Technische Universität Braunschweig

iPAT Institut für Partikeltechnik

Grinding and Dispersing of Nanoparticles in aqueous suspensions

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Possibilities for Nanoparticle production

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Top-Down Processes

Real Grinding

Laser Fragmentation

Input of Energy:

Stirred Media Mill
(Laser)
Kneader, Three-Roller-Mill,
Dissolver….

Bottom-Up Processes

Sol-Gel Syntheses

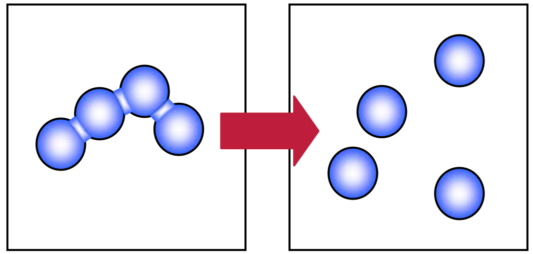
Precipitation

Pyrolyses

Often: (Re-)dispersing

List of contents

- Dispersing of nanoparticles
  - Stress mechanism and Machines
  - Experimental results and theoretical discribtions



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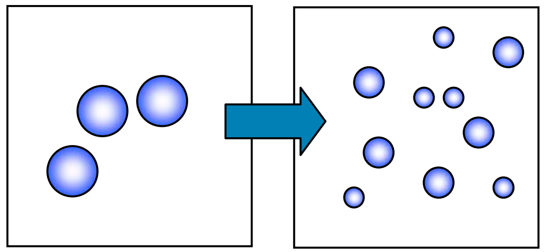
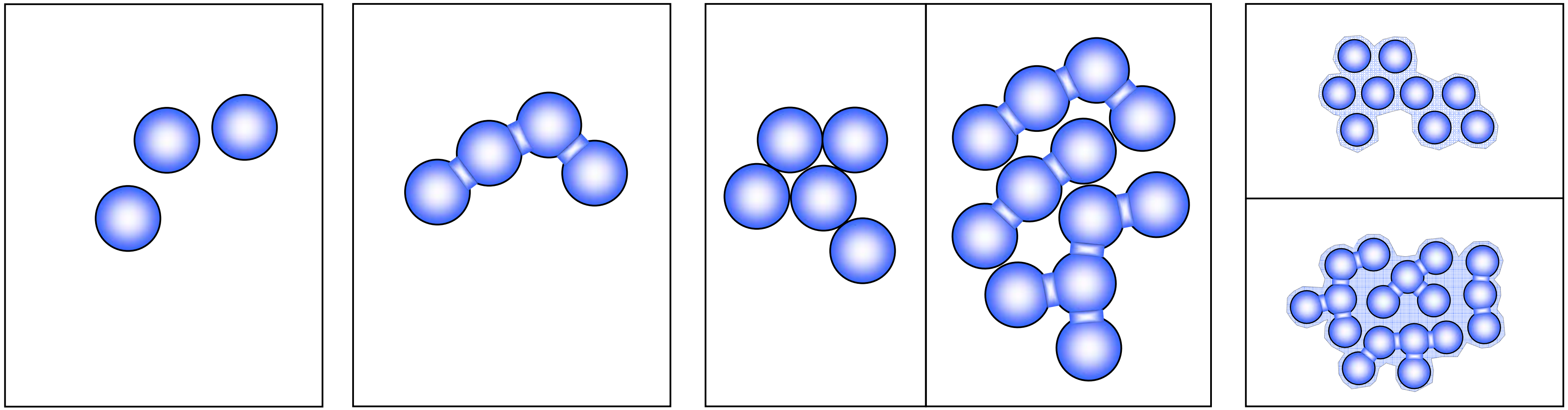
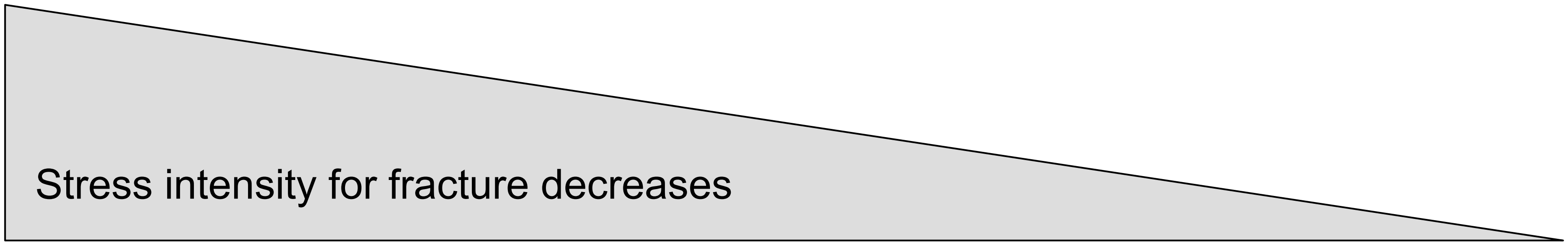
  - Influencing parameters
  - Results with different stabilisation additives
  - Modell transfer from dispersion processes



