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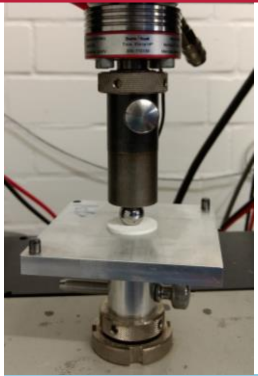
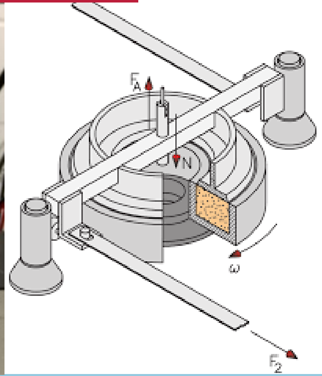
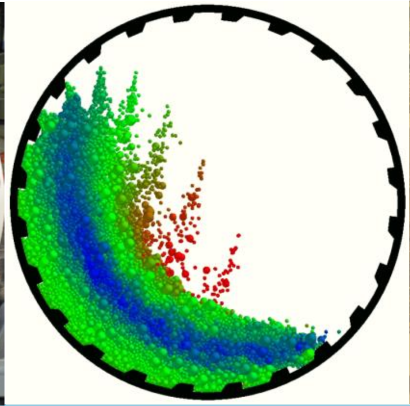






A Systems Engineering Approach to Dry-Milling with Grinding Aid Additives

2nd funding period

Tarek Sulaiman, Anderson Chagas, Arno Kwade

Project introduction

Long term objectives:

I. Obtain qualitative/quantitative effects of grinding aid additives on material behaviour, process aspects and energy flows.

II. Develop a system engineering approach for optimizing and scaling industrial dry grinding processes.

Second phase (3 years period):

I. Determine a proper powder microscopic property to represent the combination of material and GA type and dosage

II. Relate measurable microscopic particle properties with powder bulk behavior

III. Design a characterization procedure to obtain the model parameters required for axial transport simulation

IV. Model powder internal axial transport during milling

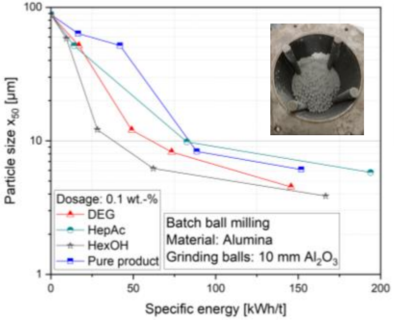
V. Validate all developed models and flowsheet simulation with industrial data

