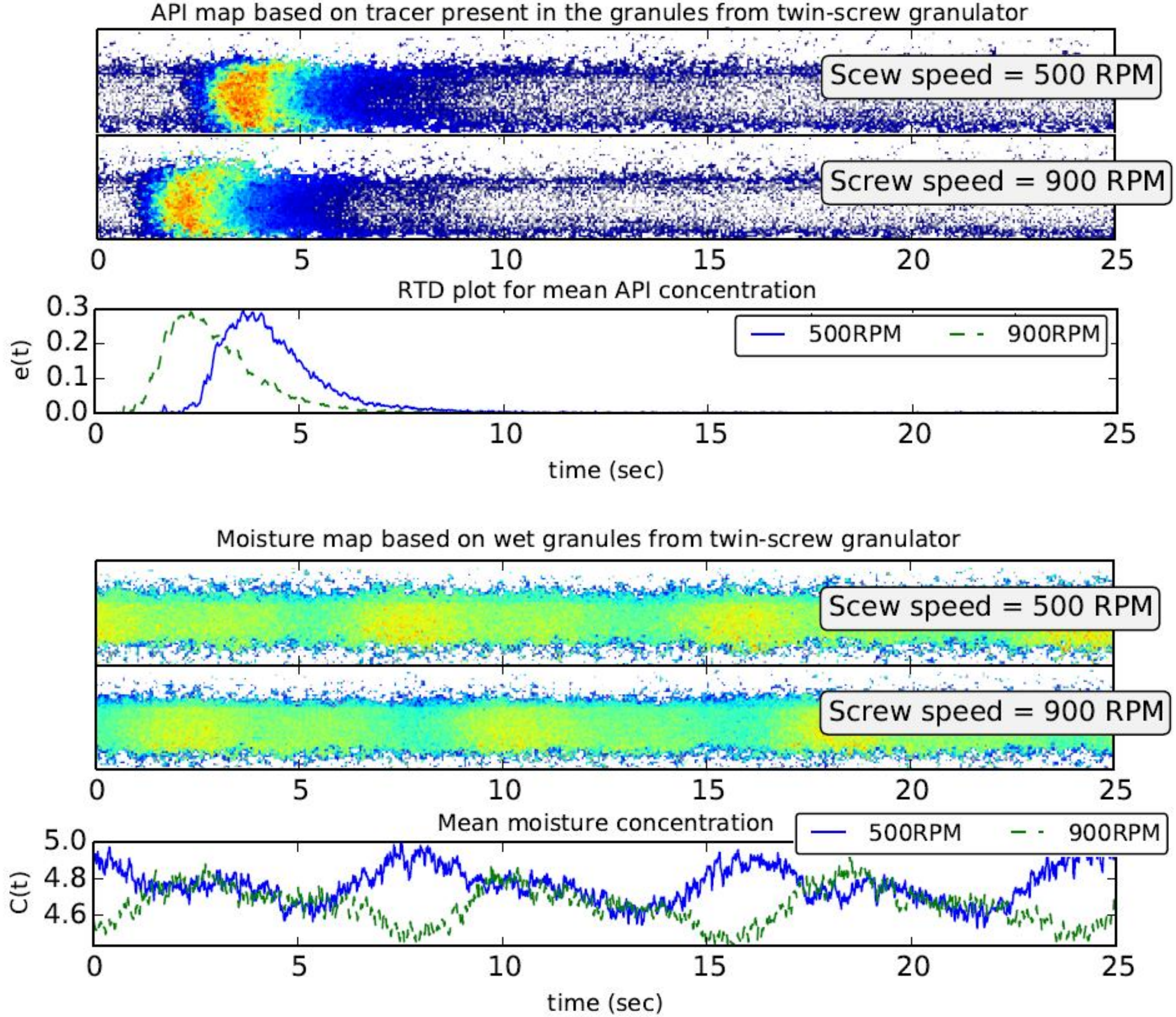
Results

Summary

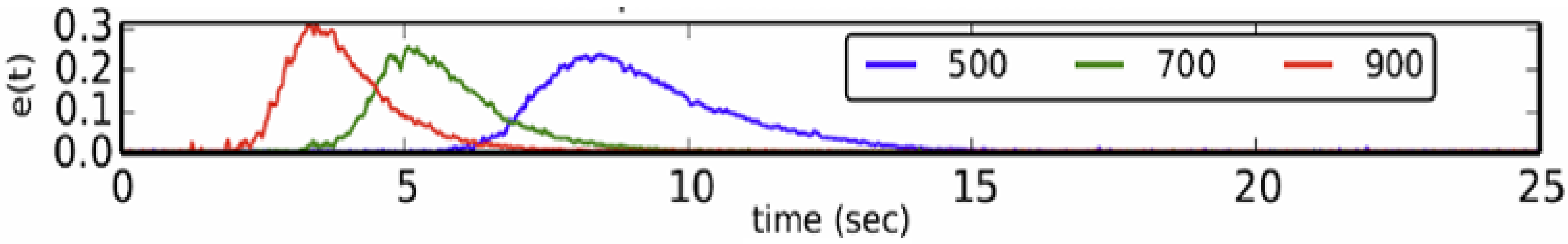
Tracer concentration in granules measured by NIR chemical imaging



Tracer maps used to measure distributions



Qualitative assessment of the RTD profiles



$$\tau = \frac{\int_0^\infty t \cdot e(t)dt}{\int_0^\infty e(t)dt}$$

Mean residence time , τ
(a measure of the mean of the distribution)

$$\sigma^2 = \frac{\int_0^\infty (t-\tau)^2 \cdot e(t)dt}{\int_0^\infty e(t)dt}$$

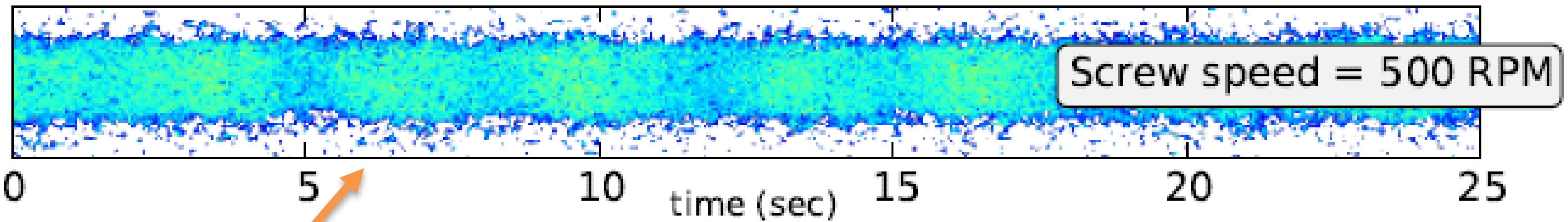
Variance, σ^2
(width of the distribution)

$$\text{Pe} = \frac{UL}{D}$$

Péclet Number, Pe

$$\left(\frac{\text{Rate of axial transport by convection}}{\text{Rate of axial transport by dispersion}}\right)$$

