Production of coated reference materials to modify the contact properties

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Introduction.

The **microscopic** interactions between particles in powders and granular media are of crucial importance and determine the physical **macroscopic** properties of solid products (fillers, pigments etc.) and natural materials (aerosols, soils, sludges or dusts).
The morphology and chemical composition of the particle surface strongly influences the interparticular interactions, therefore, it is advisable to formulate the solid surface properties by thin layers of functional materials. These layers may be adsorbed, bound to, or coated on the surface.

Problem Definition

Surface Modification and Characterization

- Thin smooth layers: Titanium dioxide; Hydrophobic silanes; Silicon dioxide
- Fine nano-particles: Titanium dioxide; Silicon dioxide

Objectives

- ✓ Micro-glass particle surface modification to alter the adhesion and **microscopic** contact properties
- ✓ **Macro mechanical** cohesive powder flow properties
- ✓ Adhesive contact properties with increasing normal load

Cooperation

K. Mader-Arndt – *University Magdeburg* : Single particle measurements

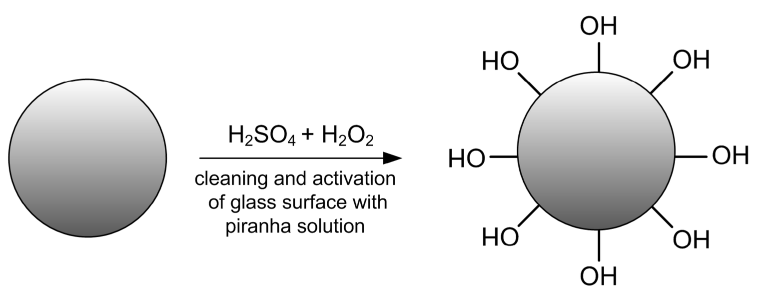
B. Torun – *University Paderborn:* Particle characterization