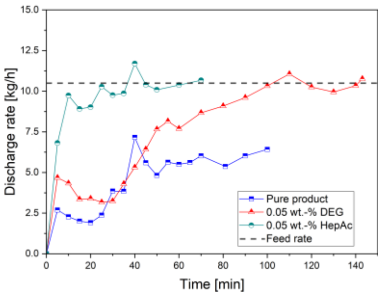
1st phase – System engineering approach

WP 1

Particle stressing

- Grinding aid mechanism
- Powder bed stressing





Ball mill process

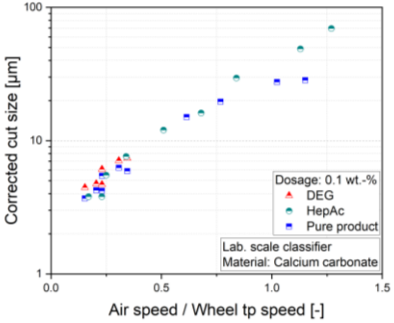
- Powder transport
- Energy consumption

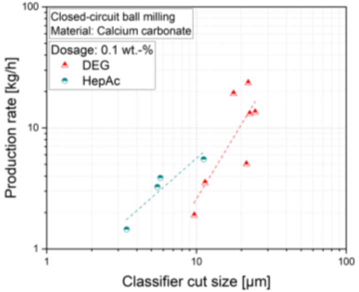
WP 2

WP 3

Size classification

- Classifier efficiency





Milling circuit

- Powder recirculation
- Plant production

WP 4

Effects of grinding aids GA
Summary 1st phase

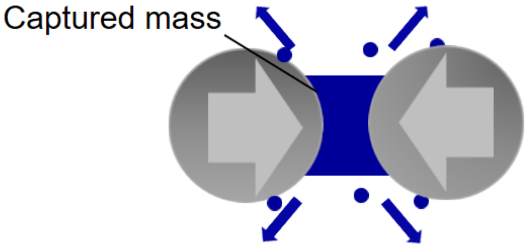
Micro scale

Stabilization against agglomeration

- As thickness of liquid GA < 1 nm, v. d. Waals are only slightly affected
- GA decrease **surface energy** & adhesion forces
- Agglomeration state affects **classifier performance**

Meso scale

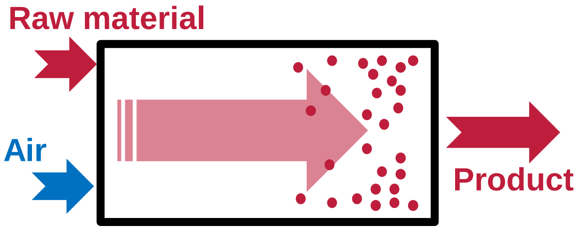
Particle capturing between balls



- **Flowability** and **captured mass** determines stress energy and by that energy efficiency and particle size distribution width

Macro scale

Material transport and mill hold-up



- Flow velocity and by that **hold-up** and **mean residence time** depend strongly on flowability

2nd phase – system engineering approach

WP 1

From Micro to Macro

- Relation between microscopic particle and macroscopic powder properties

WP 2

Powder Transport

- Modeling axial material transport
- Obtain model parameters

WP 3

Breakage and PBM

- Implementing previous WPs in flowsheet simulation

WP 4

Validation

- Flowsheet simulation with industrial plant data

Particle stressing: Grinding aid mechanism

WP 1

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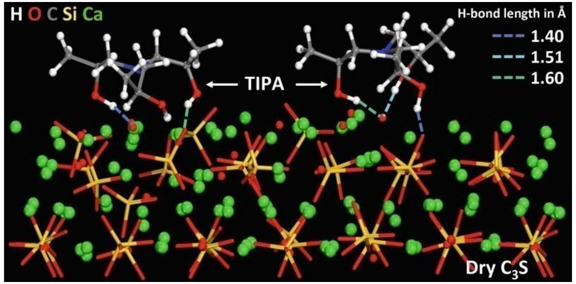
Grinding aid molecules:

- Heptanoic acid (**HepAc**)
  - Chemical formula: $C_7H_{14}O_2$
- Diethylene glycol (**DEG**)
  - Chemical formula: $C_4H_{10}O_3$
- 1-Hexanol (**HexOH**)
  - Chemical formula: $C_6H_{14}O$



[1]

Mechanism of action:

- Homogeneity of **mono-molecular layer of G.A.** on particle surface
- Polar groups of G.A. connect to OH-groups of particle surface, thus **avoiding a interaction of OH-groups of the particles** with each other
- Non-polar groups of G.A. protrude outwards and **decrease surface energy**, and thus, adhesion forces
- G.A. can make **particle surface hydrophobic**, thus decreasing adsorption of water