Qualitative assessment of the moisture maps

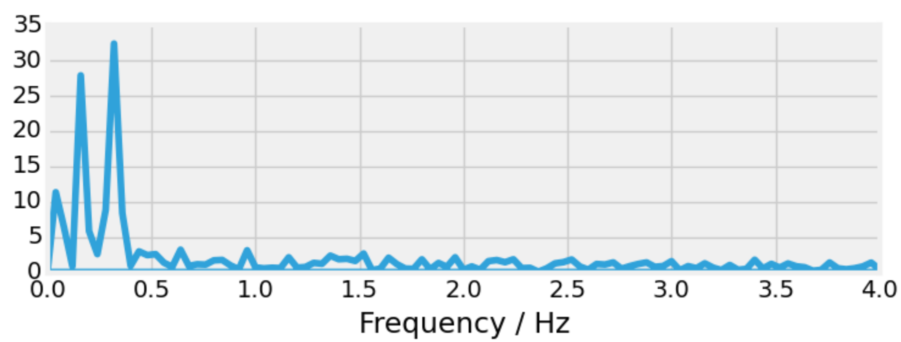


Shannon Entropy based Mixing Index

$$H(X) = \sum_{j=1}^{n} P(X)\log_{200}(1/P(X_j))$$

$$\mathrm{MI} = -\frac{1}{\log_{200}(n)}\sum_{j=1}^{n} P(X_j)\log_{200} P(X_j)$$

FFT to obtain Frequency and amplitude



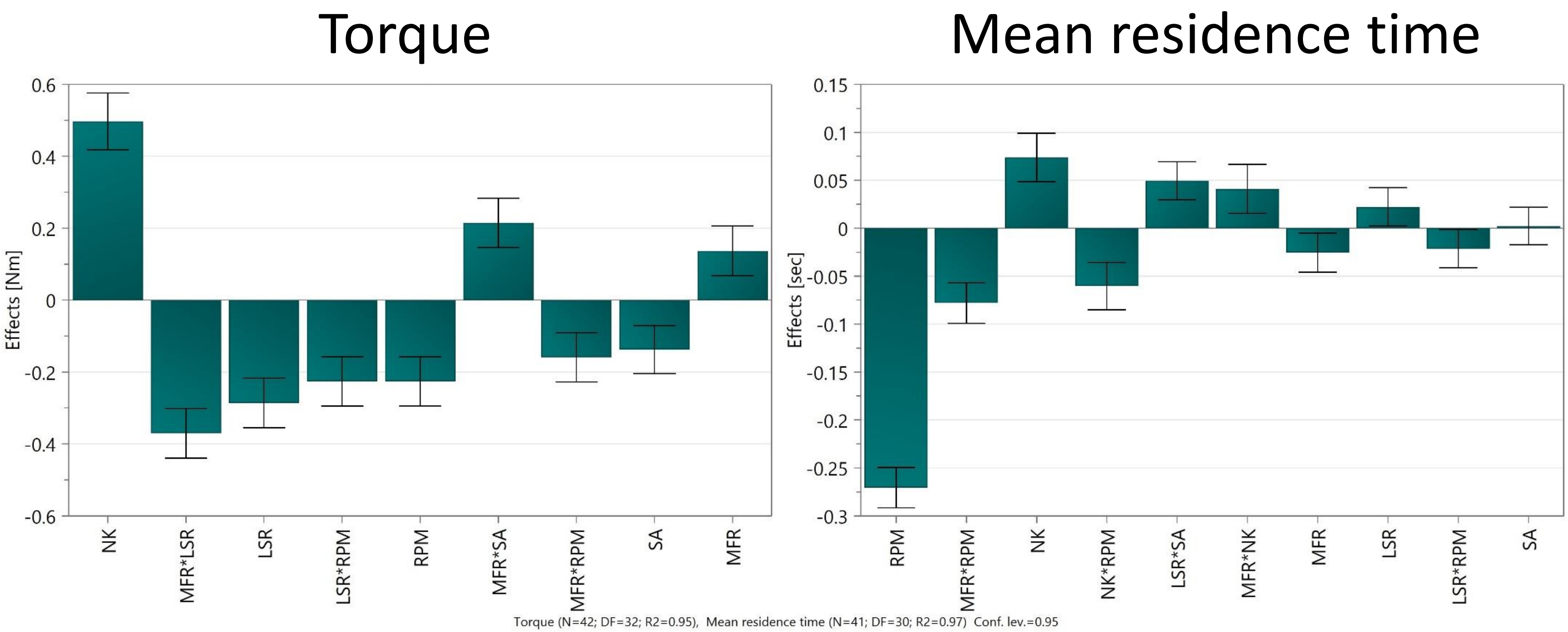
Analysis of distributions in twin-screw granulation