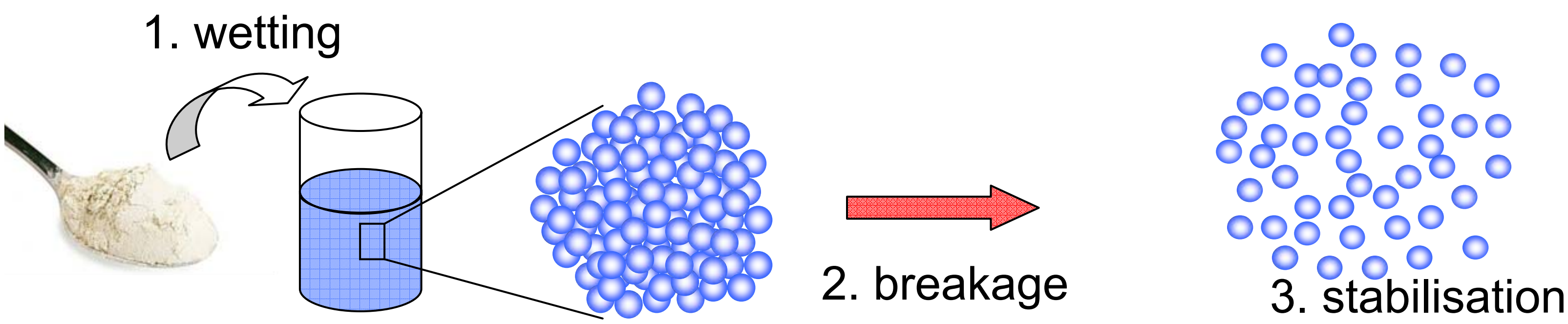
Particle Structures

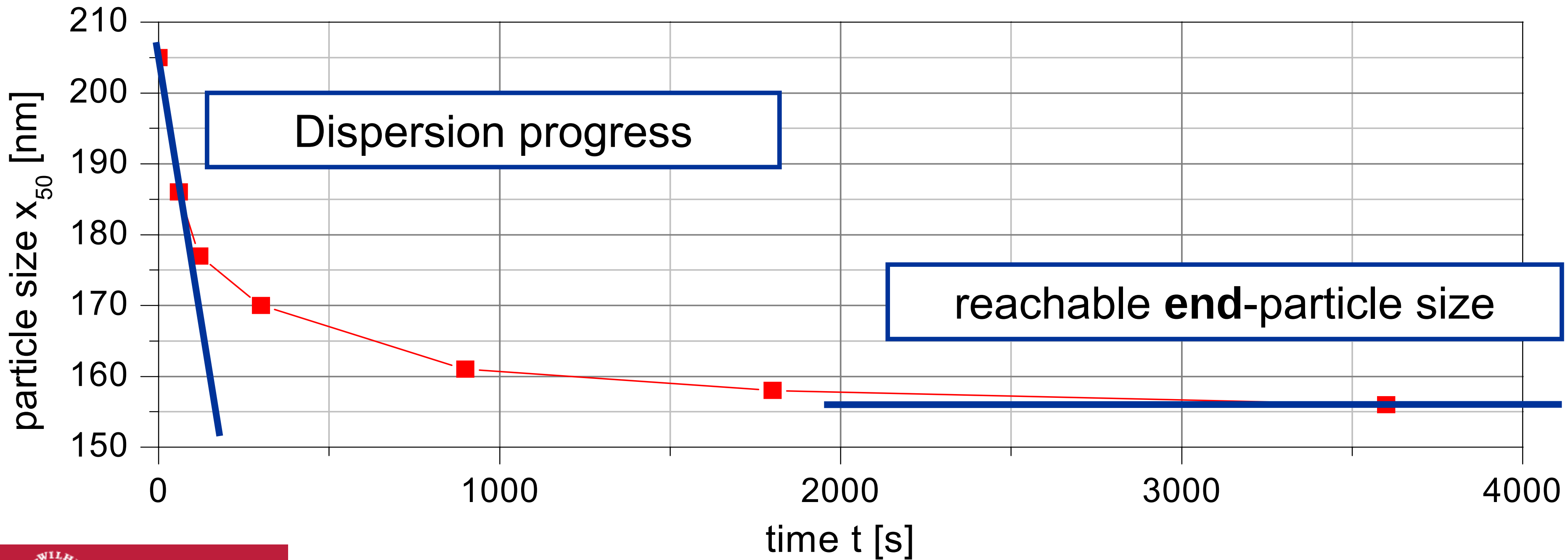


Primary particles Aggregates Agglomerates Flocculates



Dispersion process

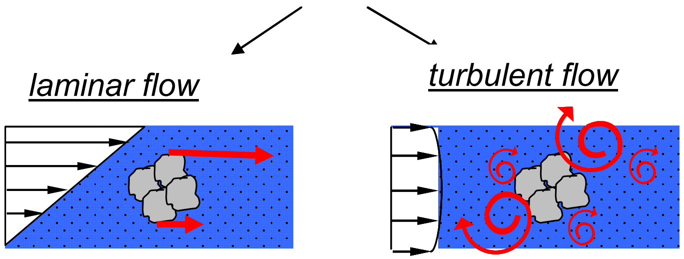




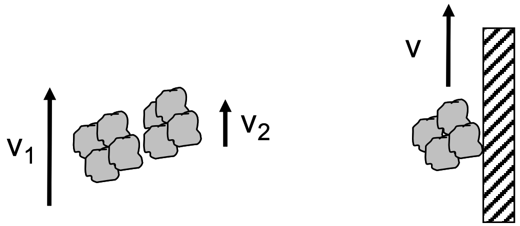
Stress mechanism

Shear stress

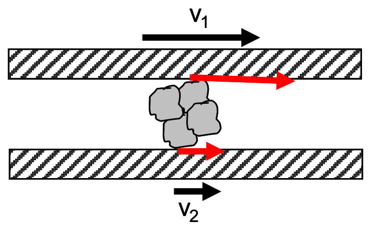
a) by means of a fluid



b) by shearing on one surface

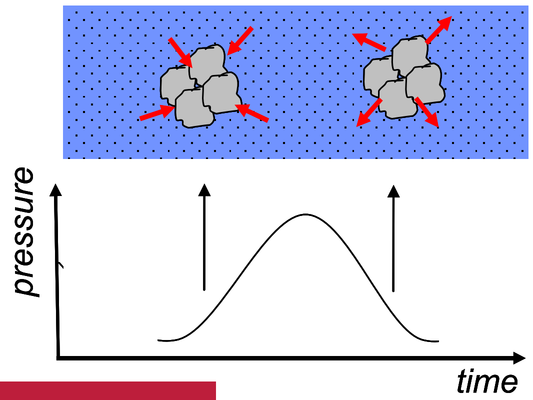


c) between two surfaces

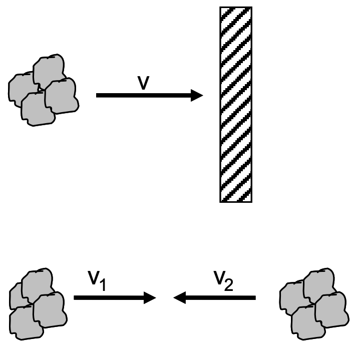


Compressive stress

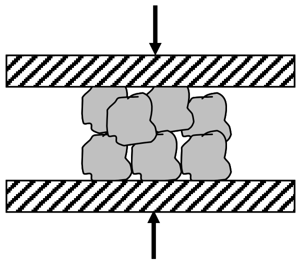
a) by means of a fluid



b) by impact on one surface



c) between two surfaces



Dispersion machines

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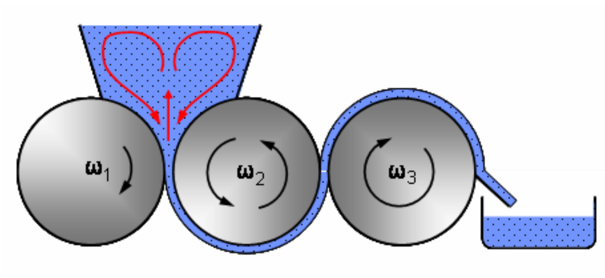
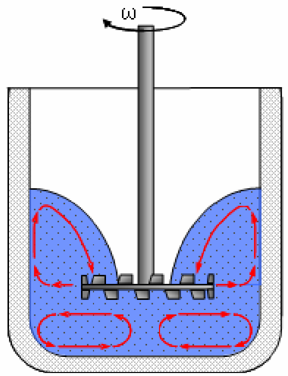
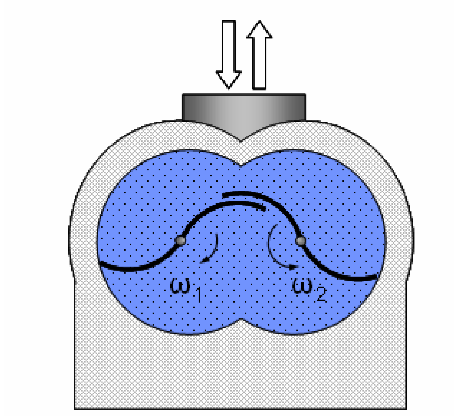