[1] Mishra, Flatt, Heinz. J. Phys. Chem. C 2013, 117, 10417−10432

2nd phase - Micro to Macro

WP 1

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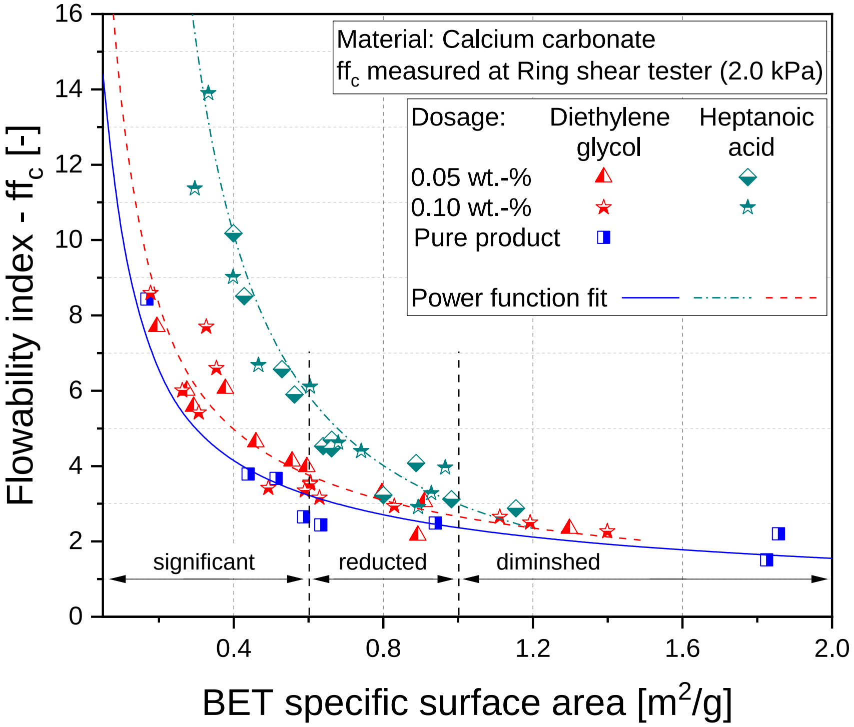
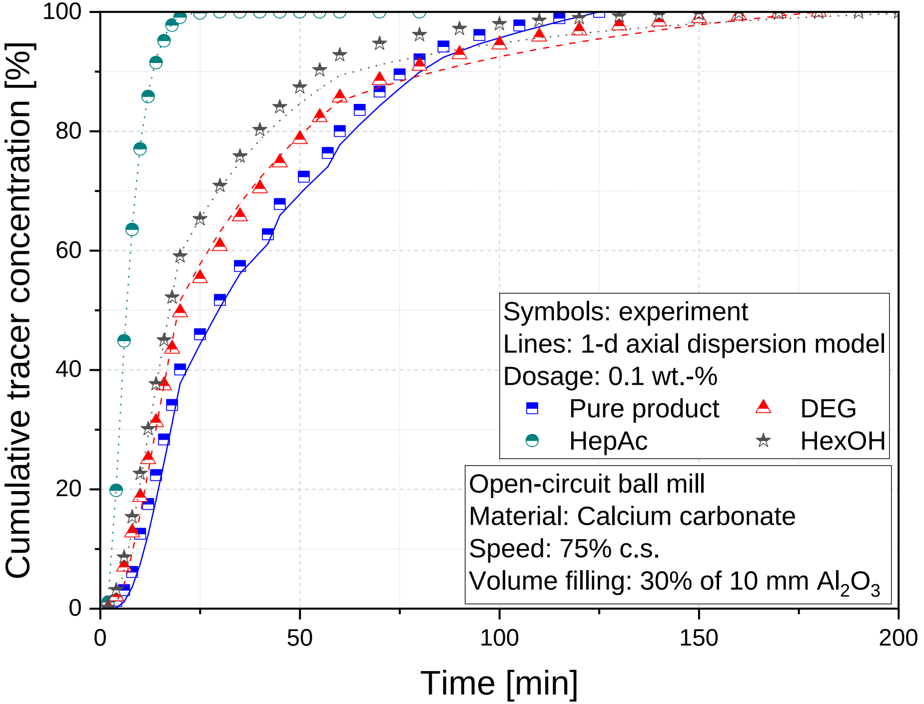