RTD Measurement by Chemical Imaging

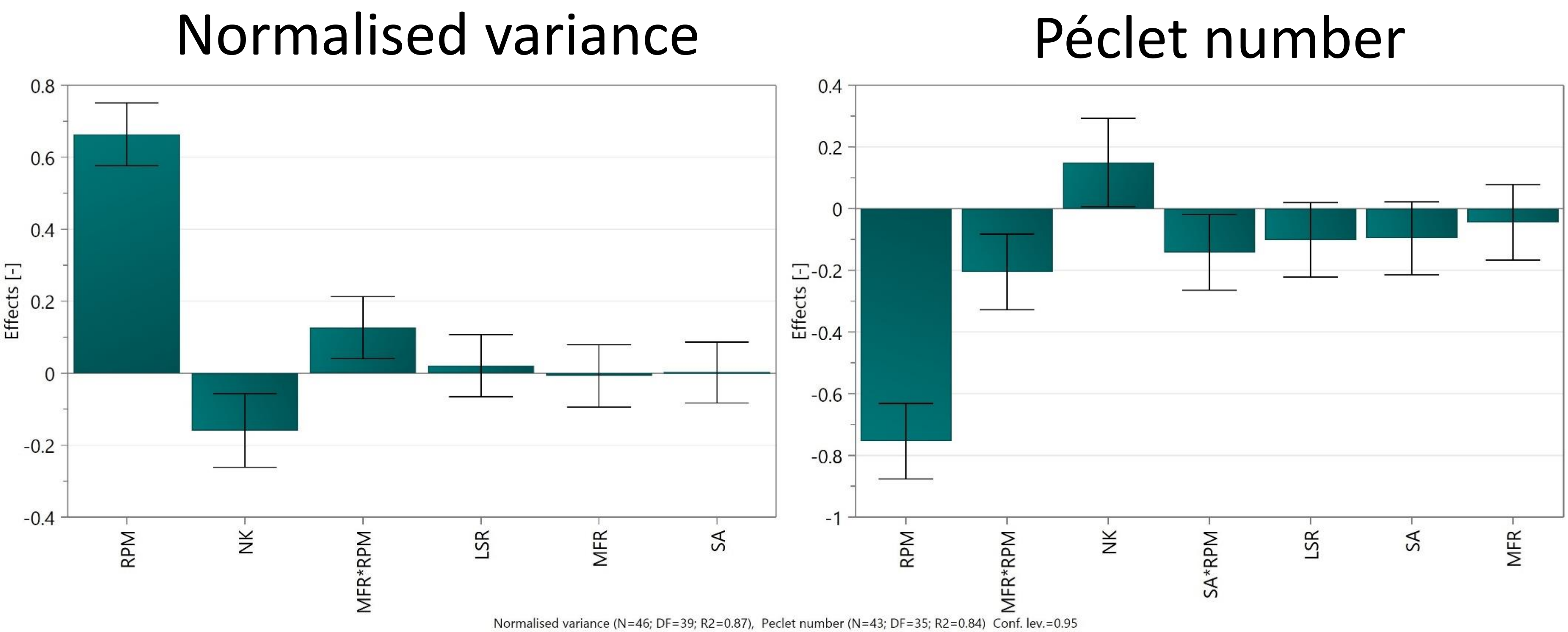
Results

Summary

Increase in L/S lubricates moving parts but flow is sluggish

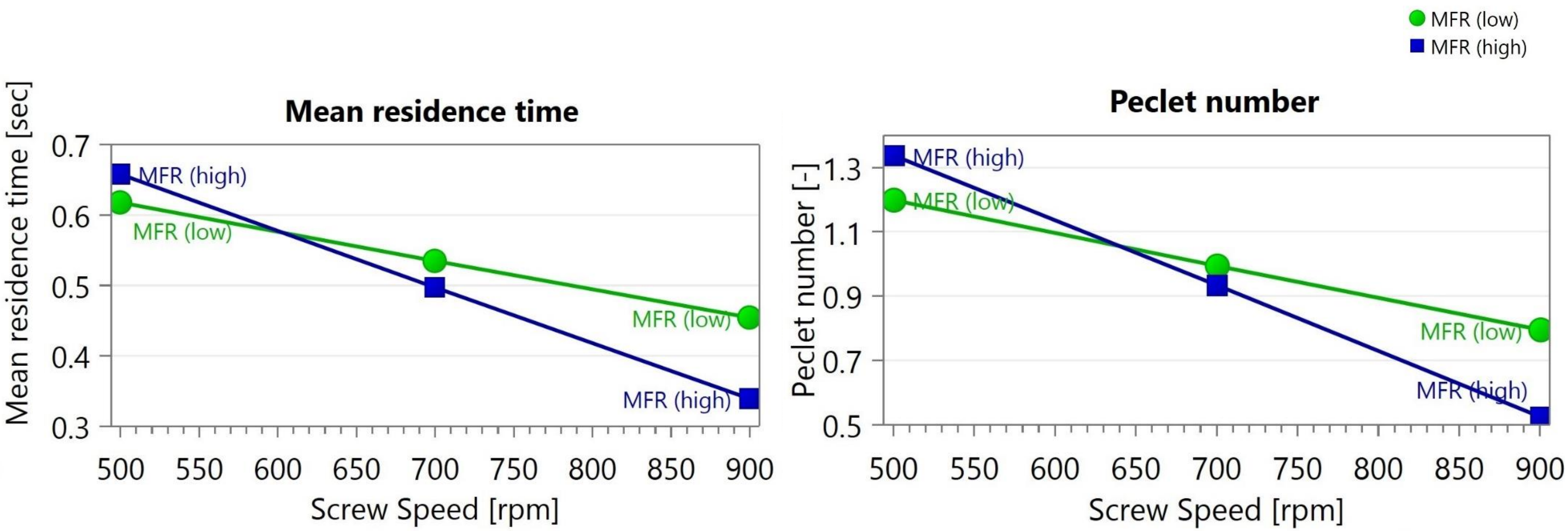


Mixed-flow transport at a high screw speed & plug-flow transport when more kneading discs

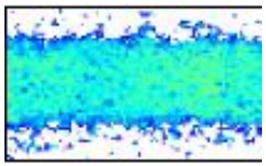


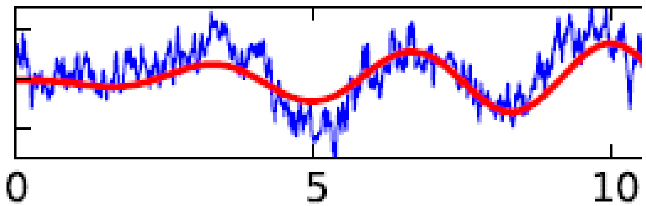
Axial mixing ↑ when Normalised variance ↑ and Péclet number ↓