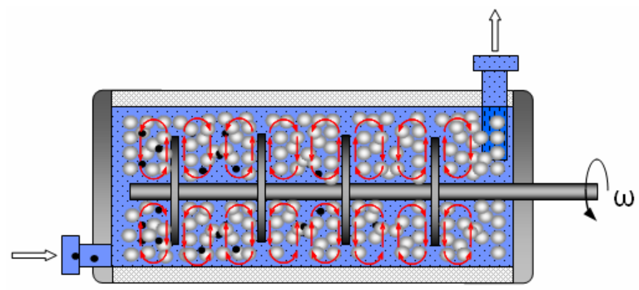
3-roller-mill



dissolver



kneader



stirred media mill

Comparison of different dispersing machines

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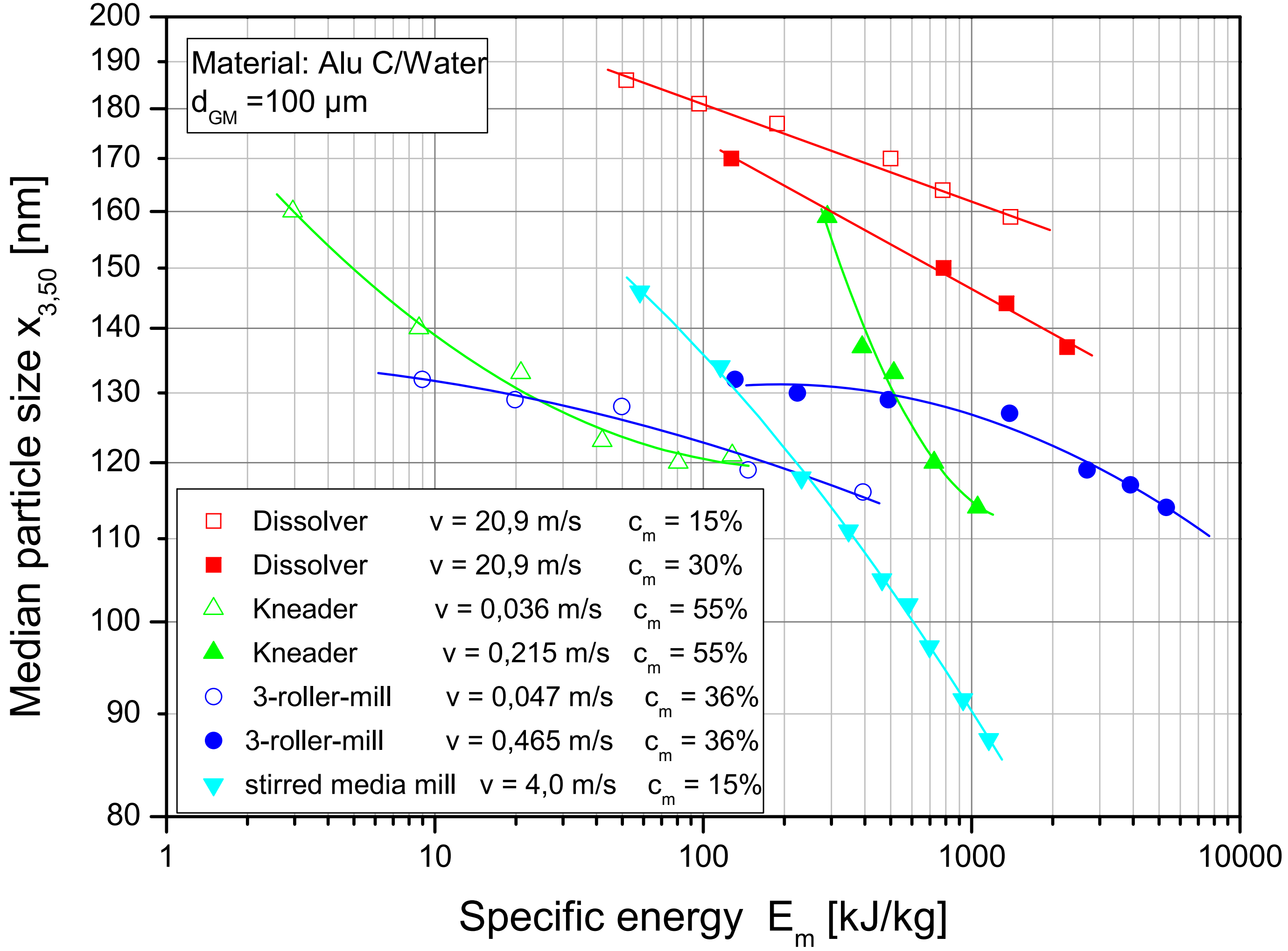

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Dispersion kinetics – dissolver

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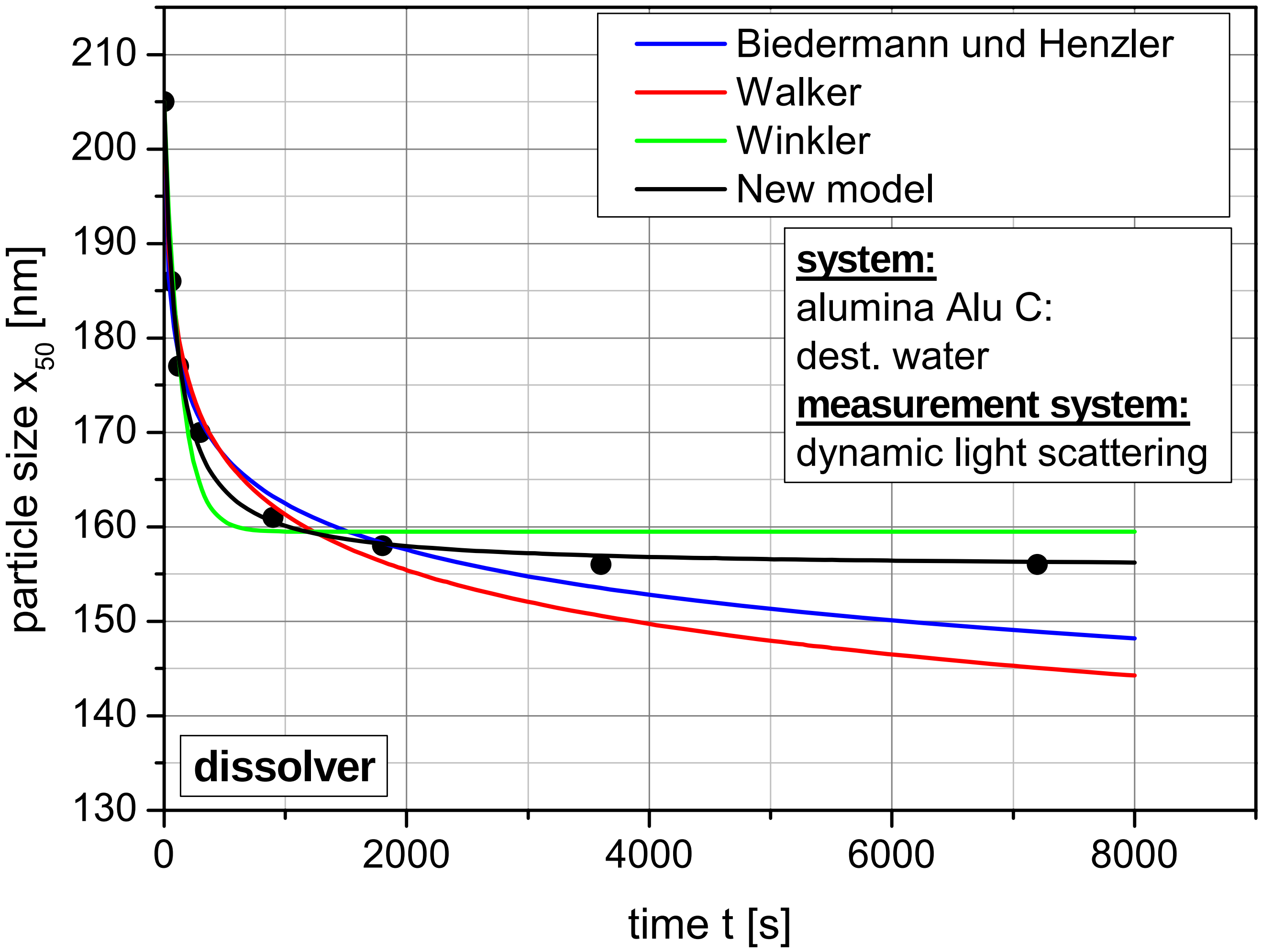