- As expected, flowability is a function of both particle size (or surface area) and grinding aids
- A **power law** relates flowability to the combination of powder surface area and type of grinding aid
- Once pre-characterized, a size population balance model can **dynamically estimates the changes in flow properties** during grinding



- On continuous ball milling, the main impact of grinding aids is on flow accross the mill
- G.A. such as glycol, presents minimal impact on flowability. However, finer products to be achieved without mill clogging
- G.A. such as Heptanoic acids, retards the loss in flowabilty with product size reduction. Allowing the product to flush throught the mill with less size reduction.

2nd phase - Powder Transport
Material hold-up

WP 2

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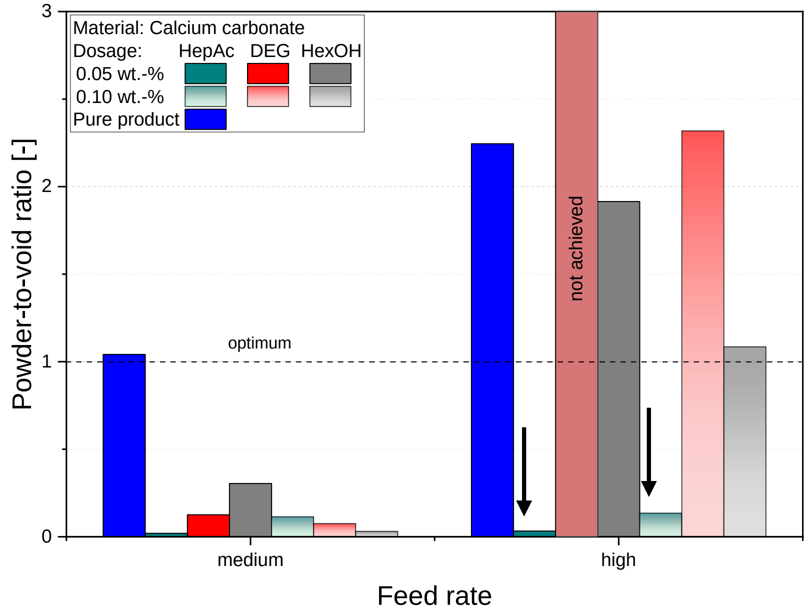

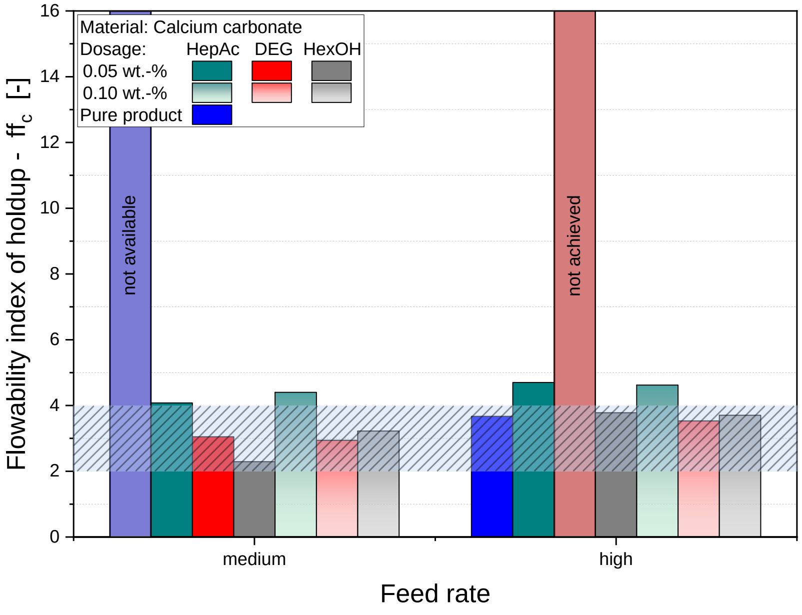

A steady-state operation of the mill within the range of desired powder-to-void ratio of 1 was achieved for ff_c-values 3-4, but for this ff_c-values also higher holdups are achieved