Throughput force is more dominant at a high screw speed

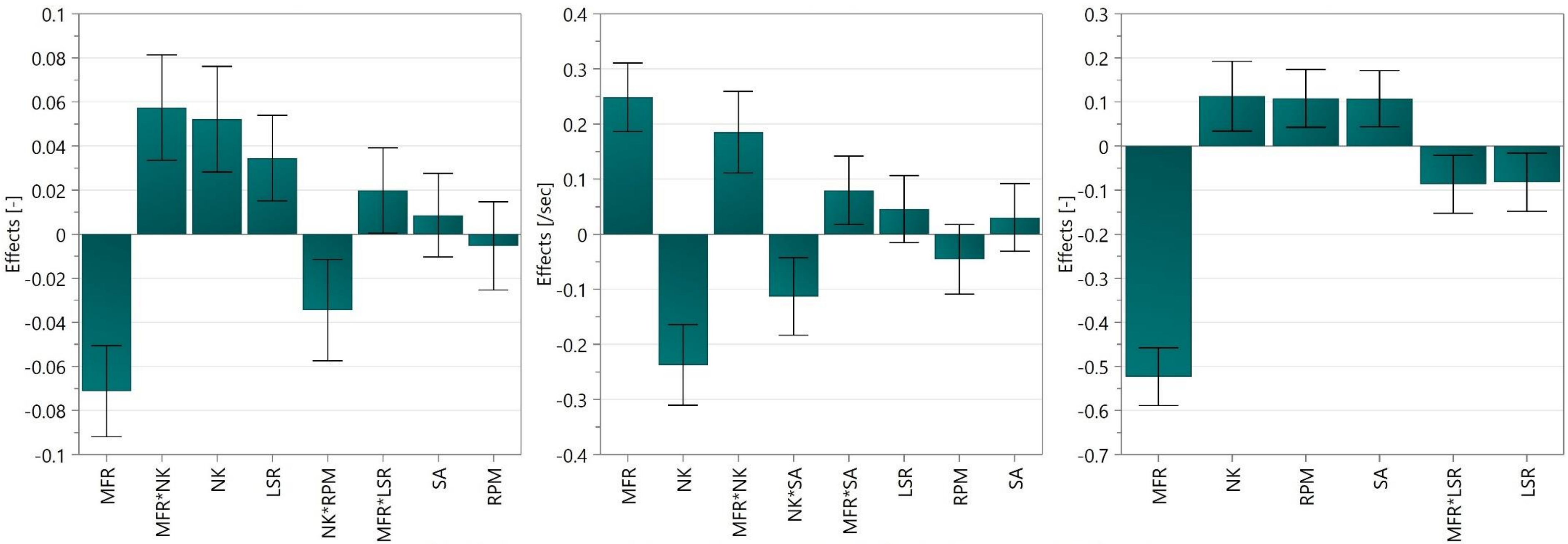


Increase no. of kneading and in L/S led to improved liquid distribution in bulk





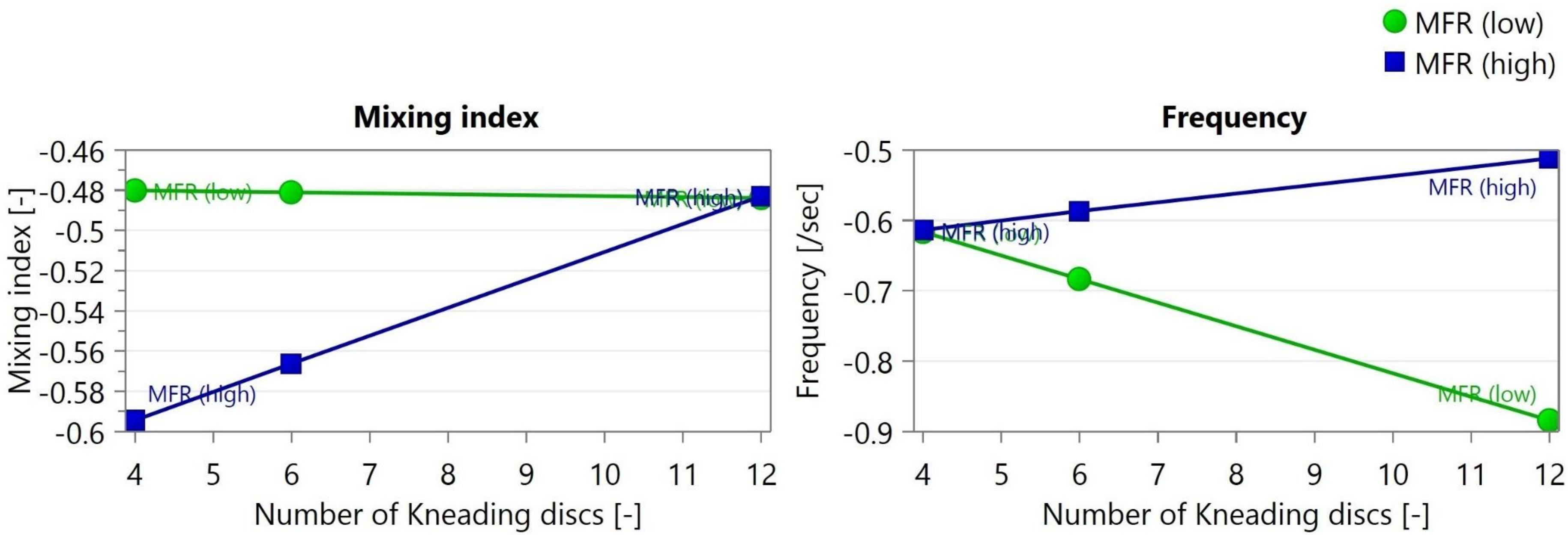
Mixing index Frequency Amplitude



Mixing index (N=43; DF=34; R2=0.80), Frequency (N=41; DF=32; R2=0.84), Amplitude (N=45; DF=38; R2=0.88) Conf. lev.=0.95

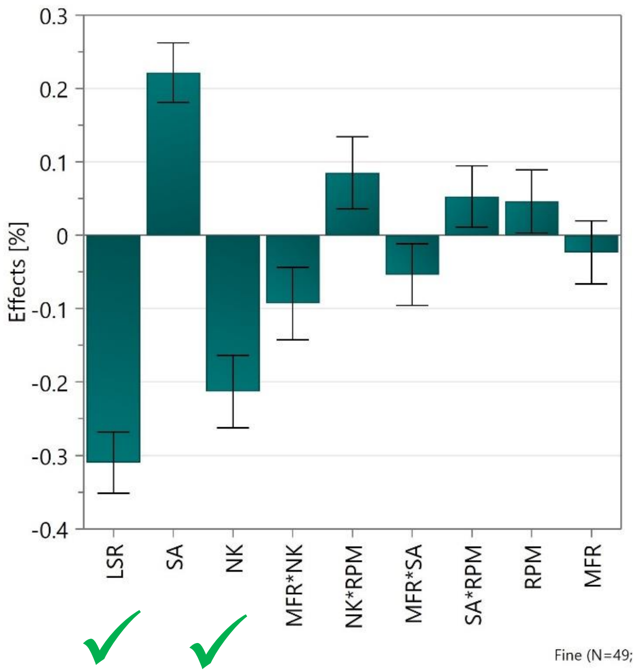
S/L mixing ↑ when MI ↑ , Frequency ↓ and Amplitude ↓

Trade-off existed between high throughput and number of kneading discs



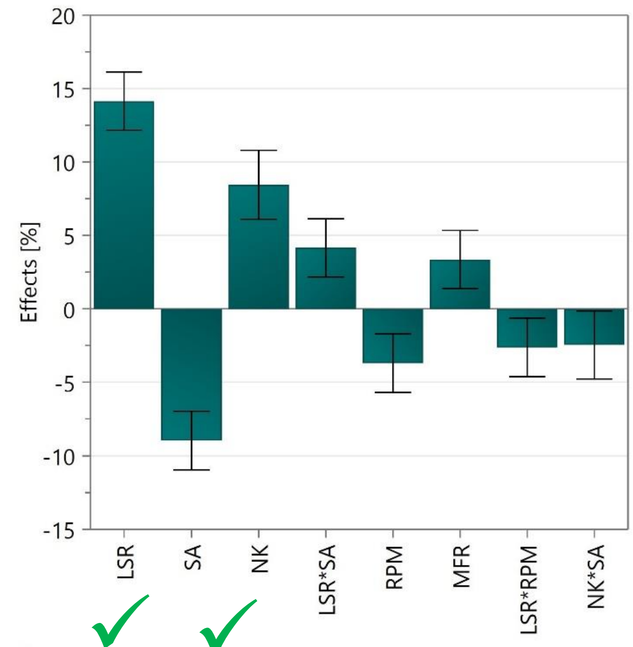
Increase in L/S and no. of kneading led to improved liquid distribution, hence less fines

