Model-based design of a roller compaction milling process

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Advanced Process Modelling Forum

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MARIE CURIE ACTIONS

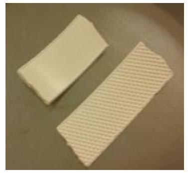
IPROCOM

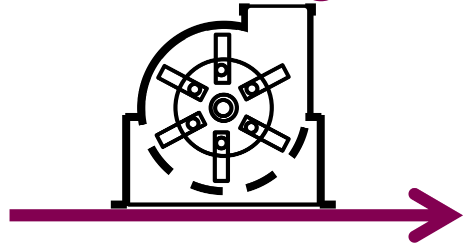
The Development of *in silico* Process Models for Roll Compaction

- Project coordinator: Chuan-Yu (Charley) Wu (University of Surrey)
- 14 partners from 8 European countries
- Goals:
  - To employ a multi-disciplinary approach to understand the fundamental mechanisms of particulate manufacturing processes involving roll compaction, milling , and die compaction.
  - To develop predictive *in silico* tools that can be used by various industrial sectors in Europe.

milling

ribbons





granules

Ribbon properties

-dimensions of ribbon

-mechanical properties of ribbon:

- porosity
- hardness
- elasticity
- brittleness
- plasticity

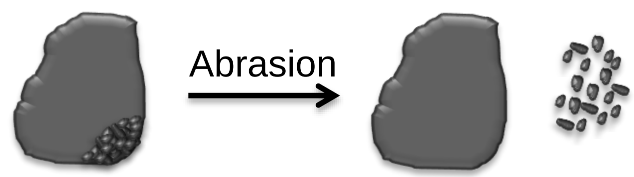
Mill properties

- mesh size
- screen type
- rotor speed
- rotor design
- distance between rotor and mesh
- feeding rate
- milling mode

<u>Breakage mechanism</u>

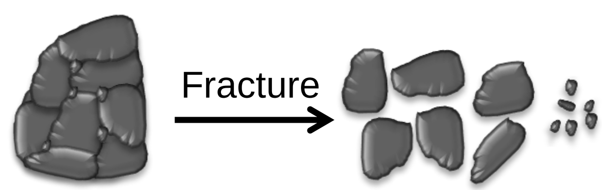
1. Abrasion

- attrition milling of low intensity stresses
- much fine particles produced
- shear failure

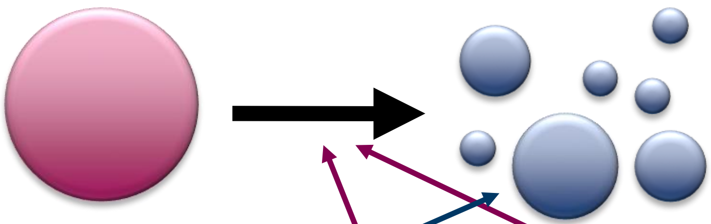


2. Fracture

- impact milling of intense stresses
- daughter particles of similar sizes and shapes
- tensile failure



Population balance model



$$\frac{\partial n(u,t)}{\partial t} = \int_u^{\infty} b(u,v)\, S(v)\, n(v)\, dv - S(u)\, n(u)$$

To model the rate of change of number concentration of particles of size *u*.

Breakage function b is the probability of formation of a particle of size class v after breakage of a particle from class *u*.

Selection function S is rate of breakage of particles.

Mechanistical models

Vogel & Peukert (2003):

$$S = 1 - \exp\left\{ f_{Mat}\, x k \left(W_{m,kin} - W_{m,\min} \right) \right\}$$

Threshold energy for particle of size x

Material strength parameter

Number of impacts

Mass specific impact energy

$$B = \left(\frac{x}{y}\right)^{q} \frac{1}{2} \left\{ 1 + \tanh\left(\frac{y - y'}{y'} \right) \right\}$$

Minimum particle size limit

Power law exponent

DeVegt (2007):

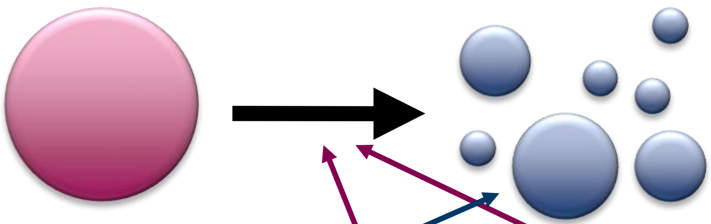
$$S = 5.85 * 10^{8} \frac{E_{kin}\, E_{fract} \sqrt{\frac{P_y}{\rho}}}{V\ H\ \sqrt{x} K_{IC}}$$

Ghadiri & Zhang (2002)

$$\xi = \frac{\rho v^2 x H}{K_C^2}$$

E_{kin}	Kinetic energy of particles
E_{fract}	Fracture energy
P_y	Yield pressure
ρ	Particle density
V	Volume of mill chamber
H	Hardness
ξ	Fractional loss per impact
v	Velocity
K_c	Critical stress intensity factor

Population balance model



$$\frac{\partial n(u,t)}{\partial t} = \int_u^{\infty} b(u,v) S(v) n(v) dv - S(u) n(u)$$

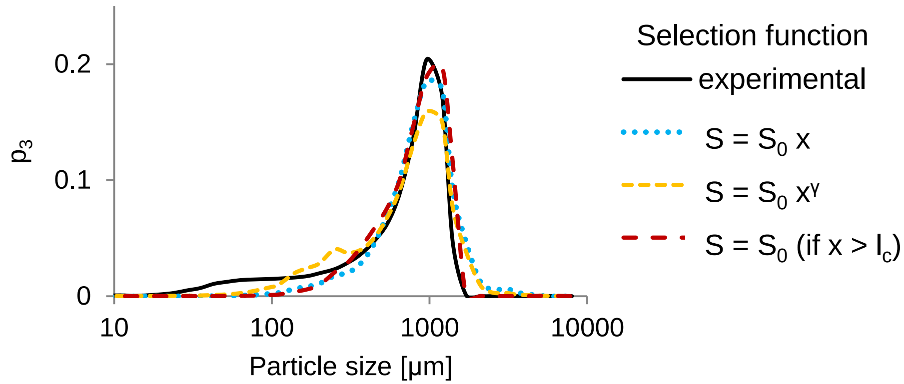
To model the rate of change of number concentration of particles of size *u*.

Breakage function b is the probability of formation of a particle of size class *v* after breakage of a particle from class *u*.
Selection function S is rate of breakage of particles.

Semi-empirical models

Selection function

- Size independent kernel: $S = S_0$
- Power law kernel: $S = S_0\ x^{\gamma}$
- Exponential kernel: $S = S_0 \exp(-\gamma\ x)$

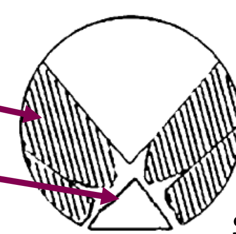


Breakage function

- Lognormal distribution
- Hill & Ng (1995)

Impact breakage → bimodal distribution (coarse + fine mode)

- Multiple fragmentation
- Localised disintegration



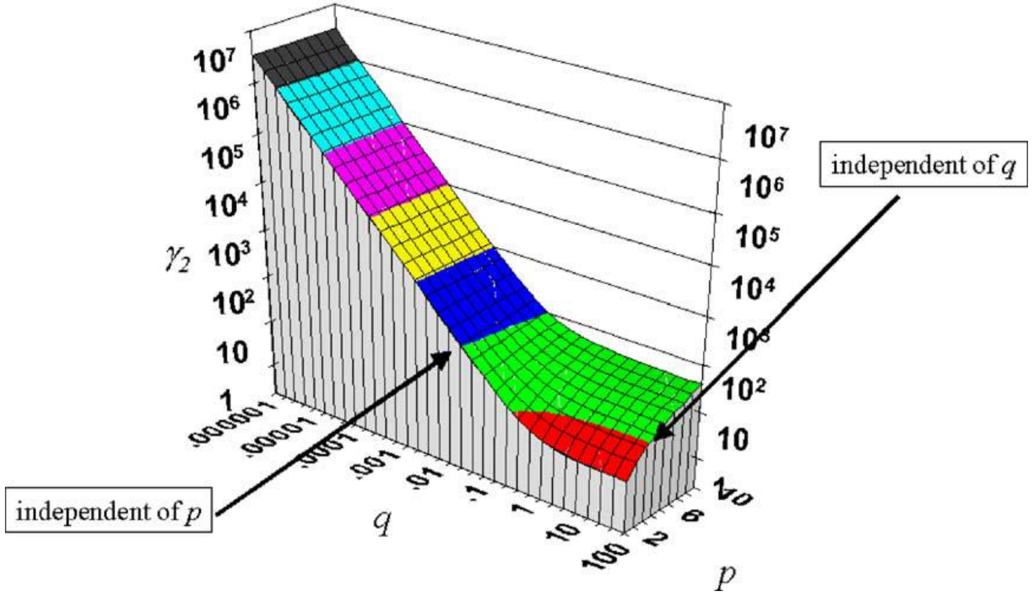
Salman *et al.*, 2004

Hill & Ng fragment distribution function

$$b(u,v)=\frac{p}{v}\frac{\left(\frac{u}{v}\right)^{q-1}\left(1-\left(\frac{u}{v}\right)\right)^{r-1}}{B(q,r)}$$