Biedermann und Henzler:

$$x = C \cdot \left(1 + A \cdot a \cdot \left(\frac{P}{V}\right)^b \cdot t\right)^{-\frac{1}{a}}$$

Walker:

$$\int_0^{t_1} t = -C \cdot \int_{x_0}^{x_1} \frac{dx}{x^n} \quad \Rightarrow \quad x_1 = \left(\frac{t \cdot (n-1)}{C} + \frac{1}{x_0^{n-1}}\right)^{-\frac{1}{n-1}}$$

Winkler

$$p_{DA} = p_{Beanspruchung} \cdot p_{Bruch} \Rightarrow x = x_0 - x_0 \cdot \left(1 - e^{-k \cdot \frac{V_{eff}}{V} \cdot t}\right) \cdot \left(1 - e^{-a \cdot \frac{P}{\sigma \cdot V}}\right)$$

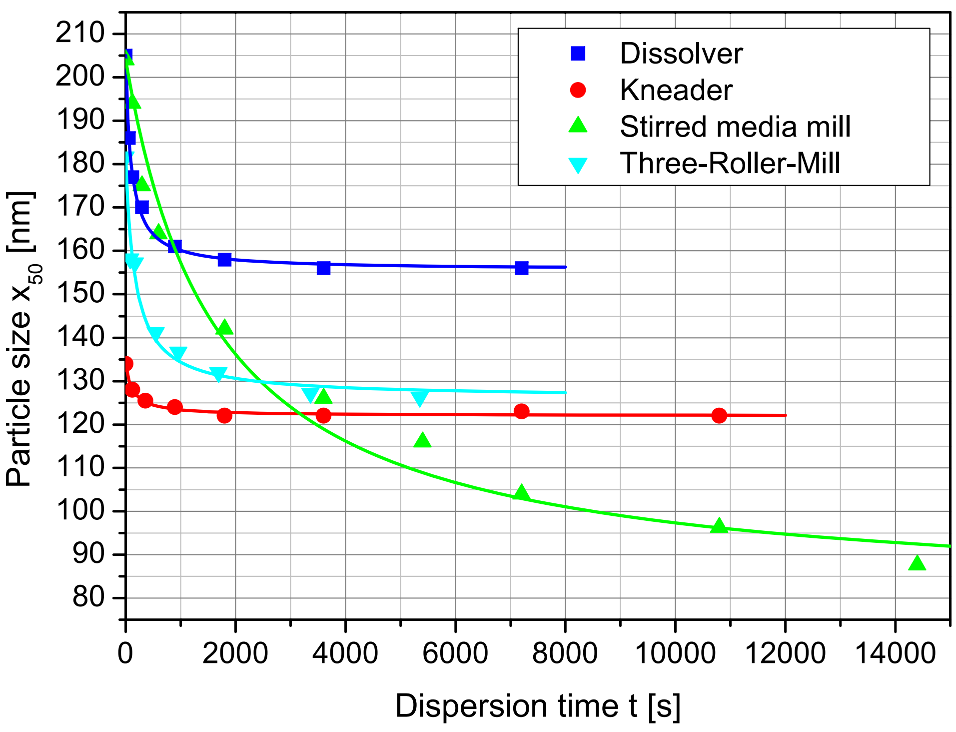
Dispersion kinetic – new model

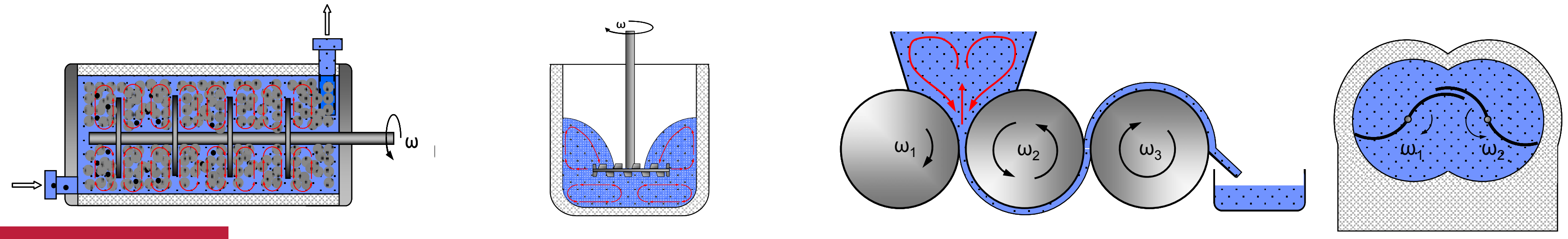
$$x(t) = x_0 + (x_{end} - x_0) \cdot \frac{t}{t + K_t}$$

$$x(E_m) = x_0 + (x_{end} - x_0) \cdot \frac{E_m}{E_m + K_E}$$

Dispersion process and kinetics

- For Dispersion processes a reachable end particle size can be determined by a kinetec model
- The model uses stress intensity and stress frequency for the describtion of the process
- A comparison of different dispersion machines and process parameter in relation to their stress intensity and frequency is possible





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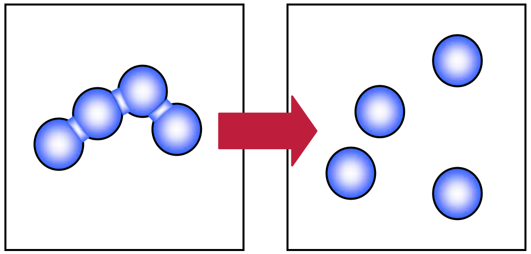

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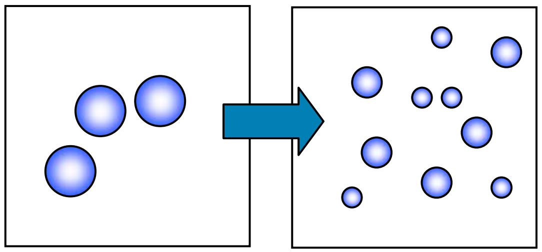

What are the challenges of nanogrinding?