Fine

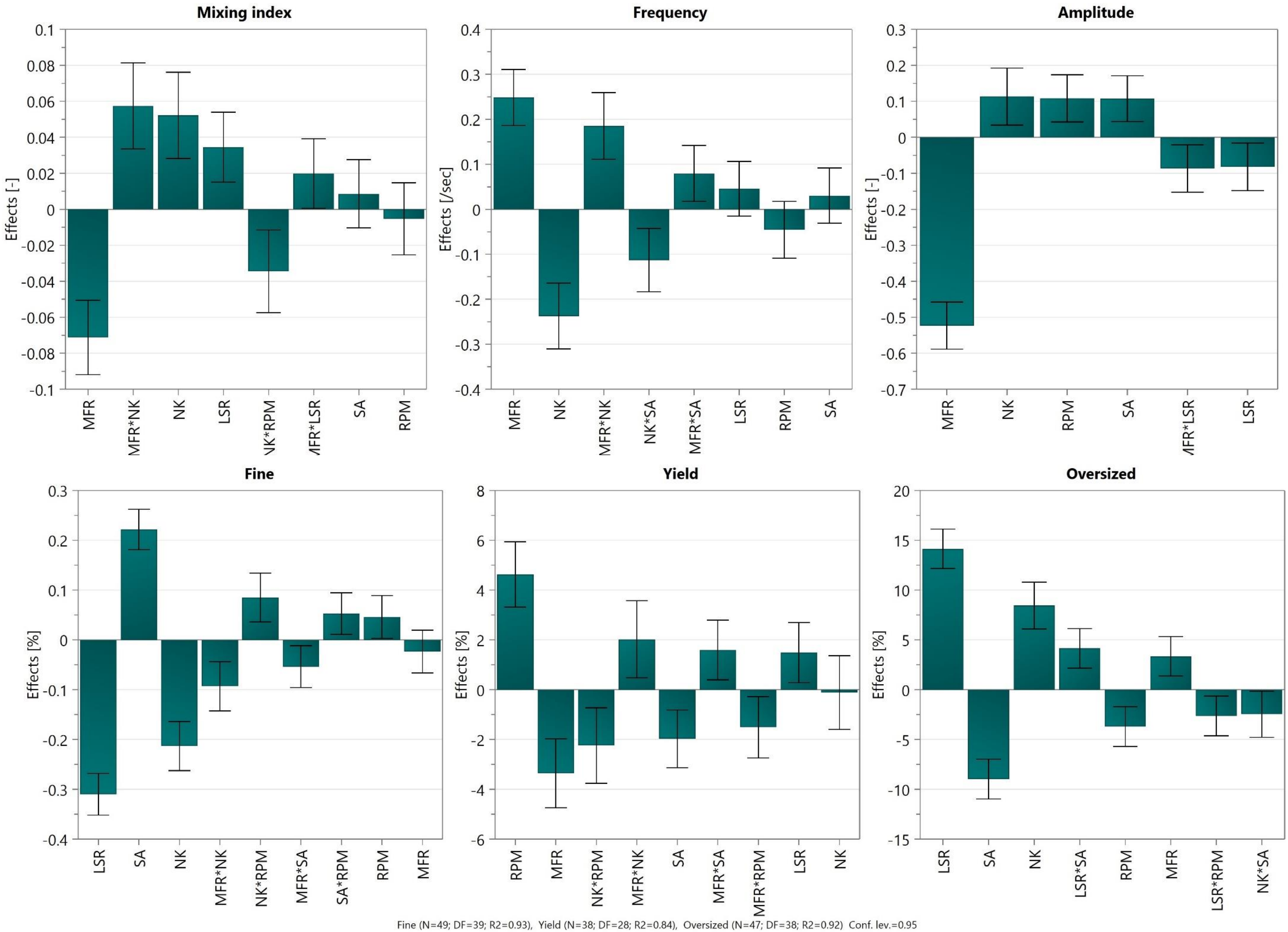


Yield

Oversized



Fine (N=49; DF=39; R2=0.93), Yield (N=38; DF=28; R2=0.84), Oversized (N=47; DF=38; R2=0.92) Conf. lev.=0.95



Mixing index
Frequency
Amplitude
Fine
Yield
Oversized
Effects [-]
Effects [/sec]
Effects [%]
MFR
MFR*NK
NK
LSR
NK*RPM
MFR*LSR
SA
RPM
NK*SA
MFR*SA
SA*RPM
MFR*RPM
LSR*SA
LSR*RPM
Fine (N=49; DF=39; R2=0.93), Yield (N=38; DF=28; R2=0.84), Oversized (N=47; DF=38; R2=0.92) Conf. lev.=0.95

Analysis of distributions in twin-screw granulation

RTD Measurement by Chemical Imaging

Results

Summary

The results showed that..

..material throughput and number of kneading discs dictate solid-liquid mixing.

…till good mixing kneading discs are not there TSG should better be operated at lower throughput.

.. non-conventional screw elements with modified geometries should be explored for improvement in solid-liquid mixing.

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Discrete element method (DEM): detailed simulation of wet granulation