p – number of fragments
q – sharpness coefficient

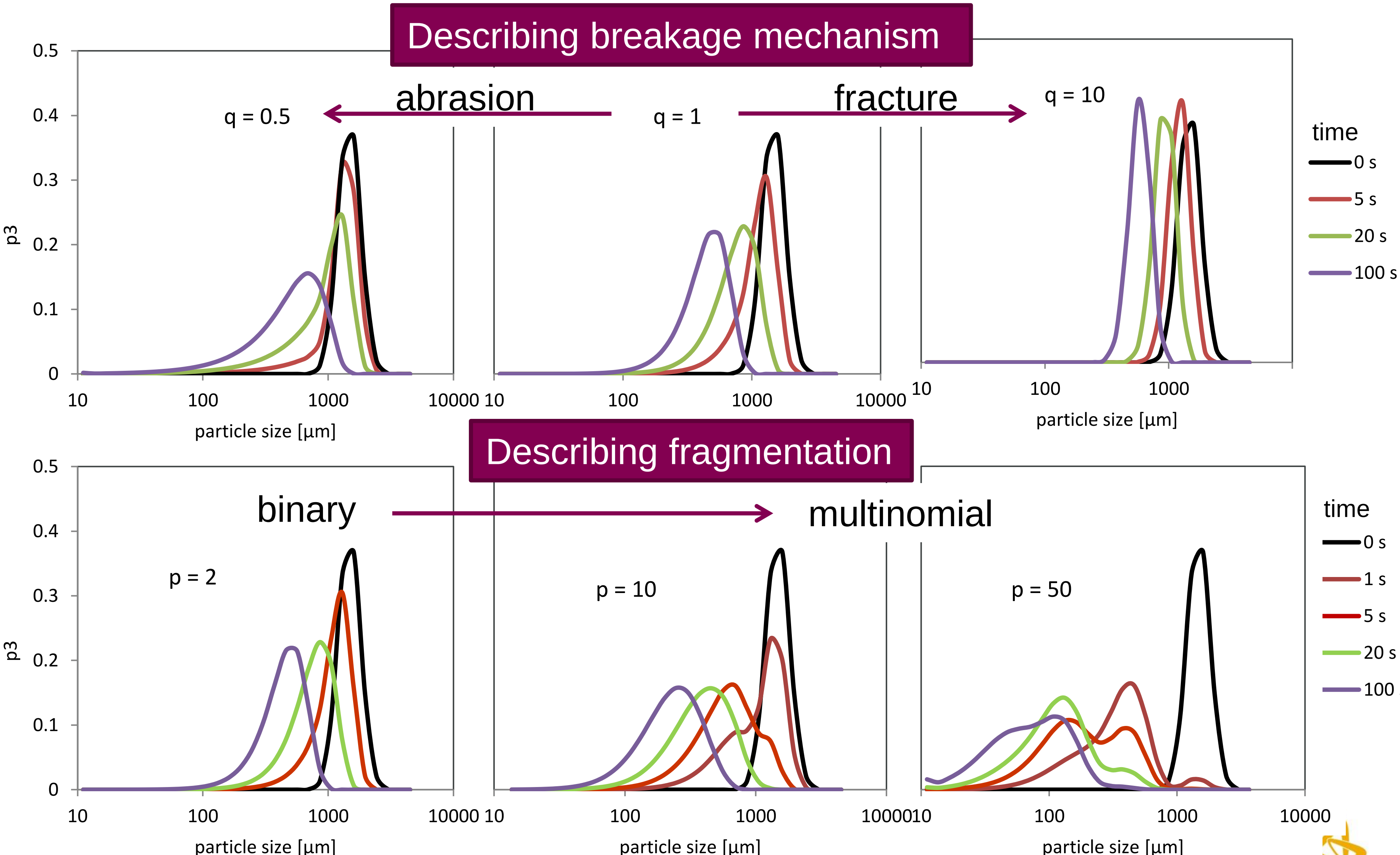
$$r=q(p-1)$$



Diemer *et al.*, 2005

Hill & Ng fragment distribution function

$$b(u,v)=\frac{p}{v}\frac{\left(\frac{u}{v}\right)^{q-1}\left(1-\left(\frac{u}{v}\right)\right)^{r-1}}{B(q,r)}$$

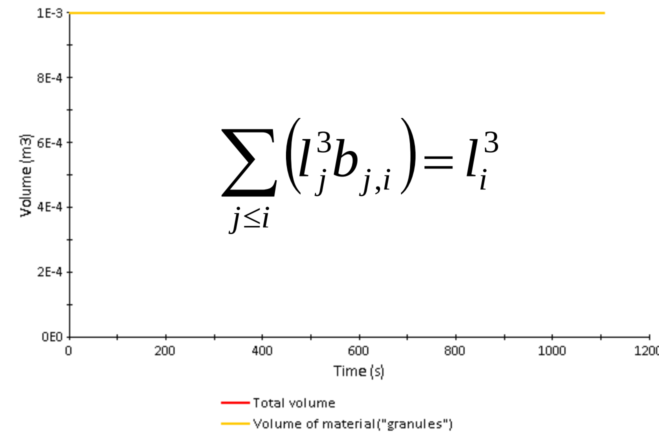


Implementing new fragment distribution models into gSolids

$$S(t,l)=l^3 \qquad b(x,l)=\frac{6x^2}{l^3}$$

Analytical solution: $n(t,l)=3l^2(1+t)e^{-l^3(1+t)}$

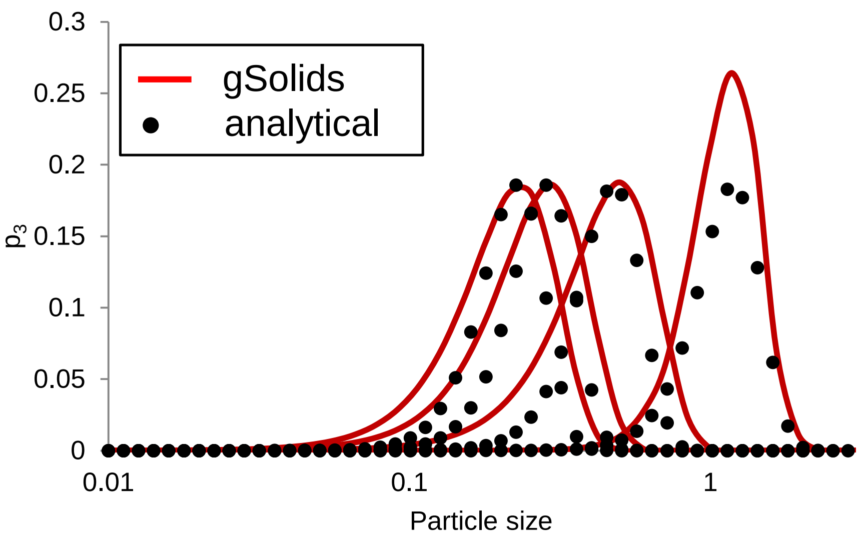
Volume is conserve

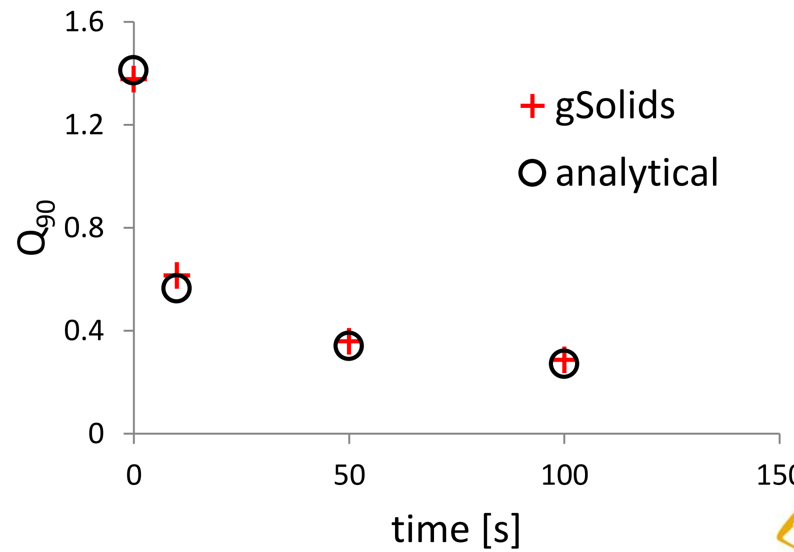


$$\sum_{j\le i}\left(l_j^3 b_{j,i}\right)=l_i^3$$

Time [s]:

100←50←10←0





Population balance equation

$$\frac{\partial n(u,t)}{\partial t} = \int_u^\infty b(u,v) S(v) n(v) dv - S(u) n(u)$$

Fragment distribution

For fine mode: lognormal

For coarse mode: Hill & Ng

$$B = \frac{\zeta}{v\sqrt{2\prod}\ln\sigma}\exp\left[-\left(\frac{\ln(v/\mu)}{\sqrt{2}\ln\sigma}\right)^2\right] + (1-\zeta)\left(\frac{p}{v}\right)\left(\frac{u}{v}\right)^{q-1}\left(1-\left(\frac{u}{v}\right)\right)^{r-1} / beta(q,r)$$

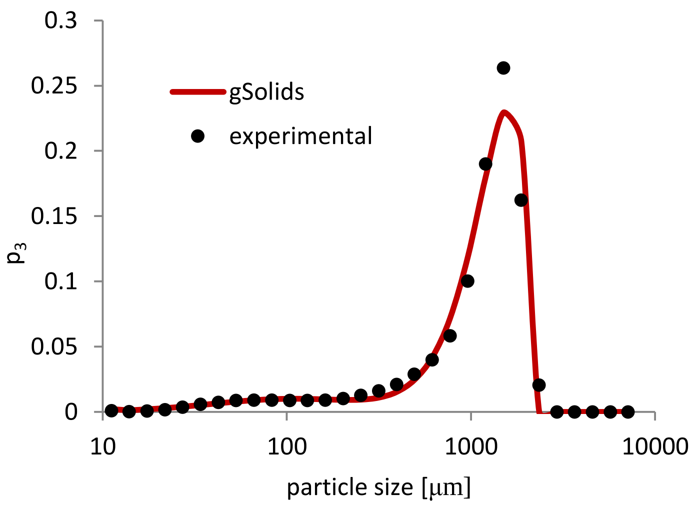
Breakage rate

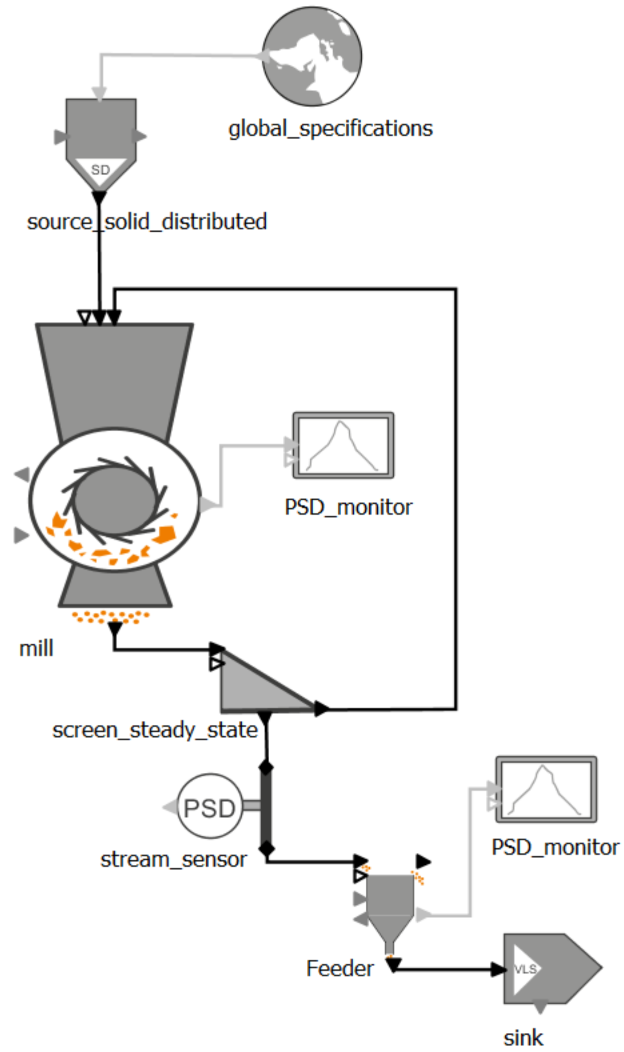
Cut-off

$$S(l) = \begin{cases} 0 & l < l_c \\ S_0 & l > l_c \end{cases}$$

Parameters

Breakage rate constant, S_0
Critical fragment size, l_c
Fraction of fines, ζ
Geometric mean, μ
Standard deviation, σ
Number of fragments, p
Sharpness coefficient, q





Parameter estimation for breakage rate

$$S(l) = \begin{cases} 0 & l < l_c \\ S_0 & l > l_c \end{cases}$$

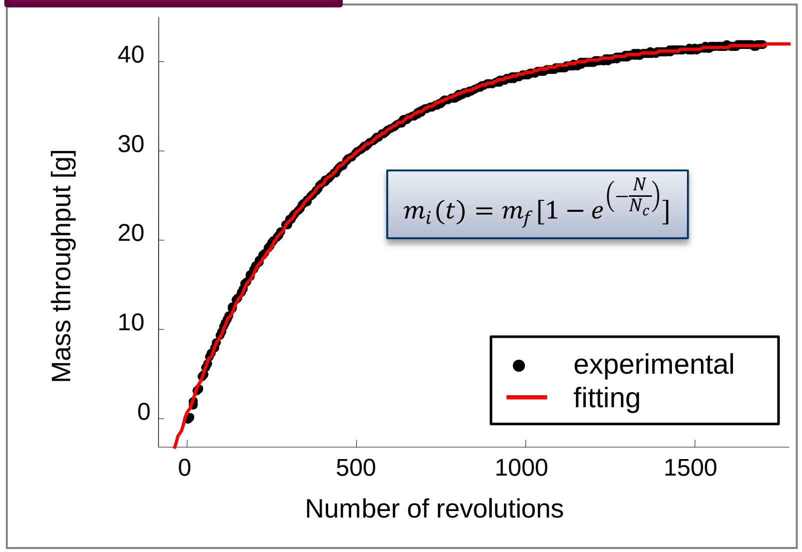
Parameters
Breakage rate constant, S_0
Critical fragment size, l_c
Fraction of fines, ζ
Geometric mean, μ
Standard deviation, σ
Number of fragments, p
Sharpness coefficient, q

$$S_0 = 1/t_{milling}$$

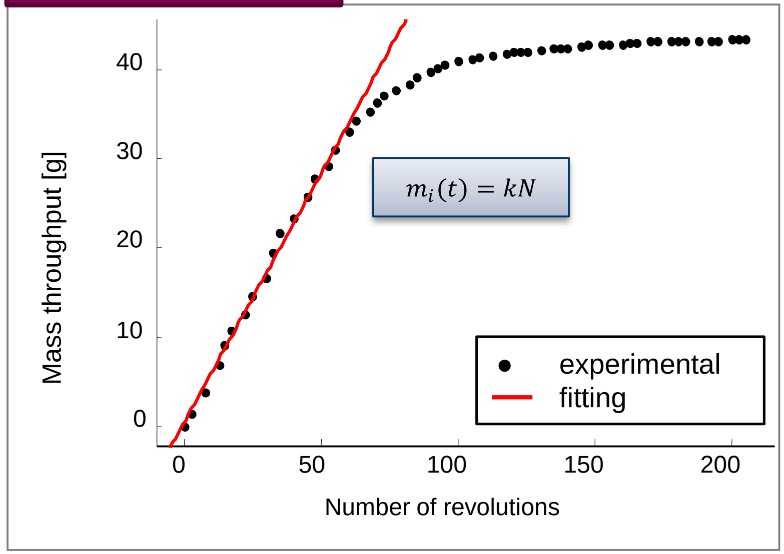
1. Milling mass throughput
2. Topmost size class PSD disappearance plot