2nd phase - Powder Transport
Material hold-up and residence time

WP 2

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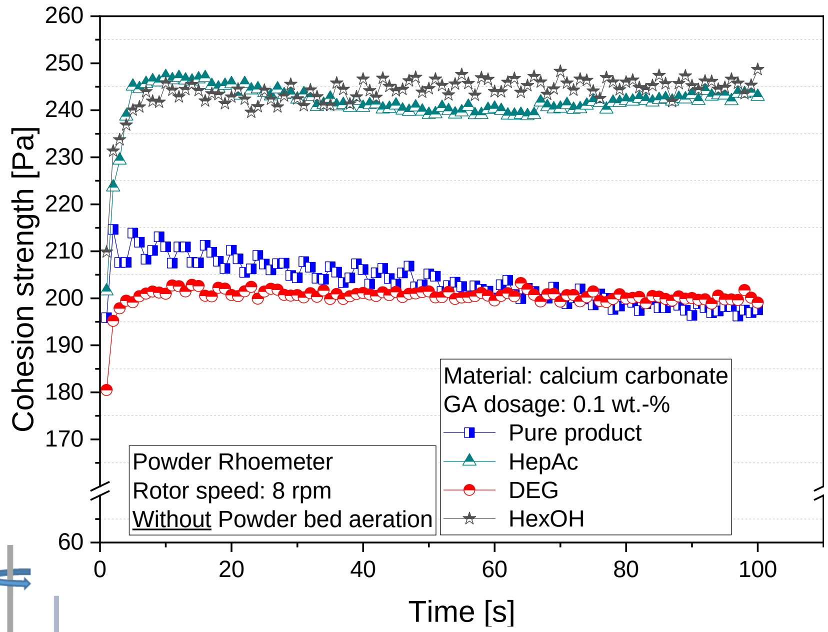
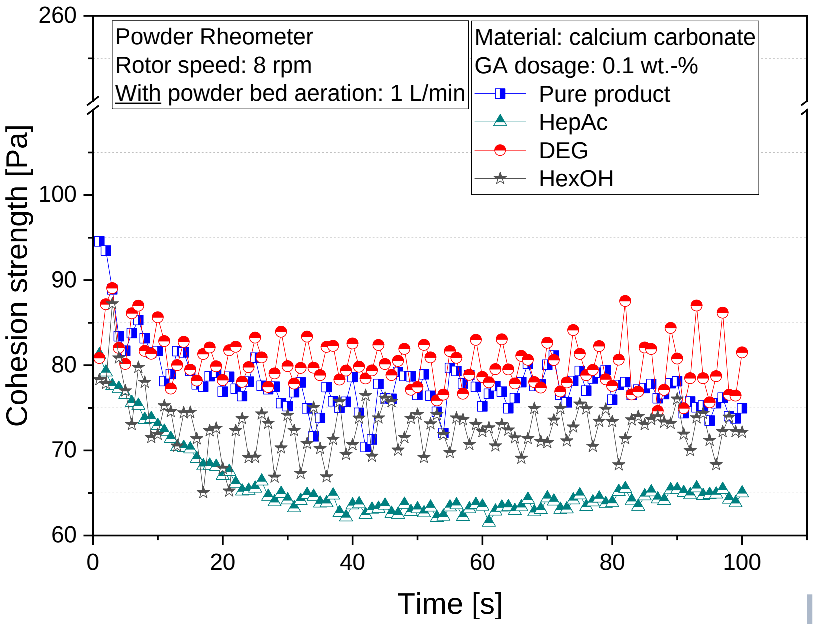
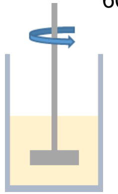
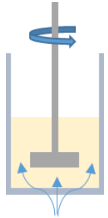






$$Cohesion\ strength\ [Pa] = Torque\ [Nm]\ x\ Calibration\ factor\ [\frac{Pa}{Nm}]$$