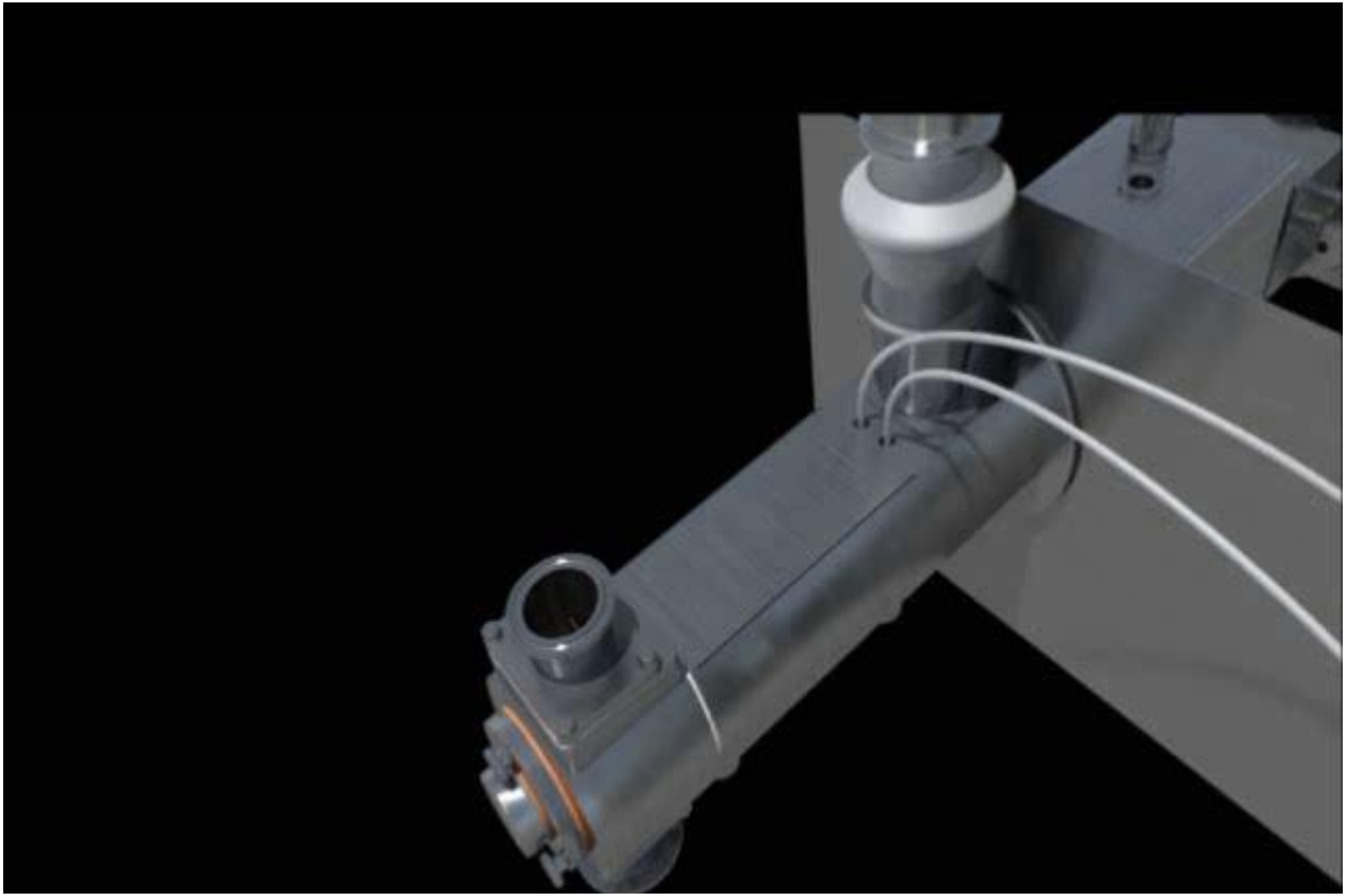
Involved physics in granulation

- Mixing
- Wetting
- Aggregation (PBM)
- Breakage (PBM)

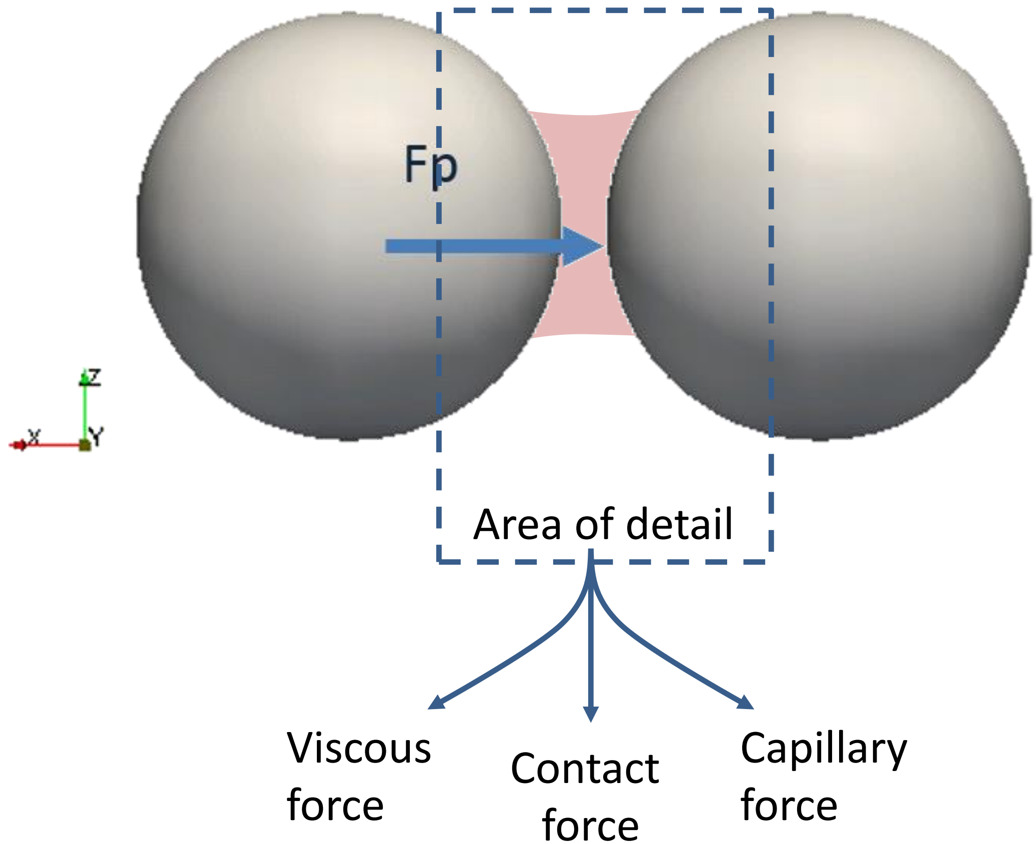
Challenges of PBM

- Does not include geometrical effect
- Rate of sub-processes (i.e. aggregation, breakage) is lumped into kernels which are
  - Size dependent
  - Energy/shear dependent
- Need extensive experimental calibration

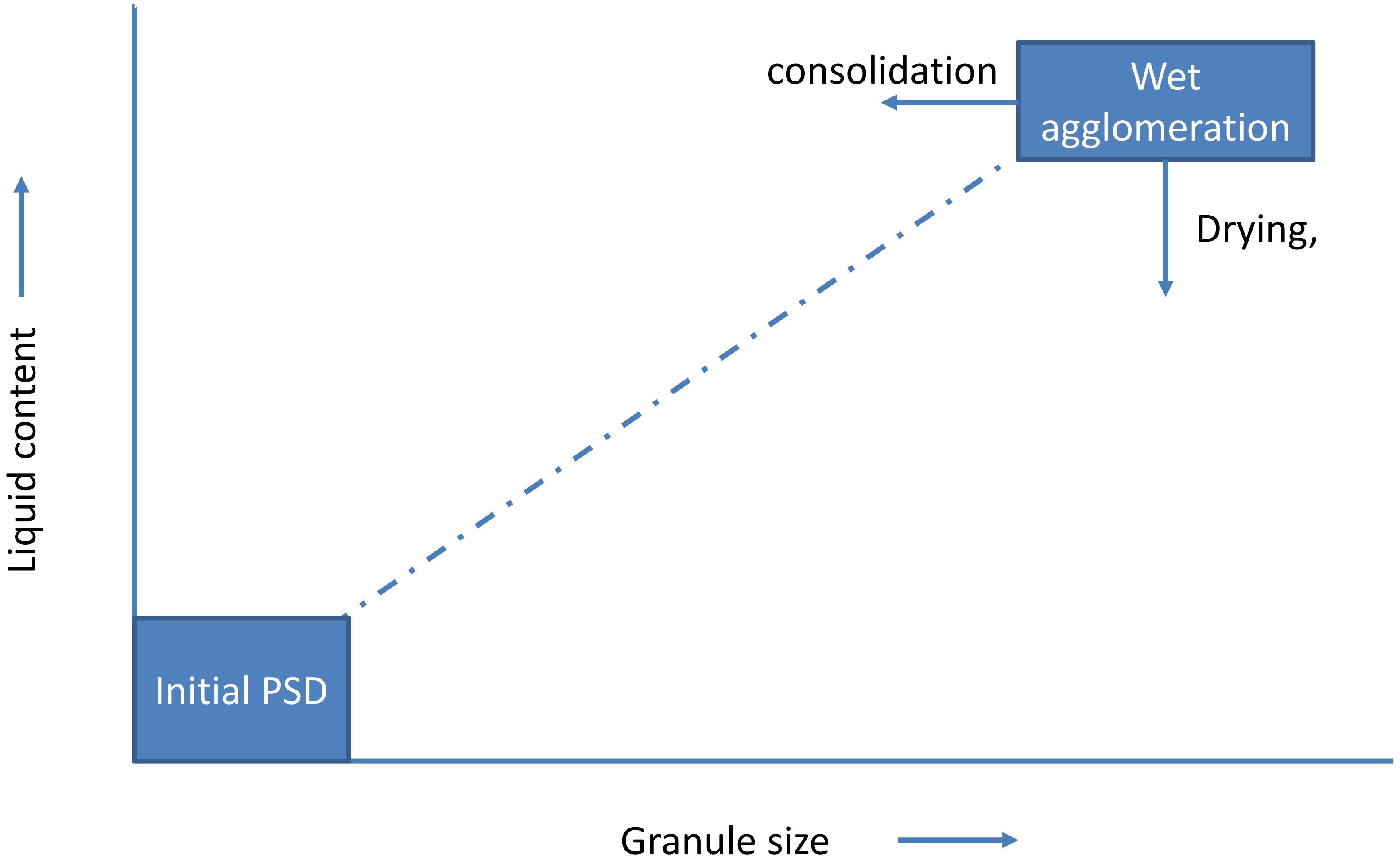
Every contact and neighborhood matter!