Milling mass throughput

MCC Avicel PH101
ε = 24 %
1 mm screen size
150 rpm impeller speed



MCC Avicel PH101
ε = 24 %
2 mm screen size
150 rpm impeller speed

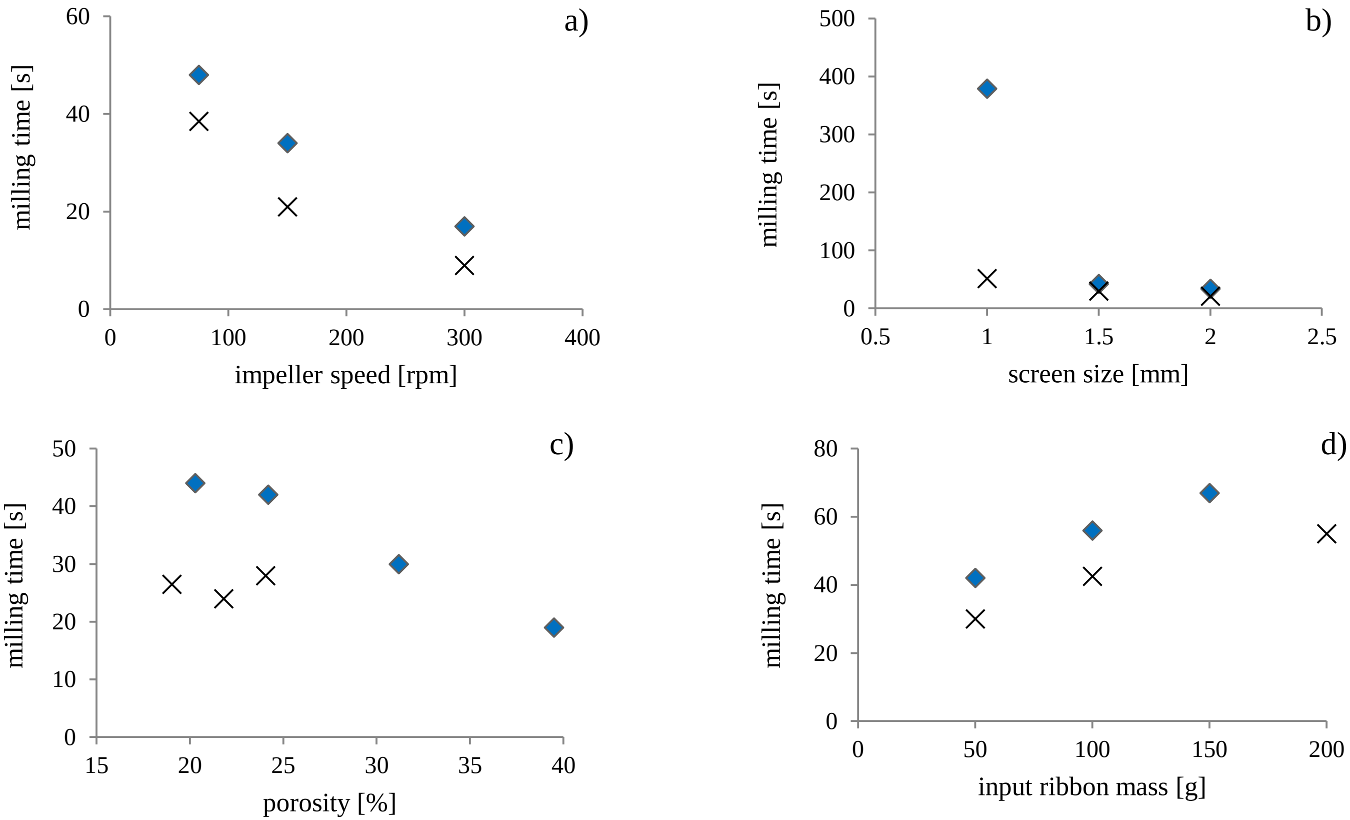


•Milling kinetic profile for MCC dependent on screen size: different breakage mechanisms.

• Milling of mannitol ribbons resulted only in linear mass throughput for all mesh apertures.

•Milling time = time(90 % of granules exit the mill)

Effect of process parameters and ribbon prosperities on milling time



Milling time for MCC (♦) and mannitol (×) ribbons when milling ribbons with:

a) 24 % ribbon porosity, 2 mm screen size and different impeller speeds

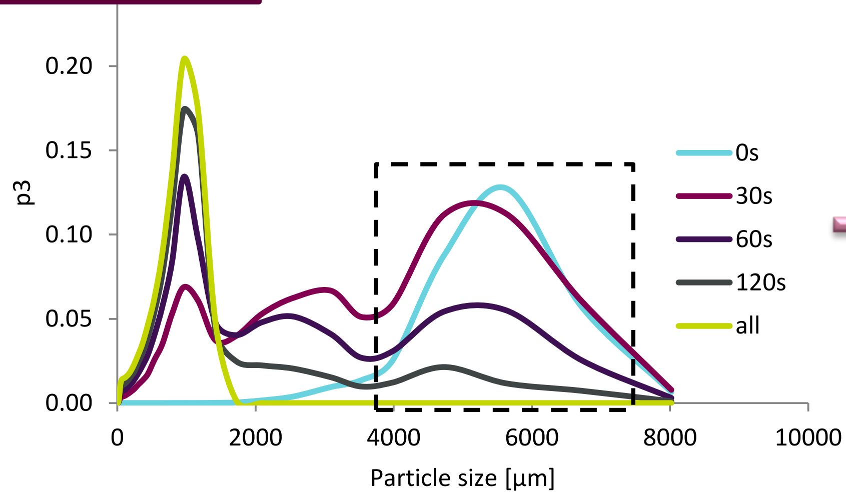
b) 24 % ribbon porosity, 150 rpm impeller speed and different screen sizes

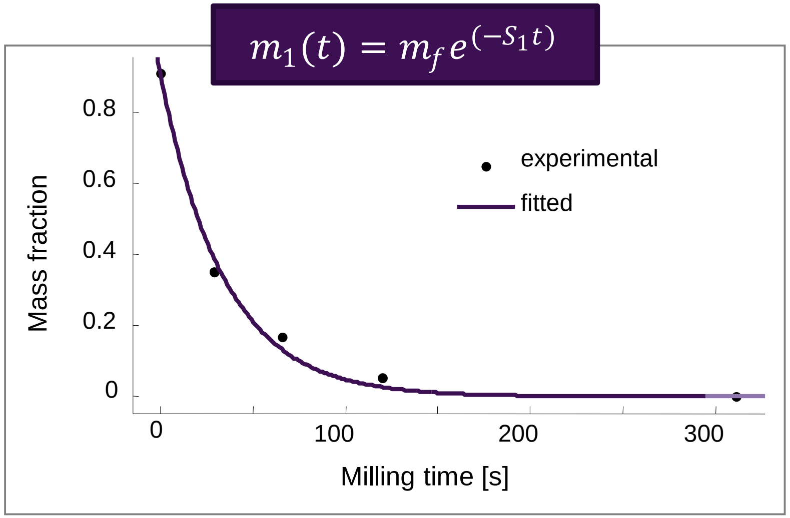
c) different ribbon porosities, 150 rpm impeller speed and 1.5 mm screen size