2nd phase - Powder Transport
Material hold-up and residence time

WP 2

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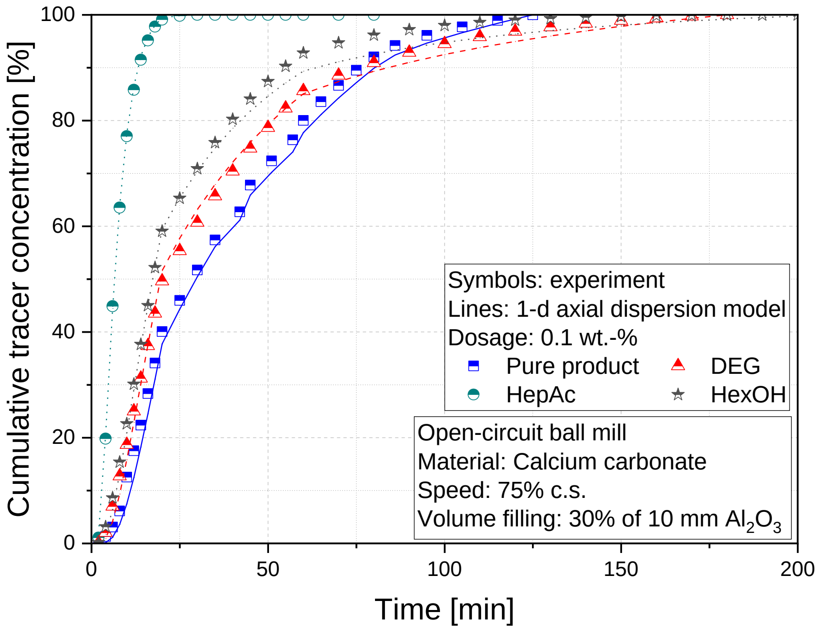
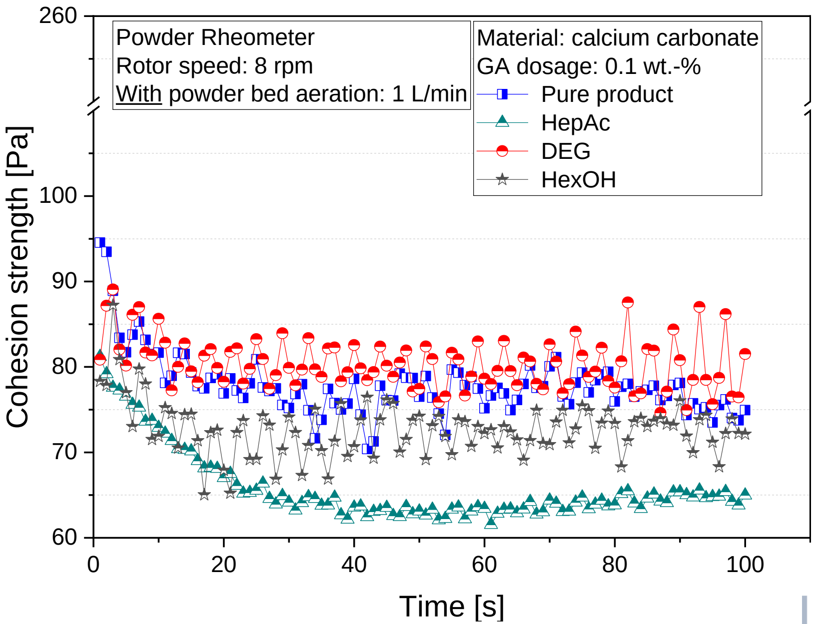
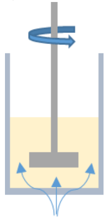