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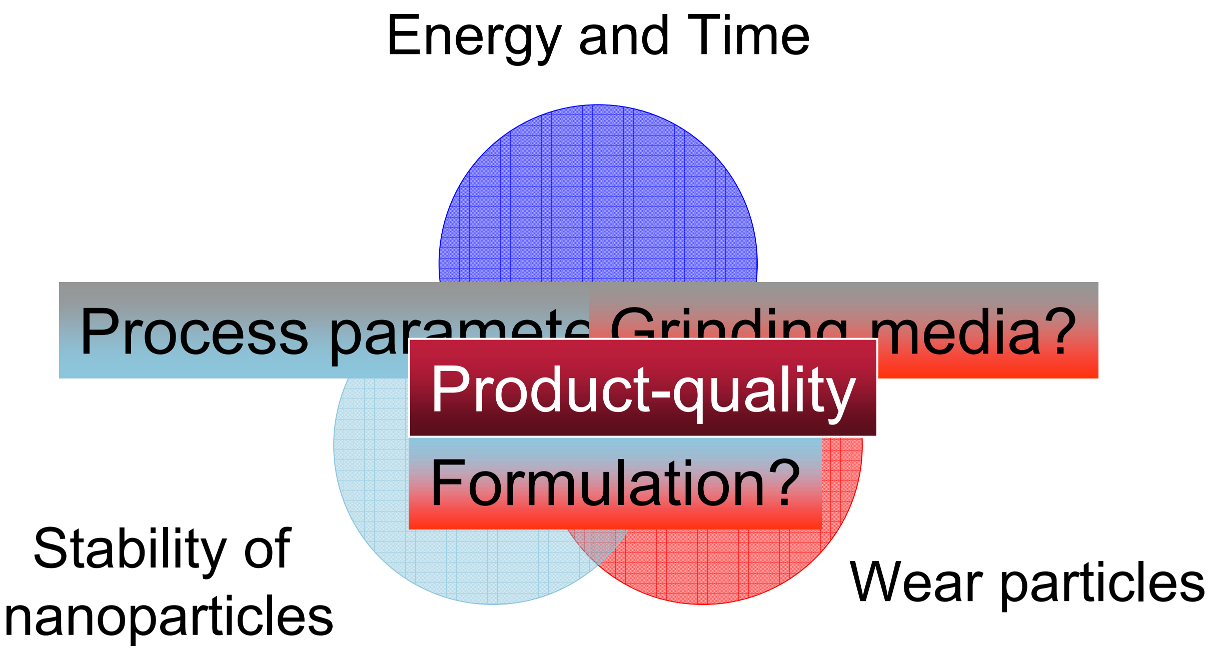
Nanoparticles tend to agglomerate, due to high surface forces

Wear particles lead to product contamination and high costs for the replacement of grinding media