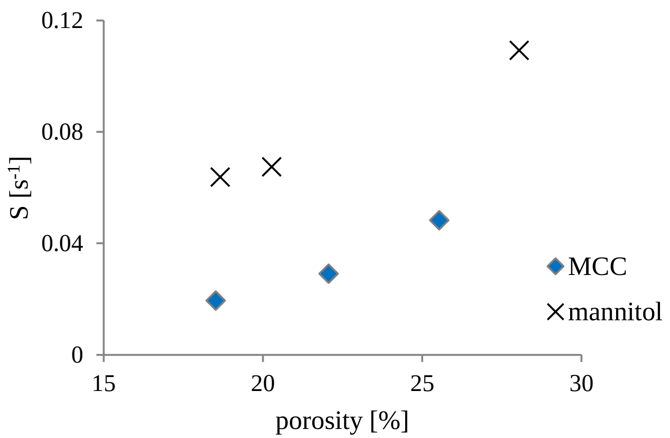
d) 24 % ribbon porosity, 150 rpm impeller speed, 1.5 mm screen size and different input ribbon masses

Study of breakage rate from topmost size class PSD disappearance plot

MCC Avicel PH101
ε = 19 %
1 mm screen size
150 rpm impeller speed







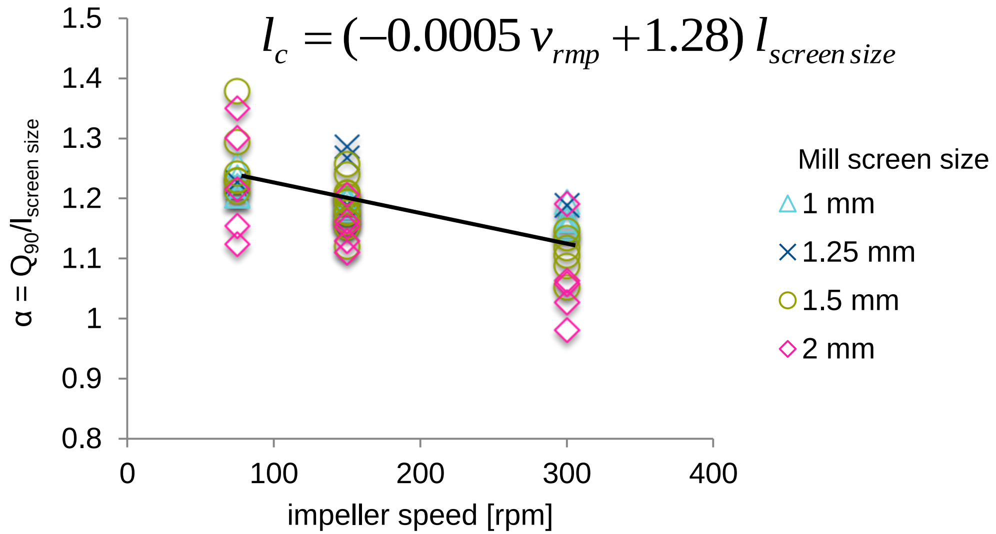
•Breakage rate increases with increasing ribbon porosity
•Breakage rate is higher for mannitol than for MCC ribbons.

Parameter estimation for breakage rate

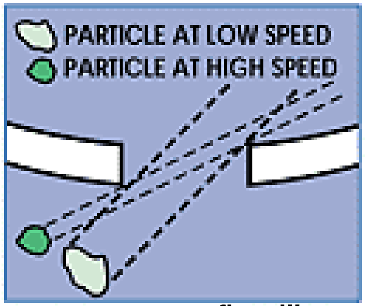
$$S(l)=\begin{cases}0 & l<l_c\\ S_0 & l>l_c\end{cases}$$

Parameters
Breakage rate constant, S_0
Critical fragment size, l_c
Fraction of fines, ζ
Geometric mean, μ
Standard deviation, σ
Number of fragments, p
Sharpness coefficient, q

$$l_c = \alpha\, l_{screen\,size}$$



- α describes a reduction in apparent screen size due to the tangential movement of the particles in the mill.
- Reduction of α with increasing impeller speed → angle between particles and the screen becomes more acute.



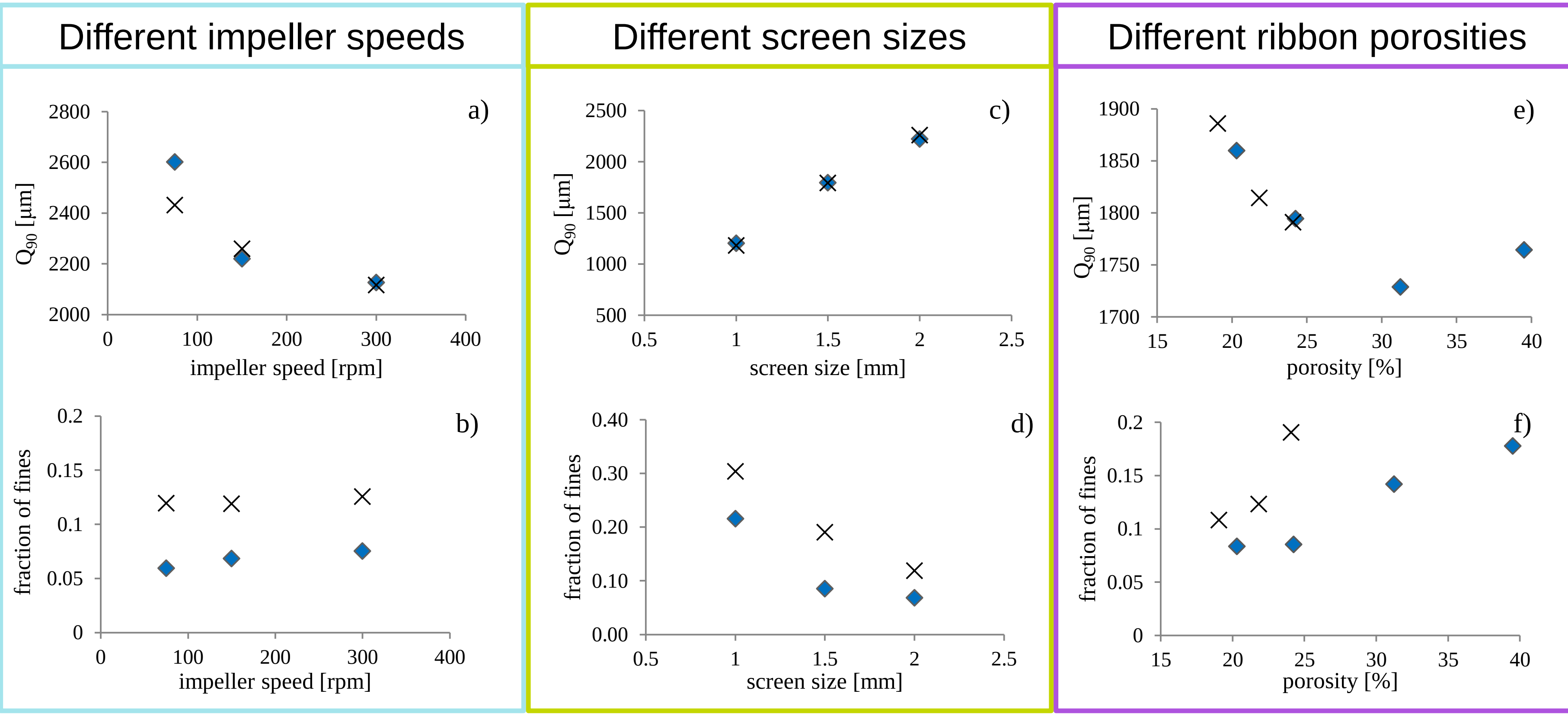
www.fitzmill.com

Parameter estimation for fragment distribution

Fragment distribution	$$B=\frac{\zeta}{v\sqrt{2\pi}\ln\sigma}\exp\left[-\left(\frac{\left(\ln\left(v/\mu\right)\right)}{\sqrt{2}\ln\sigma}\right)^{2}\right]+\left(1-\zeta\right)\left(\frac{p}{v}\right)\left(\frac{u}{v}\right)^{q-1}\left(1-\left(\frac{u}{v}\right)\right)^{r-1}/\,beta\left(q,r\right)$$
For fine mode: lognormal	
For coarse mode: Hill & Ng (1995)	$r=q\left(p-1\right)$

Parameters
Breakage rate constant, S_0
Critical fragment size, l_c
Fraction of fines, ζ
Geometric mean, μ
Standard deviation, σ
Number of fragments, p
Sharpness coefficient, q

Effect of process parameters and ribbon prosperities on Q_{90} and fraction of fines



Dependencies of Q_{90} and fraction of fines for MCC (♦) and mannitol (×) when milling ribbons with:

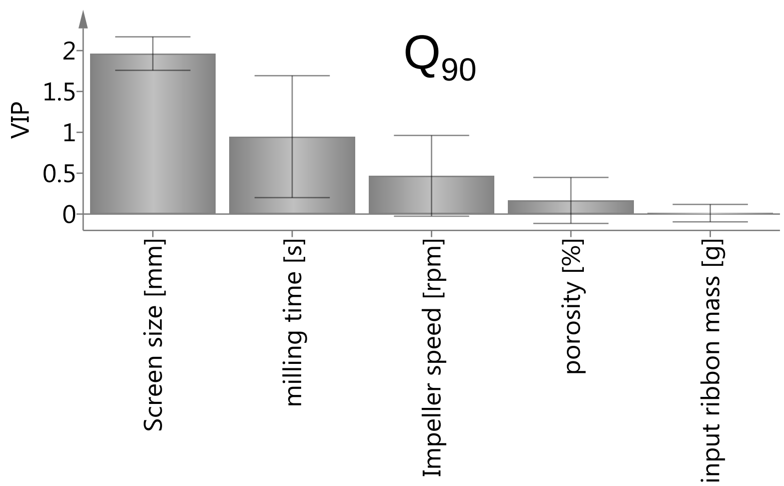
a) & b) 24 % porosity, 2 mm screen size and different impeller speeds;

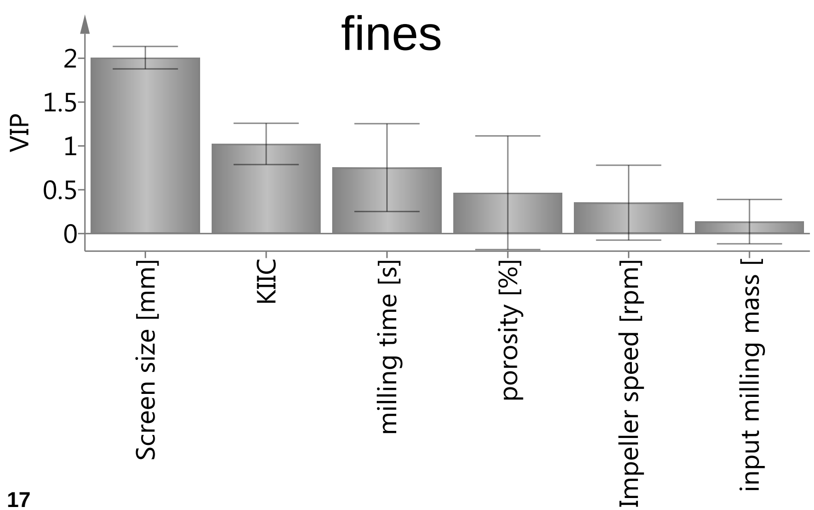
c) & d) 24 % porosity, 150 rpm impeller speed and different screen sizes;

e) & f) different porosities, 150 rpm impeller speed and 1.5 mm screen size.

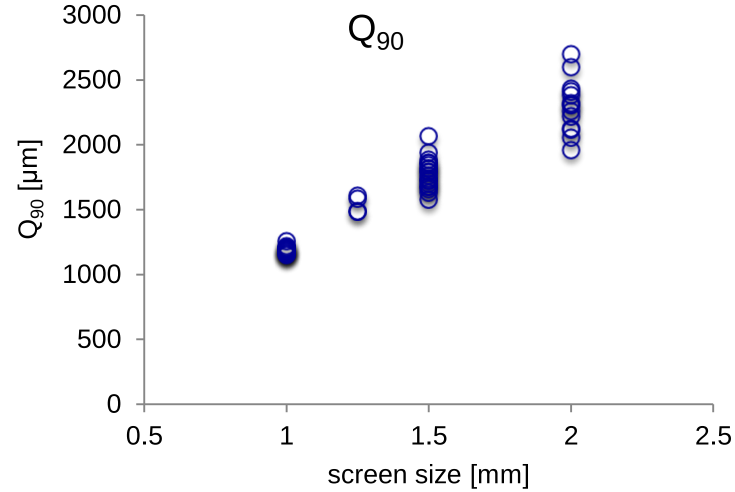
Multivariate analysis for Q_{90} and fraction of fines

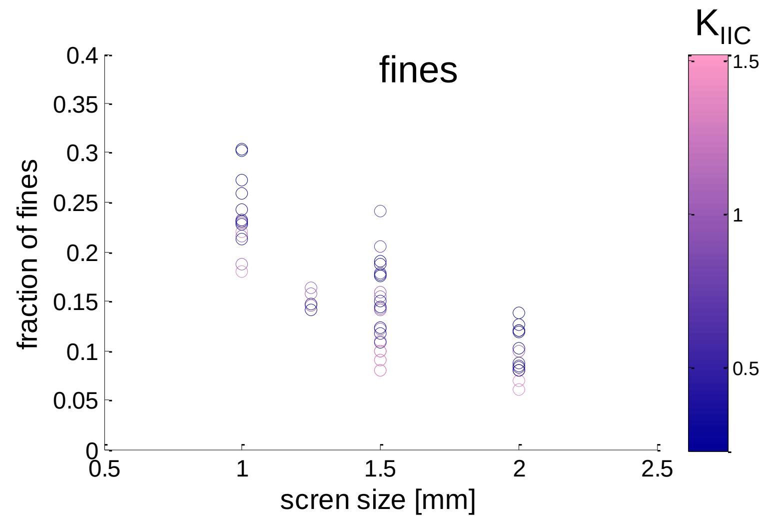
Variable importance for the projection (VIP)





Partial least squares (PLS) regression method





Parameter estimation for fragment distribution

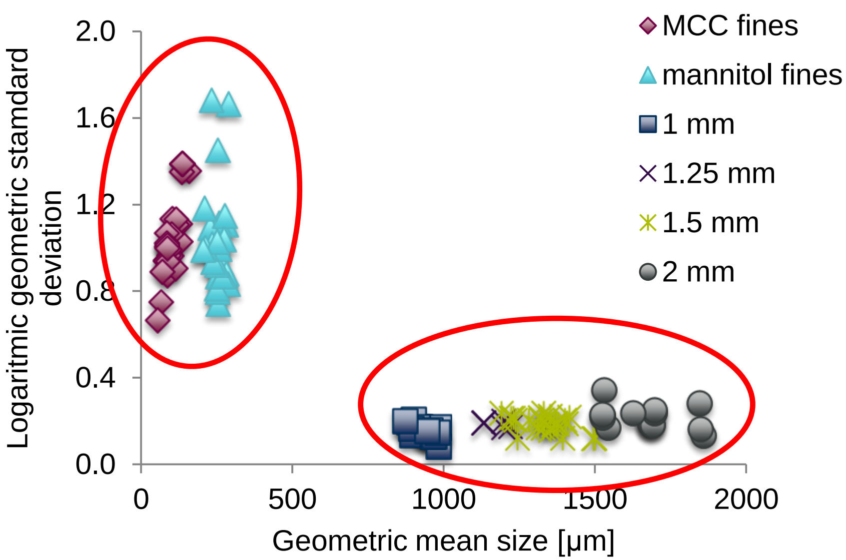
Fragment distribution For fine mode: lognormal For coarse mode: Hill & Ng (1995)	$B = \frac{\zeta}{v\sqrt{2\prod}\ln\sigma}\exp\left[-\left(\frac{\ln(v/\mu)}{\sqrt{2}\ln\sigma}\right)^2\right] + (1-\zeta)\left(\frac{p}{v}\right)\left(\frac{u}{v}\right)^{q-1}\left(1-\left(\frac{u}{v}\right)\right)^{r-1} / beta(q,r)$ $r = q(p-1)$

Parameters
Breakage rate constant, S_0
Critical fragment size, l_c
Fraction of fines, ζ
Geometric mean, μ
Standard deviation, σ
Number of fragments, p
Sharpness coefficient, q