F. Schulz and Y. Wang – *University Magdeburg:* Contact angle measurements

R. Fuchs – *University Siegen:* Particle characterization

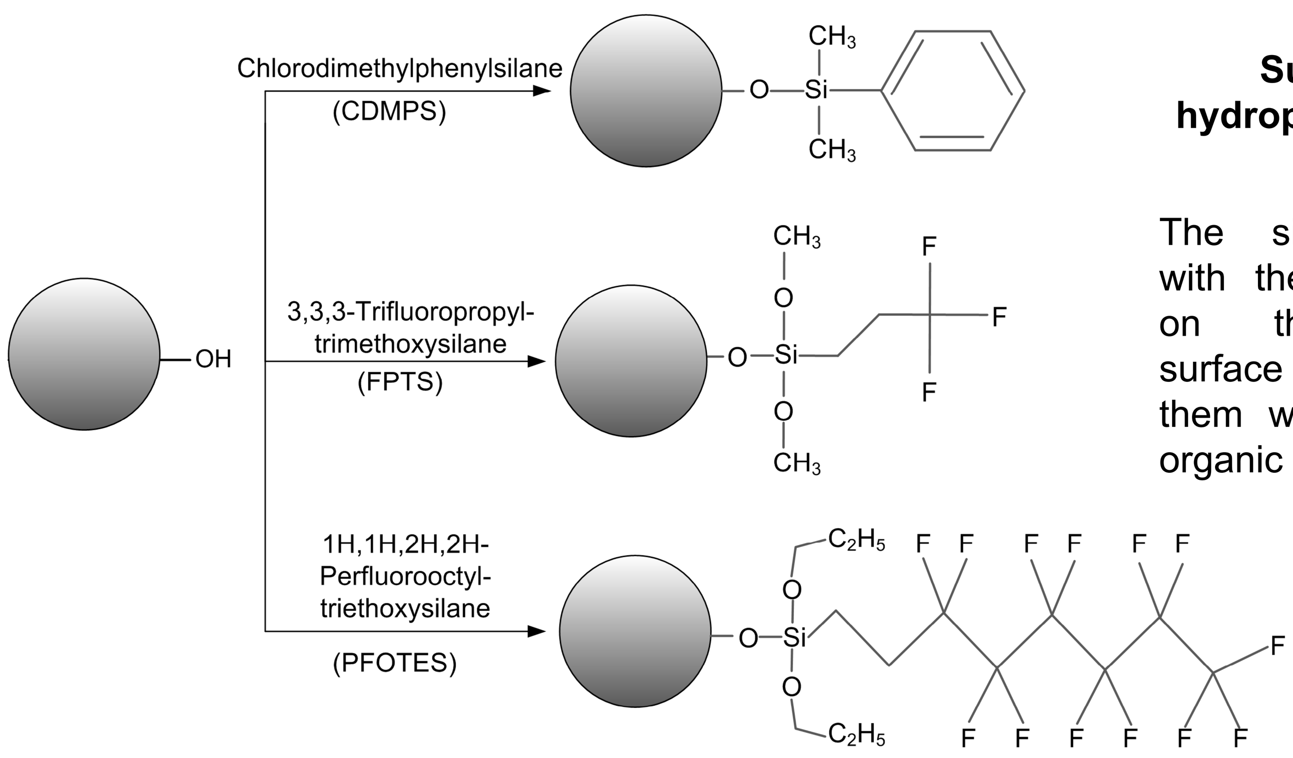
Experimental Setup

Preparation of the modification process



- ✓ The cleaning procedure is crucial for the quality of coatings
- ✓ Piranha acid ➡ thoroughly cleaned and activated surface

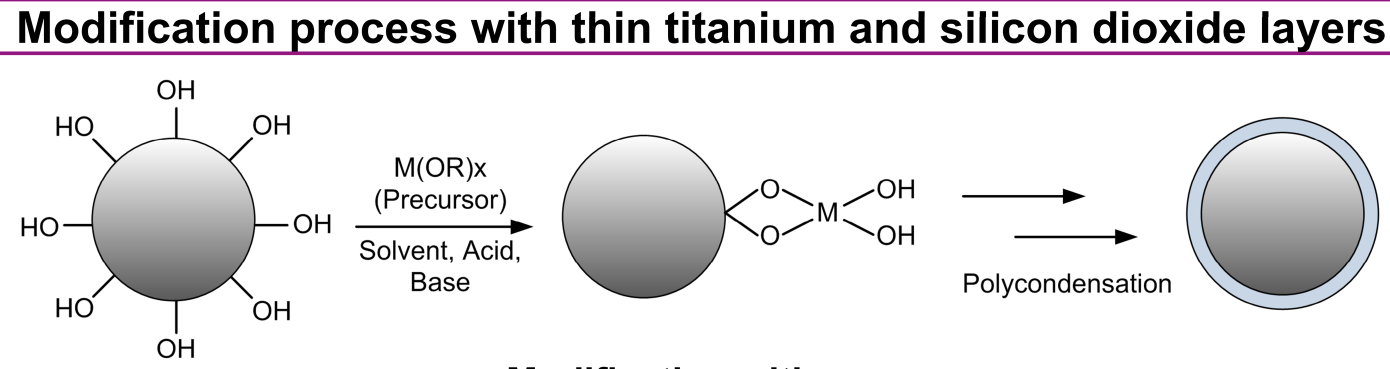
Modification process with hydrophobic silanes



Surface hydrophobization

The silanes react with the OH-groups on the particle surface and replace them with non-polar organic chains.

Modification process with thin titanium and silicon dioxide layers

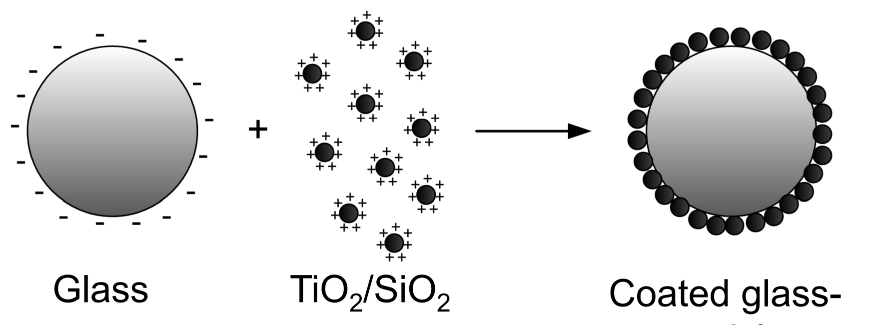


Modification with

Titanium Dioxide
- ✓ high Hamaker constant ($C_H = 12.6 \cdot 10^{-20}$ J)
- ✓ compliant contact behavior (high adhesion potential)