DEM for detailed investigation



Particle scale DEM



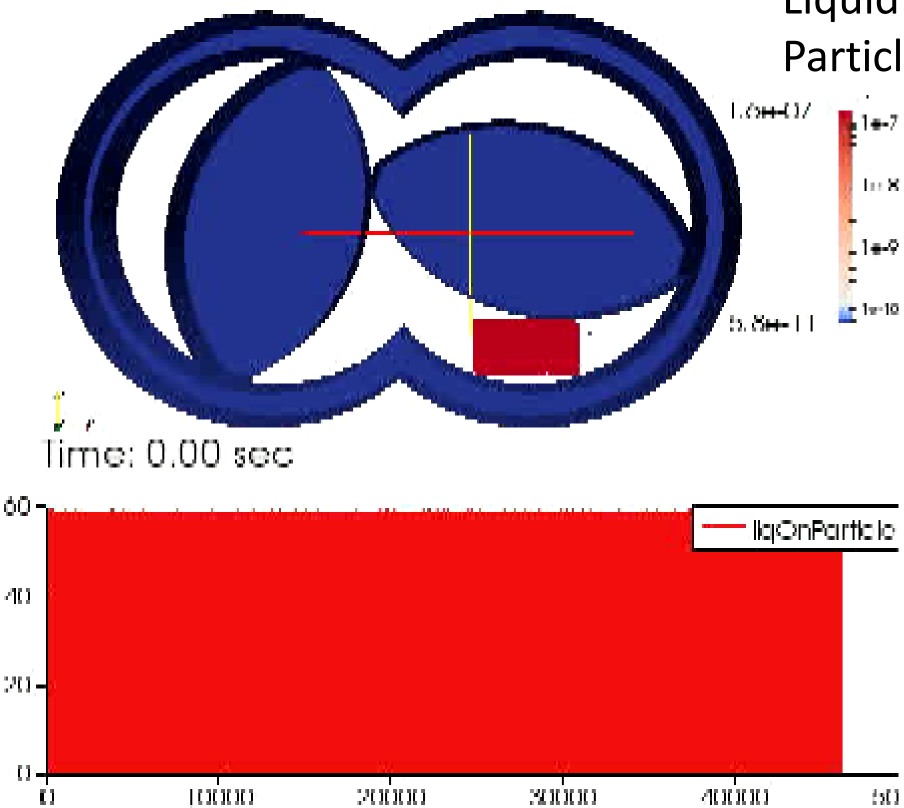
Initial parameter values

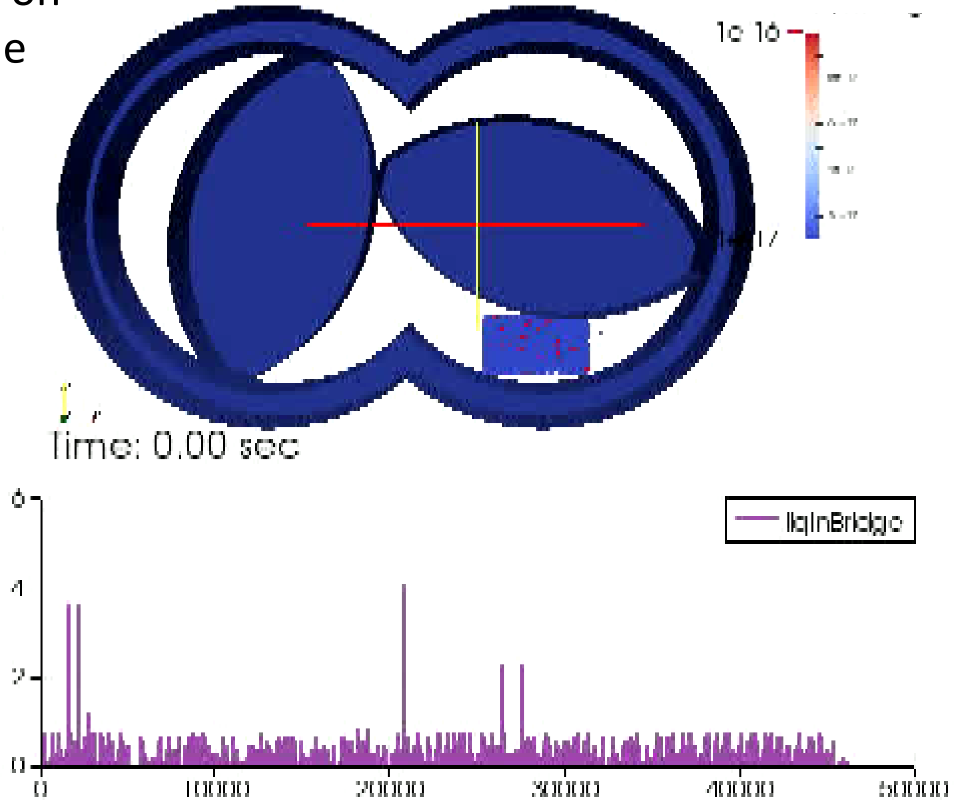
Parameter	values
Throughput	10-25 kg/h
Material density	1500 kg/m3
Bulk density	500-750 kg/m3
Particle diameter	30-70 µm
Number of particles	~60- 150 million # /sec