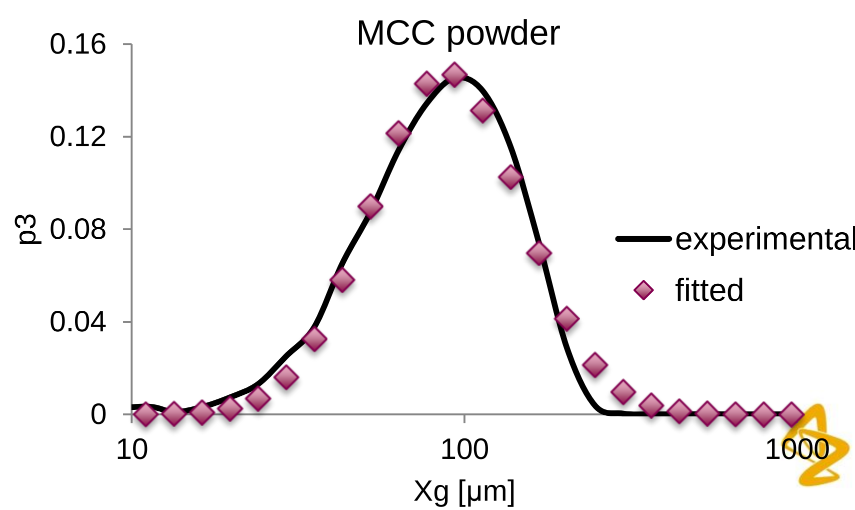
Characterization of fines

Fitting bimodal lognormal distribution with characteristic geometric mean (μ) and standard deviation (σ) for fines and coarse mode.

$$f(l) = \left(\frac{\zeta}{l\sqrt{2\pi}\ln\sigma_{1,i}}\, exp\left[-\left(\frac{\ln\left(l/\mu_{1,i}\right)}{\sqrt{2}\ln\sigma_{1,i}} \right)^2 \right] + \frac{1-\zeta}{l\sqrt{2\pi}\ln\sigma_{2,i}}\, exp\left[-\left(\frac{\ln\left(l/\mu_{2,i}\right)}{\sqrt{2}\ln\sigma_{2,i}} \right)^2 \right] \right)$$



powder	Mean [μm]	Standard deviation
MCC	79.9	1.69
mannitol	167.4	1.35

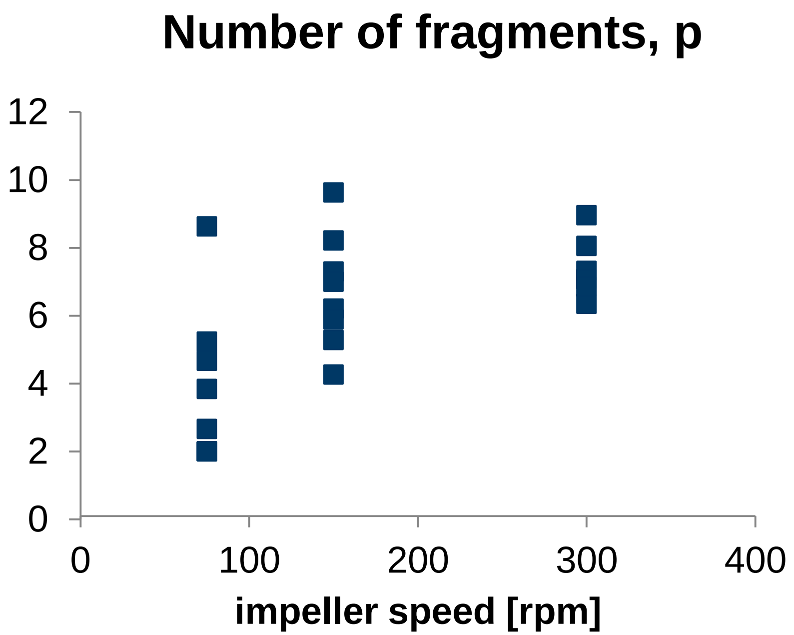


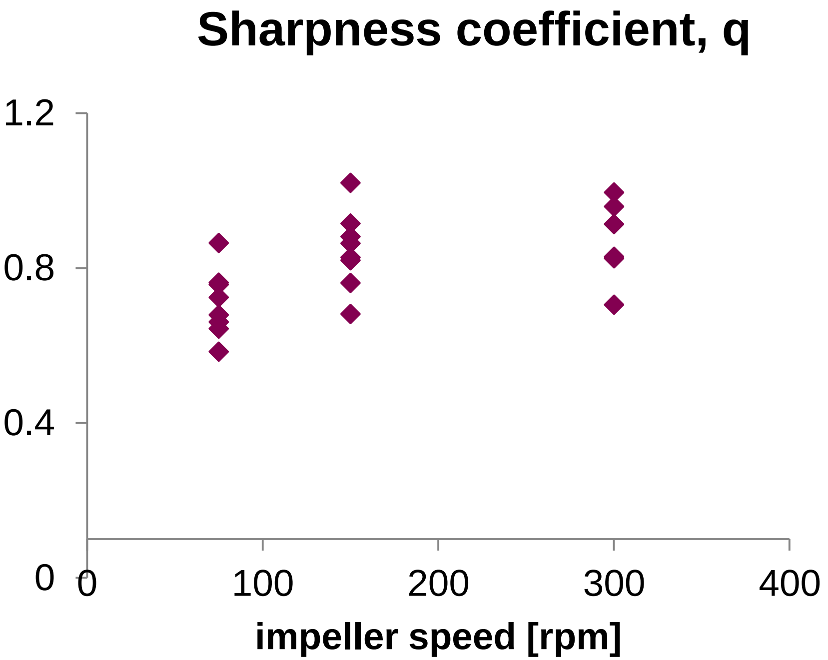
Parameter estimation for fragment distribution

Fragment distribution For fine mode: lognormal For coarse mode: Hill & Ng (1995)	$B = \frac{\zeta}{v\sqrt{2\prod}\ln\sigma}\exp\left[-\left(\frac{(\ln(v/\mu))}{\sqrt{2}\ln\sigma}\right)^2\right] + (1-\zeta)\left(\frac{p}{v}\right)\left(\frac{u}{v}\right)^{q-1}\left(1-\left(\frac{u}{v}\right)\right)^{r-1} / beta(q,r)$ $r = q(p-1)$

Parameters
Breakage rate constant, S_0
Critical fragment size, l_c
Fraction of fines, ζ
Geometric mean, μ
Standard deviation, σ
Number of fragments, p
Sharpness coefficient, q

Parameter p and q estimation with gSolids





With increasing impeller speed more fragments are produced per breakage event.

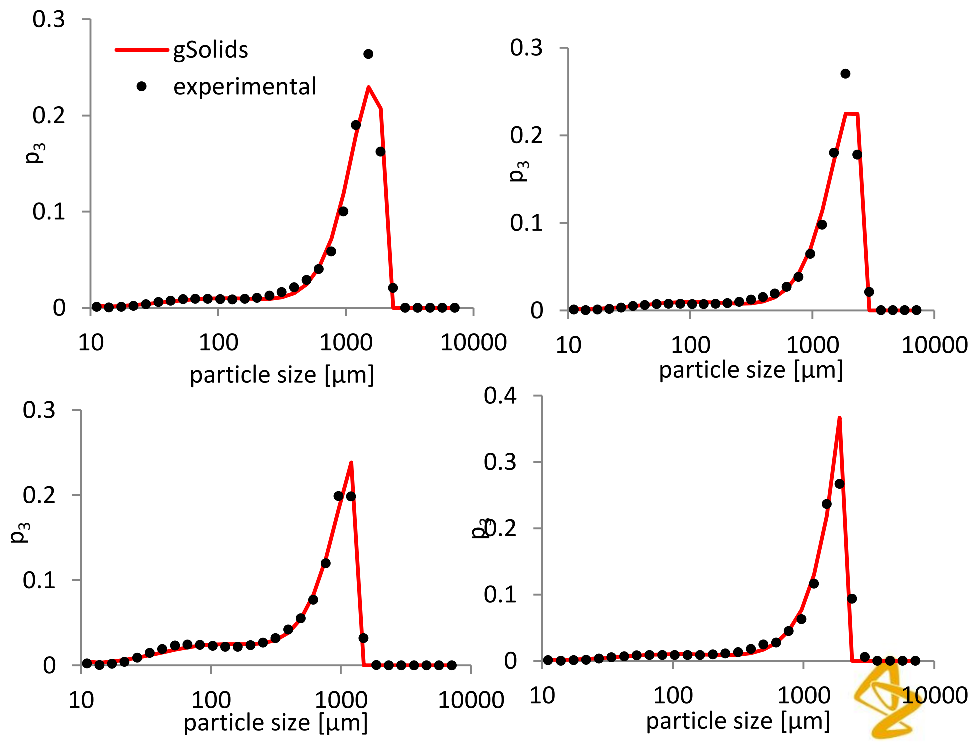
With increasing impeller speed breakage mechanism slightly changes toward fracture.