Silicon dioxide
- ✓ low Hamaker constant ($C_H = 3 \cdot 10^{-20}$ J)
- ✓ rigid contact behavior (similar to those of glass)

Modification process with titanium and silicon dioxide nano-particles



Hetero-coagulation coating process

Coating of micro-glass particles with nano-scaled (TiO_2 and SiO_2) on the basis of hetero-coagulation coating process to alter the surface roughness.

Analytical Description

Shear testing

Determination of:

Powder properties at macroscopic scale

- ✓ Yield loci $\tau = f(\sigma)$ (major principal stress σ_1, uniaxial compressive strength σ_c, flow function ff_c, angle of internal friction φ_i, effective angle of internal friction φ_e, bulk density ρ_b)
- ✓ Stationary yield locus $\sigma_{R,st} = f(\sigma_{M,st})$ (stationary angle of internal friction φ_{st}, isostatic tensile strength of the unconsolidated powder σ_0)
- ✓ Compression function $\rho_b = f(\sigma_1)$ (bulk density of the unconsolidated powder $\rho_{b,0}$, compressibility index n)

Model-based back-calculation

Reverse micro-macro transition

Particle properties at microscopic scale

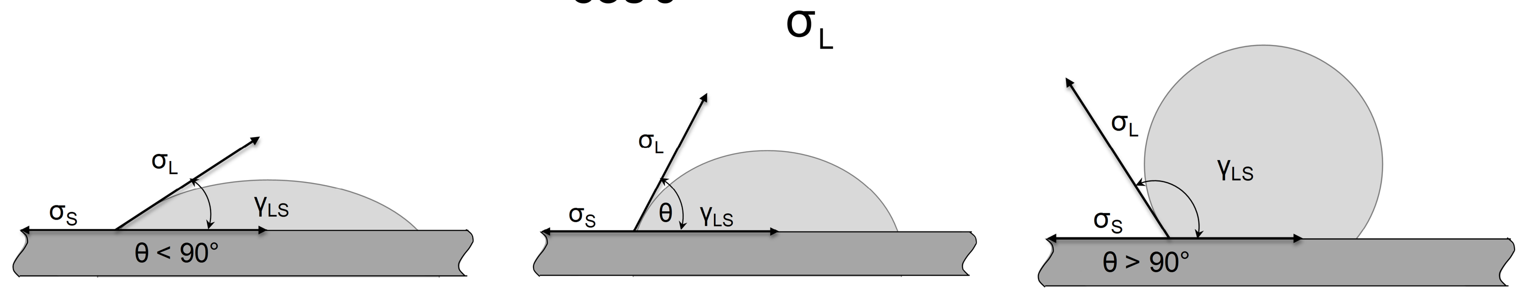
- ✓ Characteristic mean adhesion force between two particles of unconsolidated powder F_{H0}
- ✓ Elastic-plastic contact consolidation coefficient κ
- ✓ Plastic repulsion coefficient κ_p
- ✓ Total adhesion force between two particles in contact F_H

Measurement of surface wettability and energetics

YOUNG equation:

$$\cos\theta = \frac{\sigma_S - \gamma_{SL}}{\sigma_L}$$

Hydrophilic surface / **Hydrophobic surface**

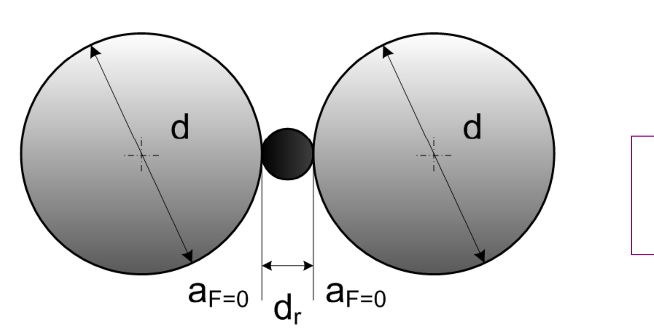


low	contact angle	high
good	adhesiveness	poor
good	wettability	poor
high	solid free surface energy	low