A lot of detail can be extracted

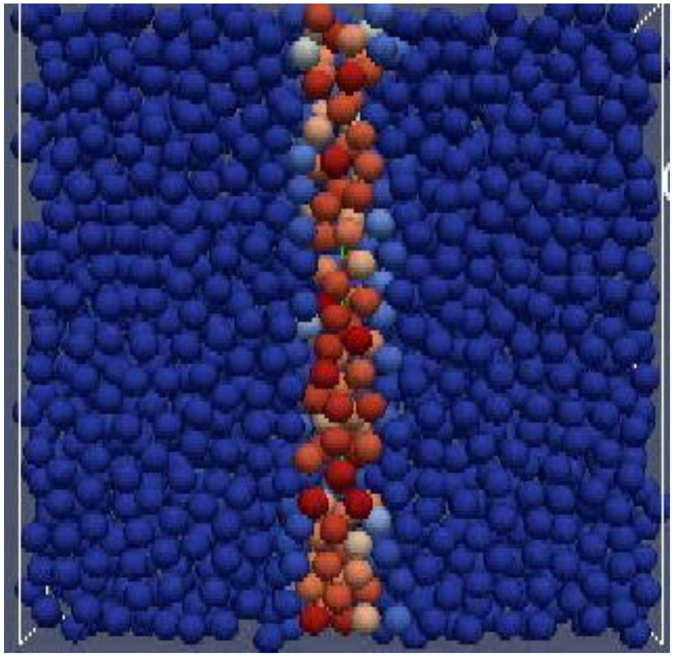
Liquid on Particle

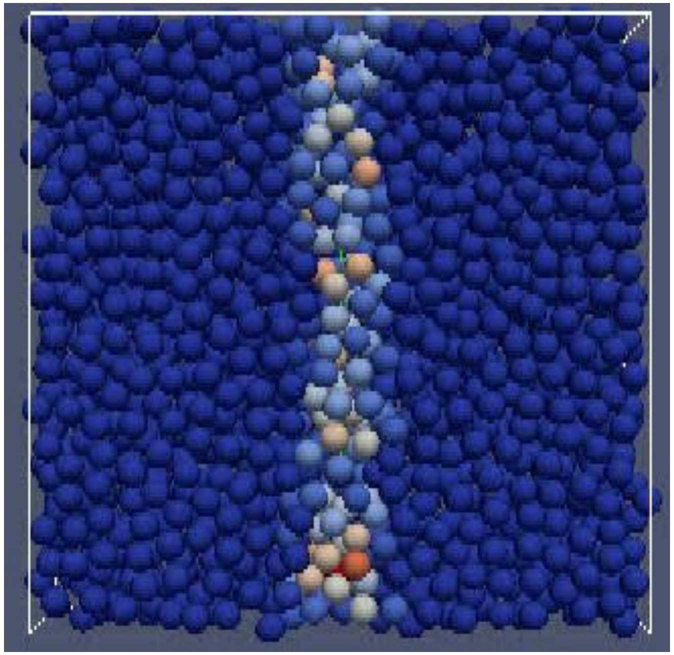
Liquid In Bridge



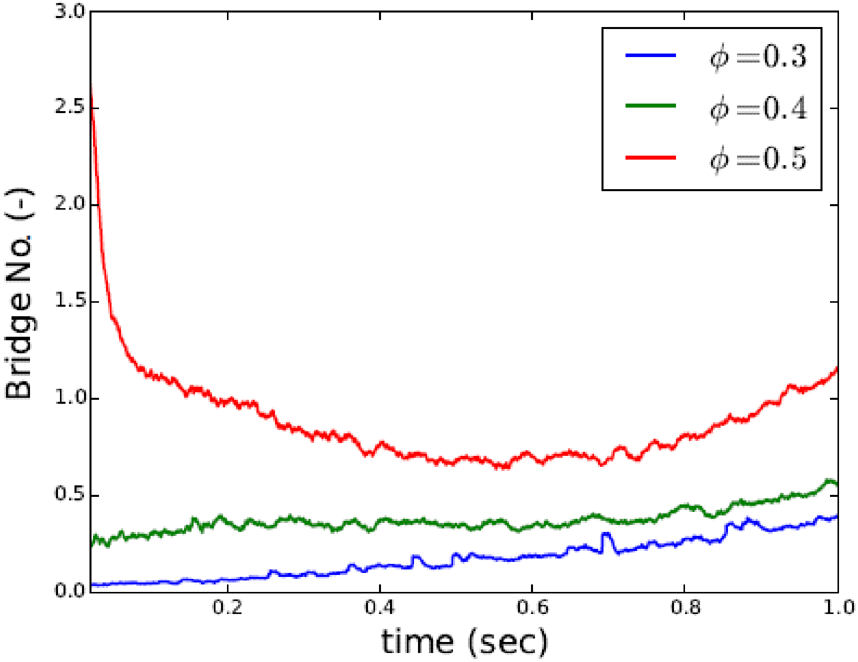


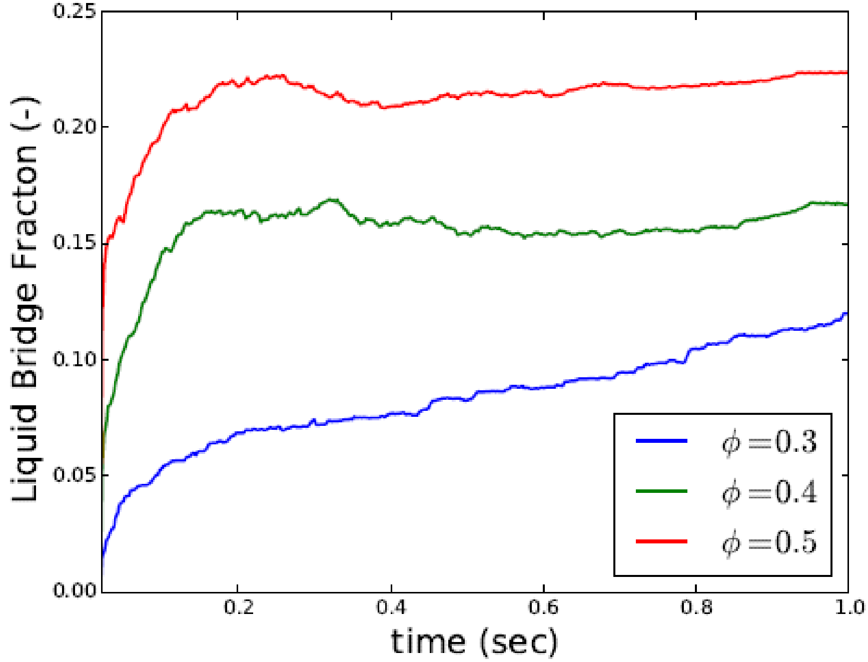
Sheared-box simulation





Breakage is dominant mechanism at high fill ratio





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Model-based analysis and optimization of bioprocesses

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