Population balance modelling with gSolids — conclusion

$$\frac{\partial n(u,t)}{\partial t} = \int_u^\infty b(u,v)S(v)n(v)dv - S(u)n(u)$$

Fragment distribution For fine mode: lognormal For coarse mode: Hill & Ng (1995)	$B = \frac{\zeta}{v\sqrt{2\Pi}\ln\sigma}\exp\left[-\left(\frac{(\ln(v/\mu))}{\sqrt{2}\ln\sigma}\right)^2\right] + (1-\zeta)\left(\frac{p}{v}\right)\left(\frac{u}{v}\right)^{q-1}\left(1-\left(\frac{u}{v}\right)\right)^{r-1} / beta(q,r)$ $r = q(p-1)$
Breakage rate cut-off	$S(l) = \begin{cases} 0 & l < l_c \\ S_0 & l > l_c \end{cases}$

Parameters	**Factors**
Breakage rate constant, S_0	Milling time
Critical fragment size, l_c	Screen size, impeller speed
Fraction of fines, ζ	Screen size, K_{IIC}, milling time, ribbon porosity, impeller speed
Geometric mean, μ	Geometric mean of initial powder
Standard deviation, σ	Standard deviation of initial powder
Number of fragments, p	Estimation with gSolids
Sharpness coefficient, q	Estimation with gSolids



Acknowledgements

Gavin Reynolds

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PSE

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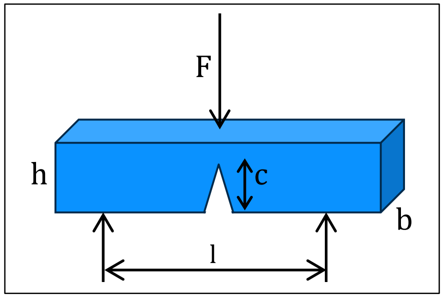
MARIE CURIE ACTIONS

Methodologies to measure tensile and shear fracture properties as a function of tablet porosity

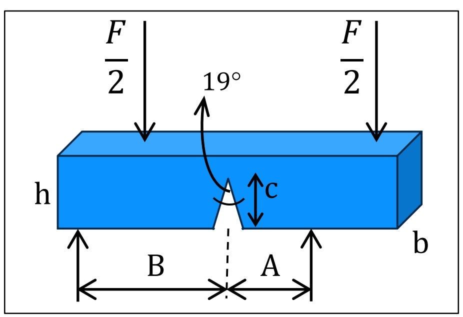
Method	Equation

Three point bend test – 3PBT



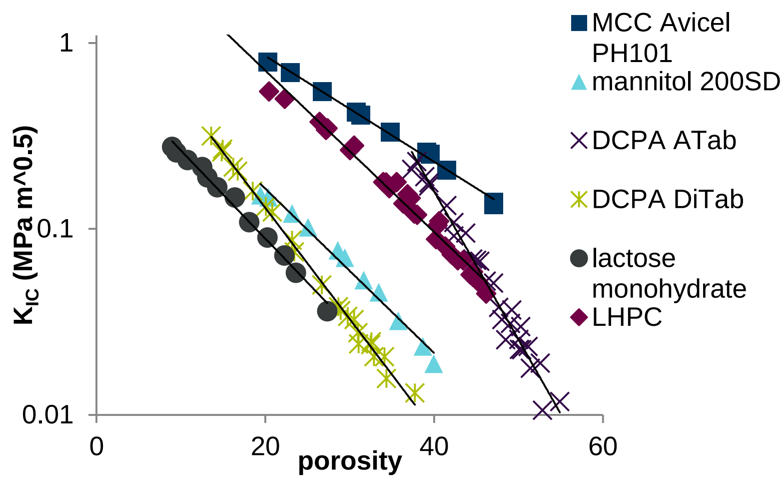
$$K_{IC} = \gamma_1 \frac{3Fc^{1/2}}{2bh^2 l}$$

Four point shear test – 4PST

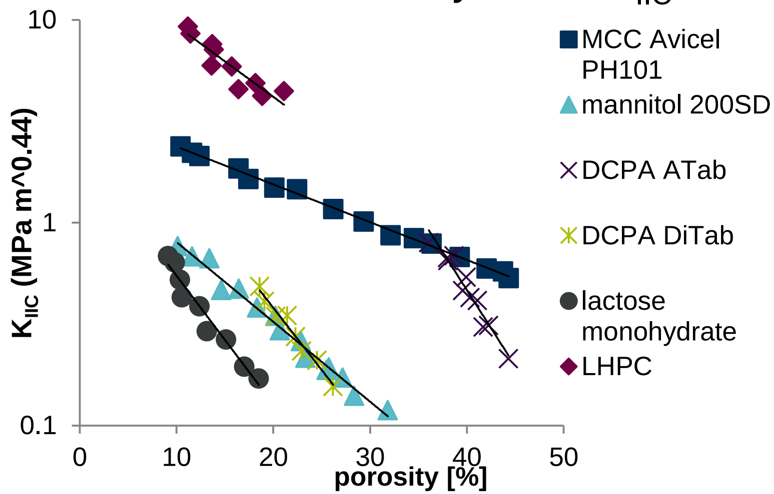


$$K_{IIC} = \gamma_2 \frac{F}{hb}\frac{B-A}{B+A} h^{1-\lambda^{II}}$$

Critical stress intensity factor-K_{IC}



Shear stress intensity factor-K_{IIC}

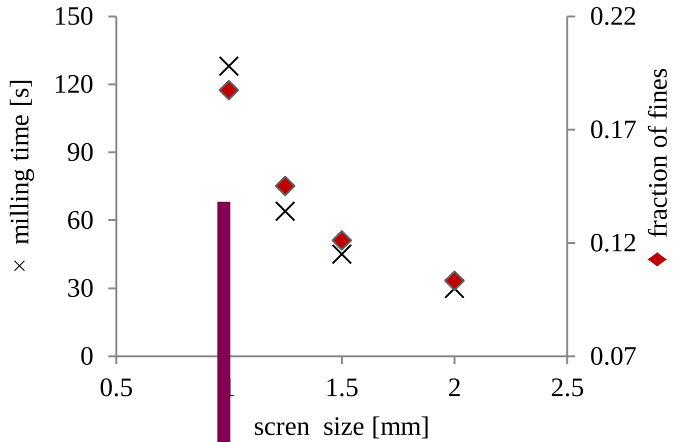


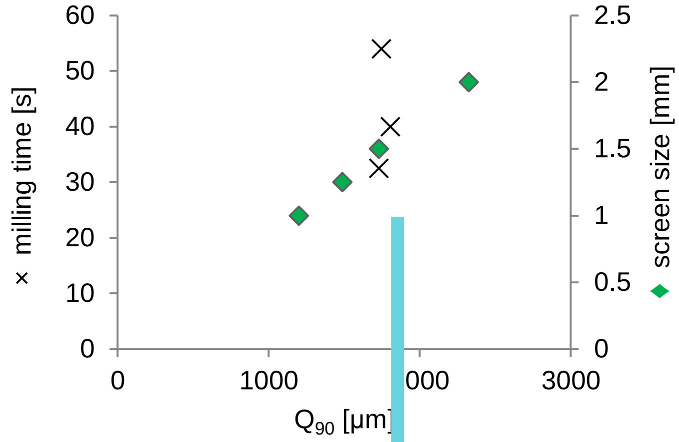
$$K_{IC} = K_{IC0} e^{-b\varepsilon}$$

$$K_{IIC} = K_{IIC0} e^{-b\varepsilon}$$

Material	$K_{IC,0}$	$K_{IIC,0}$
DCPA ATab	247	468.5
LHPC	5.3	/
MCC Avicel PH101	3.6	2
DCPA DiTab	2	6.4
Mannitol 200SD	1.1	3.6
Lactose monohydrate	0.8	1.6

Milling time and breakage mechanism





MCC Avicel PH101, ε = 31 %, 150 rpm impeller speed.

Abrasion breakage is more pronounced when using smaller screen sizes and it takes longer time to mill the ribbons with abrasion compared to impact breakage.

Different occupancy of mill chamber doesn't affect the granule size distribution.