σ_L surface tension of the liquid; σ_S surface energy of the solid
γ_{LS} interfacial tension between the liquid and the solid phase; θ contact angle

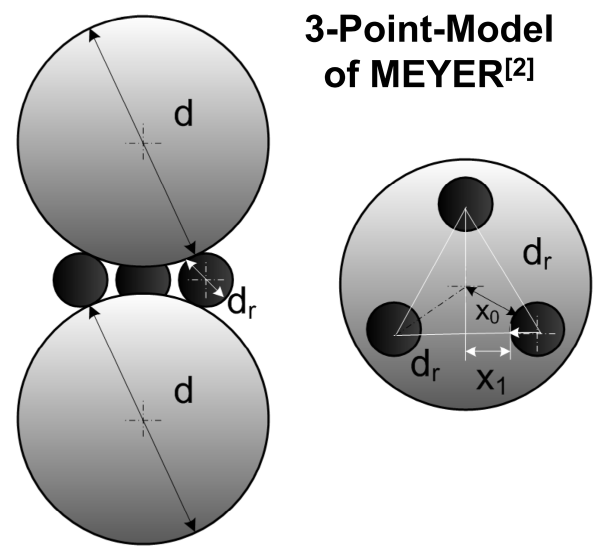
Effect of surface roughness to reduce particle adhesion

Sandwich-Model of Zimmermann[1]



Adhesion reduction

3-Point-Model of MEYER[2]

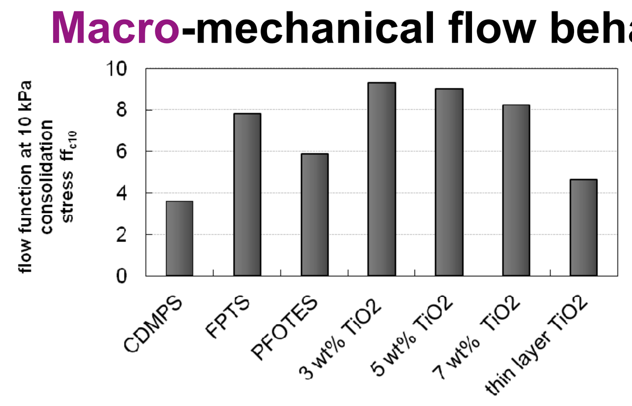


$$F_{H0} = \frac{C_{H,sls}}{24} \cdot \left[\frac{d}{(d_r + 2 \cdot a_{F=0})^2} + \frac{d_r}{a_{F=0}^2} \right]$$

[1] I. Zimmermann, M. Eber, K, Meyer , Z. Phys. Chem., **218**, 51-102,(2004).
[2] K. Meyer, Nanomterialien als Fließregulierungsmittel, Dissertation, University Würzburg, (2003)

Results and Discussion

Macro-mechanical flow behavior



Jenike Flow-function

$$ff_c = \frac{\sigma_1}{\sigma_c}$$

Micro-mechanical contact properties

Coating type	Adhesion force F_{H0} in nN	Coating type	Adhesion force F_{H0} in nN
1) uncoated glass	106.4	5) 3 wt% TiO_2	35.11
2) CDMPS	127.8	6) 5 wt% TiO_2	46.06
3) FPTS	258	7) 7 wt% TiO_2	62.93
4) PFOTES	72.71	8) thin layer TiO_2	32.23

Conclusions

Hydrophobic surface
- ⇒ decreased surface free energy
- ⇒ lower interparticle interaction
- ⇒ better flowability

Nano-scaled particles
- ⇒ increased surface roughness
- ⇒ decreased particles adhesion

Thin TiO_2 layers
- ⇒ surface coated with soft material
- ⇒ increased particle adhesion with increasing normal load

DFG-Graduate School 1554 “Micro-Macro-Interactions in Structured Media and Particle Systems”