The process requires a high energy input and is time consuming



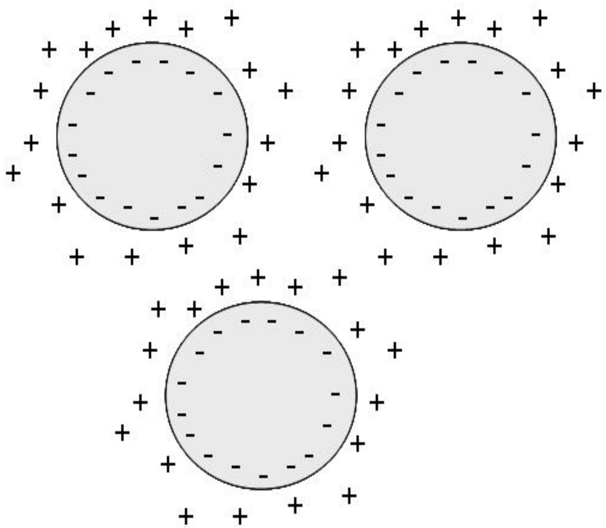
Stabilizing possibilities

Electrostatic Stabilization

Addition of potential determined ions ➔ Zeta-Potential

Acids/Basis ➔ pH-value

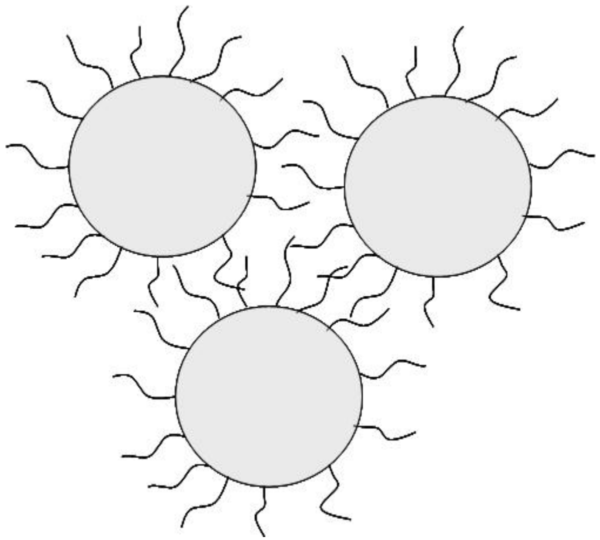
Pe
Nit
Hy
Ac
Ph



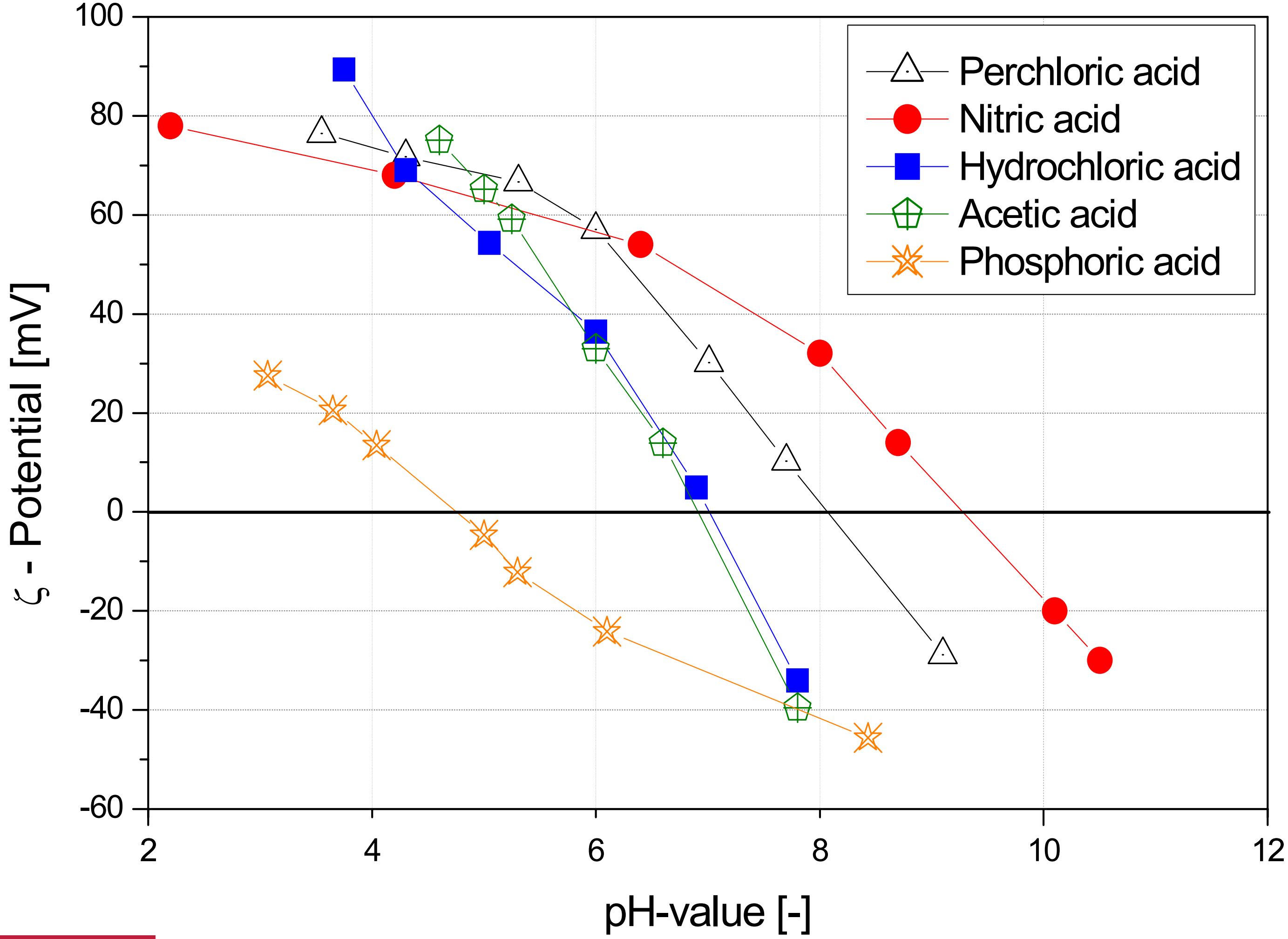
Steric Stabilization

Addition of Oligo-/Polymers ➔ Adsorptionsisotherms/Rheology

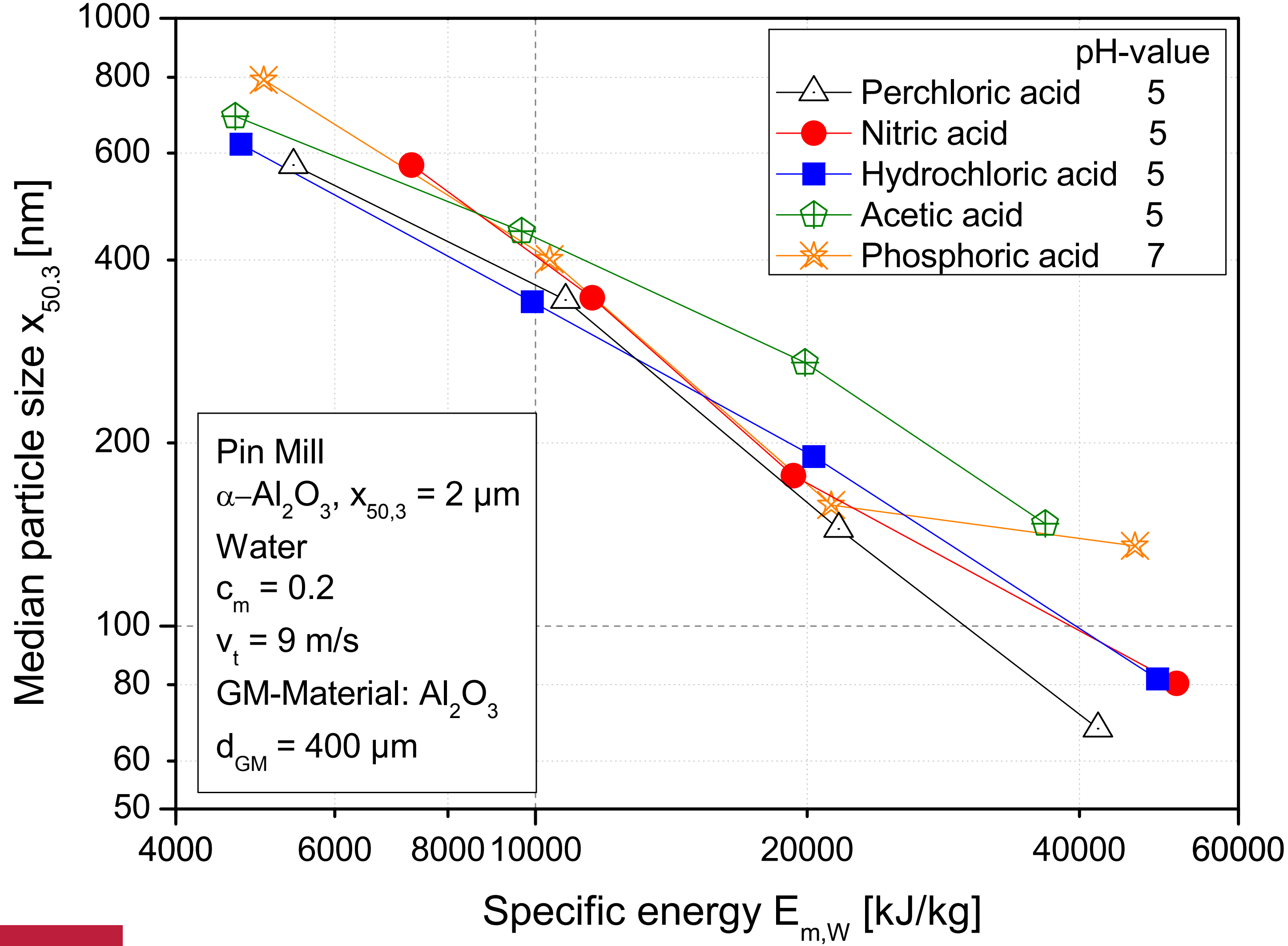