The cohesion strength described at a aeration flow rate of 1 L/min describes well the residence time behaviour of the powders with different grinding aids

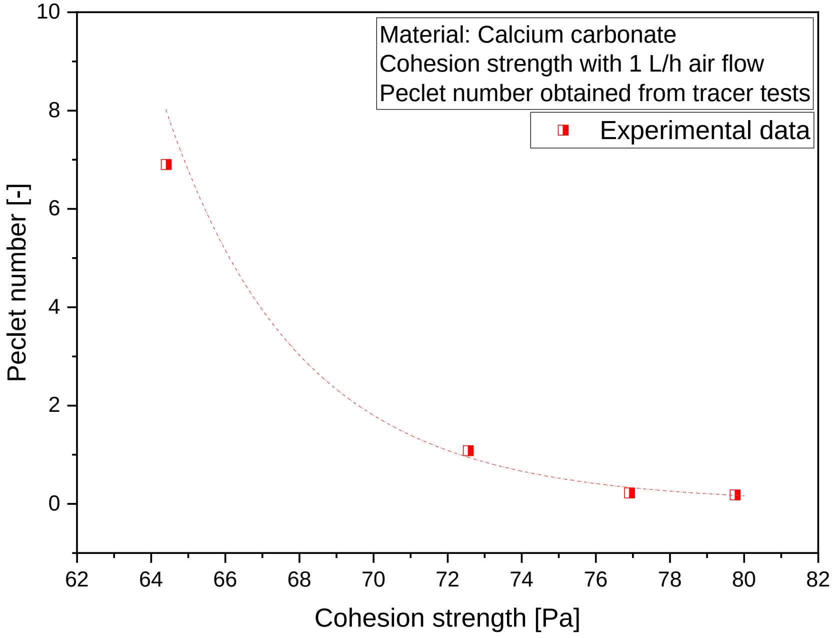
Transport model

- **One-dimensional axial dispersion model**

$$\frac{\partial Q}{\partial t} = D_x \frac{\partial^2 Q}{\partial x^2} - u_x \frac{\partial Q}{\partial x} \qquad Pe = L\frac{u_x}{D_x}$$

$$Q(t) = \frac{1/\theta}{2\sqrt{\pi \cdot Pe \cdot \left(t/\theta\right)^3}} exp\left[-\frac{\left(1 - t/\theta\right)^2}{4 \cdot Pe \cdot \left(t/\theta\right)}\right]$$

One parameter model



2nd phase - Powder Transport
Obtain model parameters

WP 2

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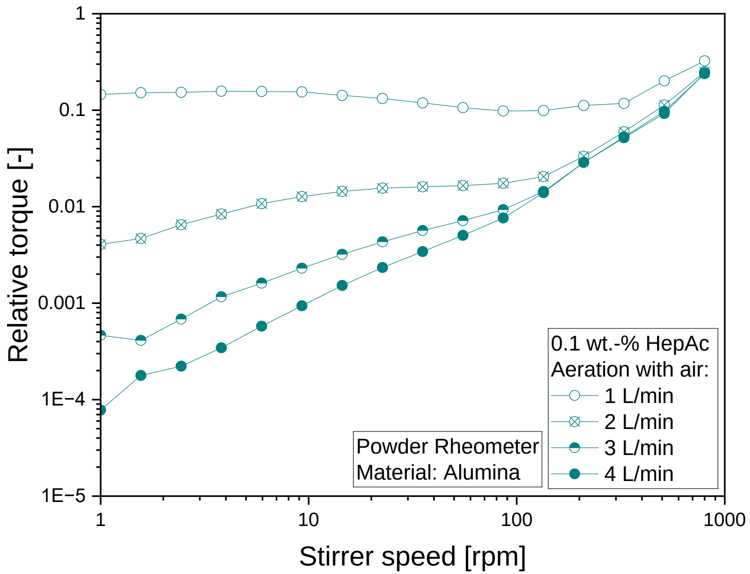
	no GA	HepAc	DEG	HexOH
Holdup [%]	224.5	13.42	231.85	108.5
Mean RT [min]	38.74	8.52	35.65	27.62
ff_c [-]	16.48	11.15	7.8	13.71
CS [Pa] 0 L/min	199.25	241.55	200.21	245.41
CS [Pa] 1 L/min	76.91	64.41	79.76	72.56