Functional groups



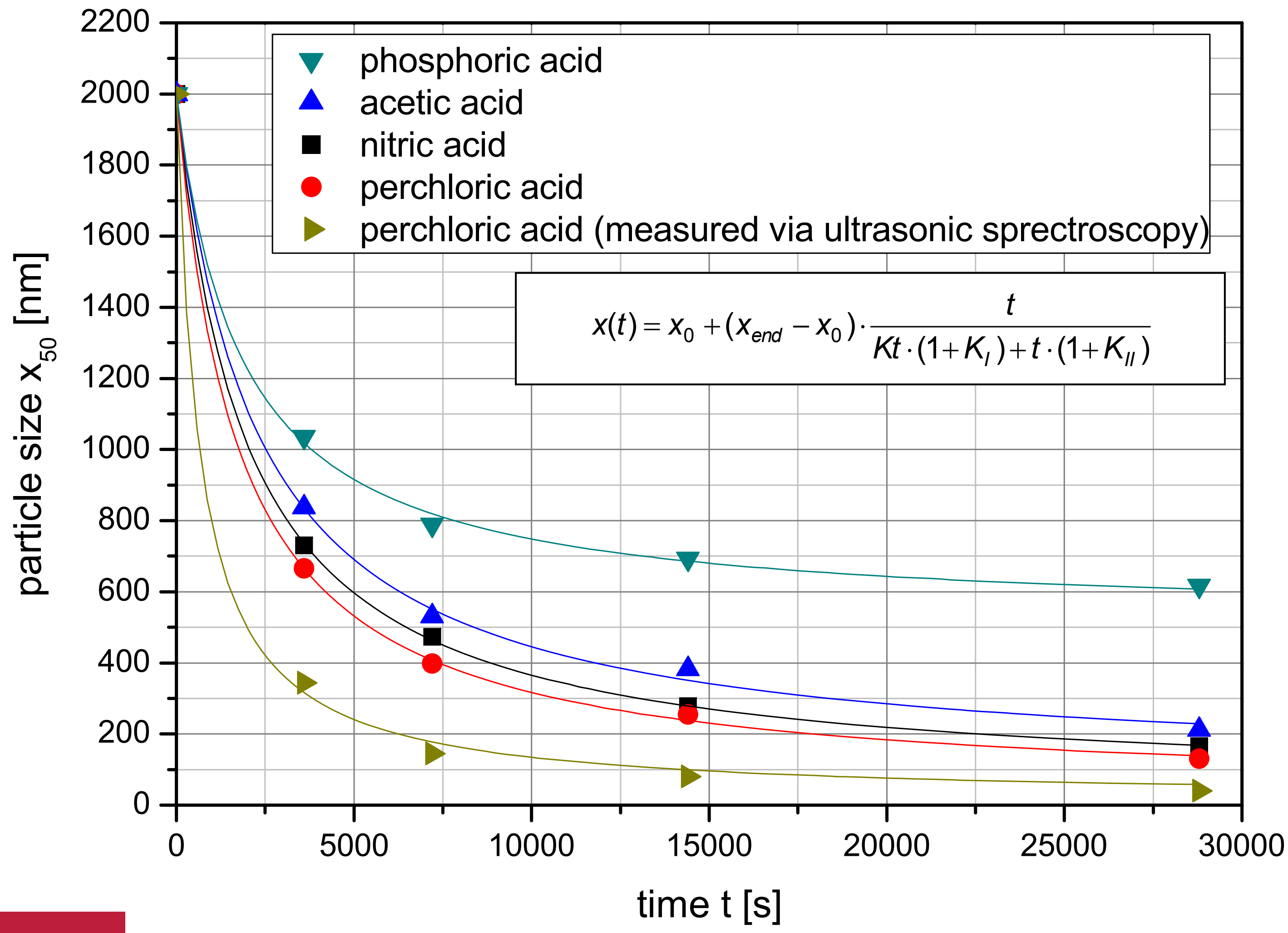
Determination of isoelectric point



Nanogrinding with different acids



Transfer of the new dispersion modell to a grinding process



Conductivity in dependence of particle surface area

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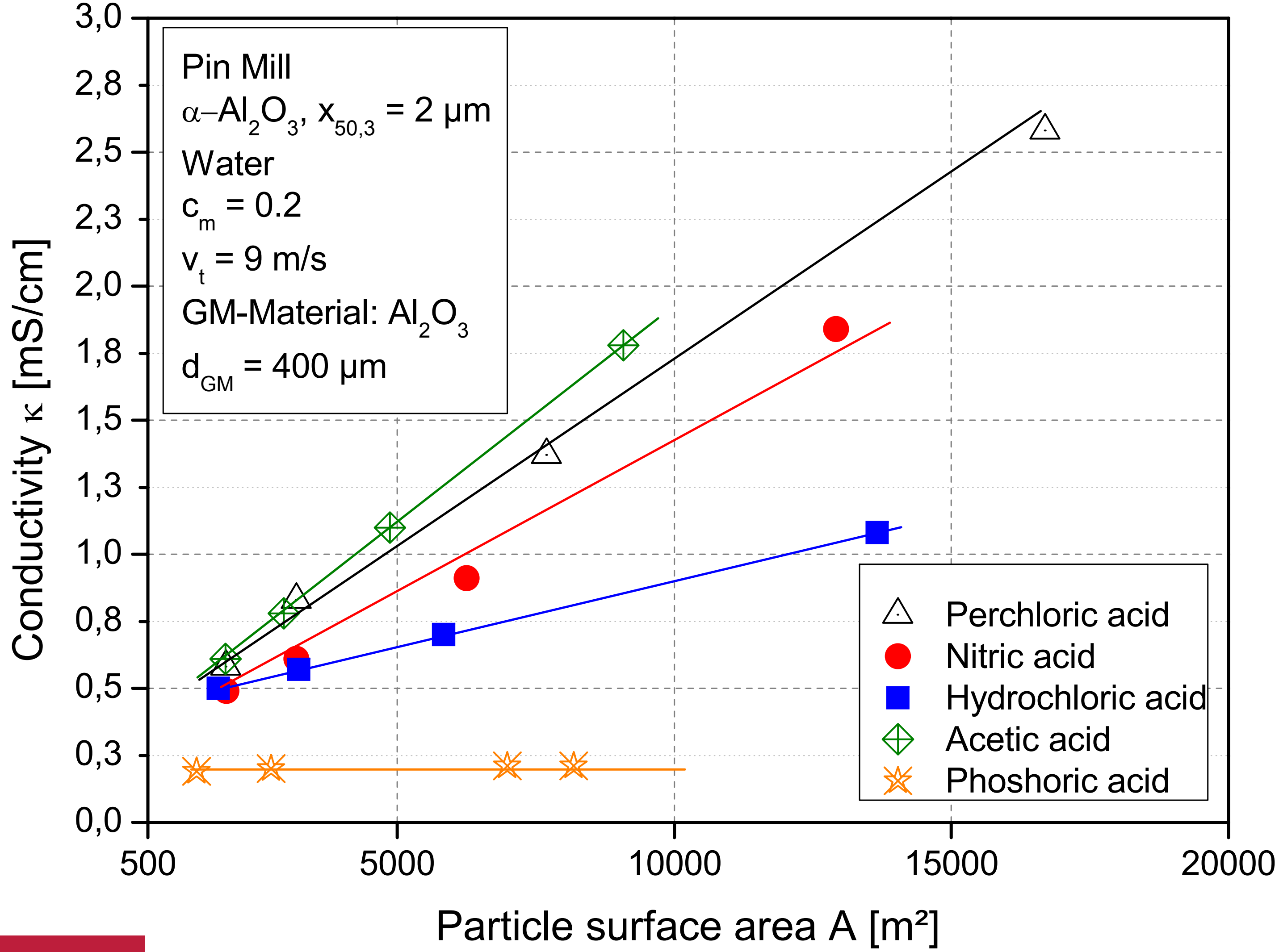
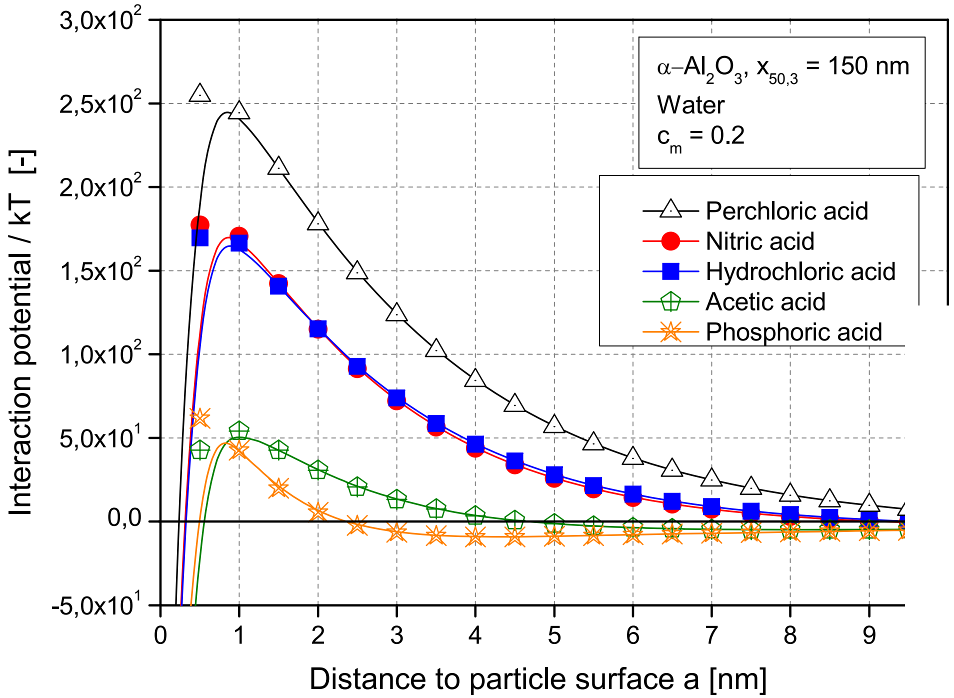
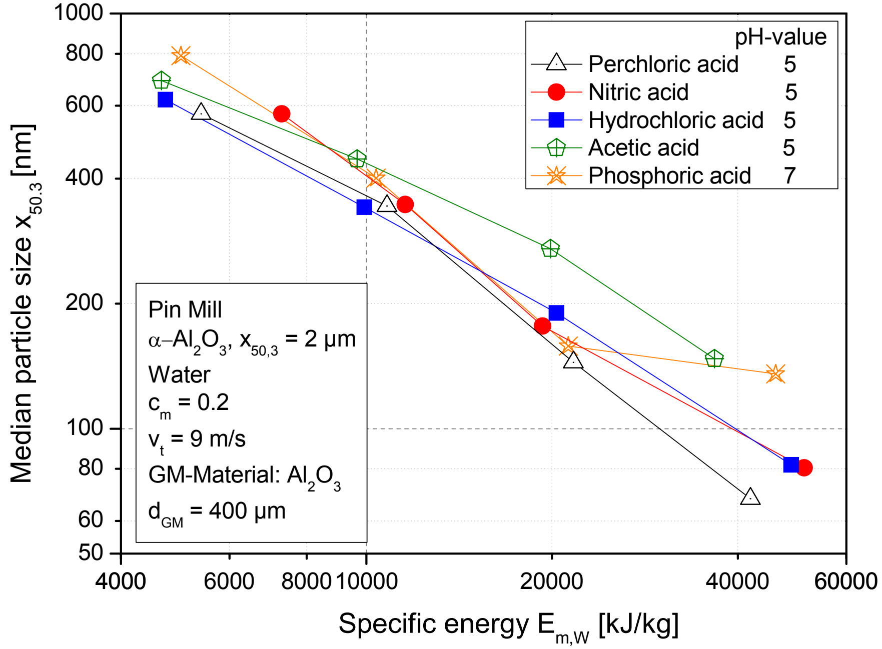