Dynamically obtained bulk powder properties represent better property for estimating the material hold-up and residence time

2nd phase - Breakage and PBM

WP 3

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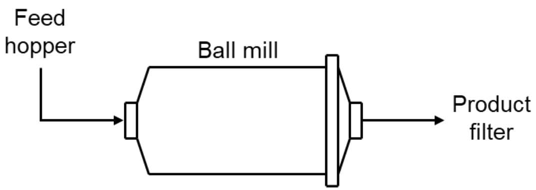
PBM transport simulation end of 1st phase:

- **Static flowsheet inputs:**
  - Feed rate
  - mill holdup and residence time
  - Material flow parameters

- **Limitations end of 1st phase:**
  - Increase in cohesion is not simulated
  - Material flowability is constant



Recent improvements:

- **From Micro to Macro**
  - Accounting for change in flowability through the relationship between the specific surface area and flowabilty index

- **Material transport**
  - Adopting the 1-d axial dispersion model
  - Obtain model parameter from dynamic-al measuring technique of powder prop-erties

2nd phase - Validation

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WP 1

From Micro to Macro

WP 2

Powder Transport

WP 3

Breakage and PBM

WP 4

Validation

- Validation with industrial plant data
- Product sampling rounds would be conducted to obtain
  - Product size and flow rates
  - Energy consumption
- Product sample will be characterized and the process simulated in a flowsheet tool

Acknowledgements

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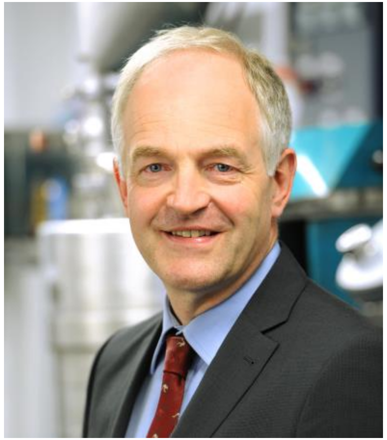

Prof. Dr. -Ing. Arno Kwade

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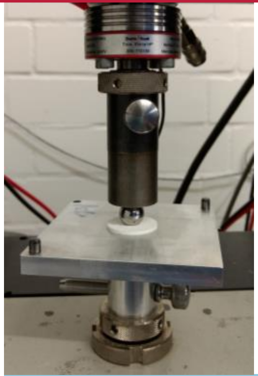
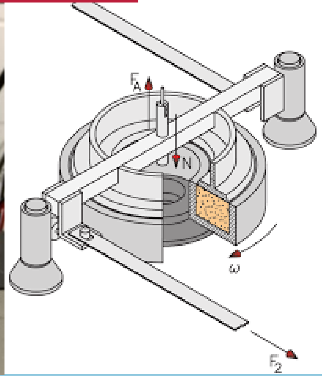

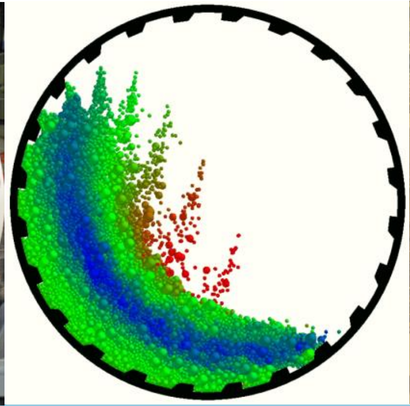


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