Interaction-potential





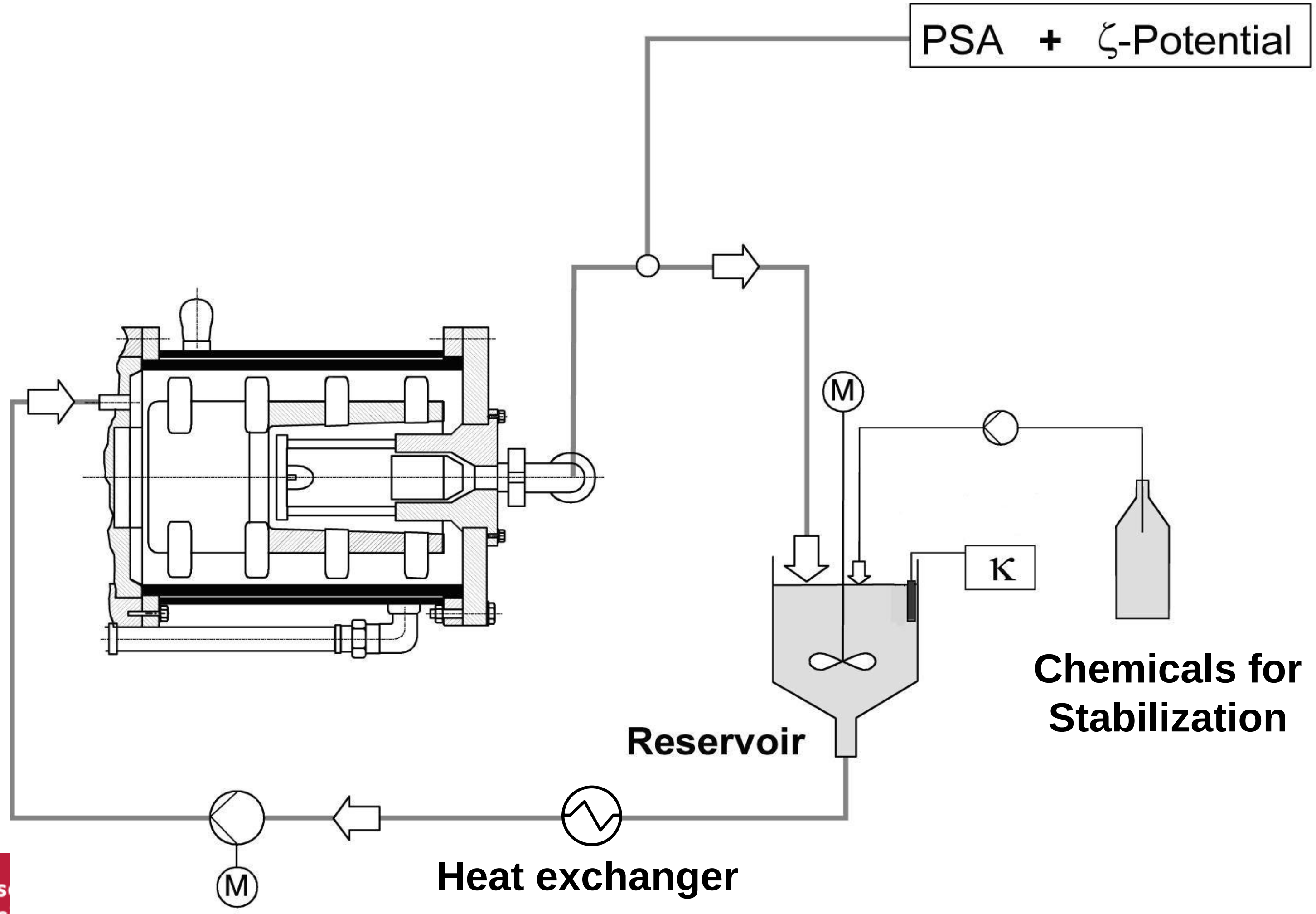
Summary

Dispersing of Nanoparticles

- Different machines lead to different stress mechanism and therewith to different dispersion results
- With a kinetic modell the reachable end particle size can be determined

Real Grinding

- Only stirred media mills can be used for reaching nanoparticles by grinding due to their high energy densities
- Stabilization of the suspension is necessary for controling the viscosity during the grinding process
- Kinetic modell for discribing dispersion processes is transferable on grinding processes


PSA + ζ-Potential
M
κ
Chemicals for Stabilization
Reservoir
M
Heat exchanger