Particle Adhesion Fundamentals and Bulk Powder Consolidation

Jürgen Tomas
The Otto-von-Guericke-University Magdeburg,
Mechanical Process Engineering

Abstract

The fundamentals of particulate solids consolidation and flow behaviour using a reasonable combination of particle and continuum mechanics are explained. By means of the model "stiff particles with soft contacts" the combined influence of elastic, plastic and viscoplastic repulsion in particle contacts is derived. Consequently, contact normal force displacement $F_N(h_K)$ and adhesion force models $F_H(F_N)$ are presented to describe the stationary, instantaneous and time consolidation behaviour in particle contacts as well as in the bulk. On this particle mechanical basis, the stationary, instantaneous and time yield loci as well as uniaxial compressive strength $c_0(\sigma_0)$, effective angle of internal friction $\phi_e(\sigma_0)$, bulk density $\rho_b(\sigma_0)$ are derived and shown for a very cohesive sub-micron titania powder. Finally, these models in combination with accurate shear cell test results are used as constitutive functions for computer aided silo design for reliable flow and pressure calculations.

1. Introduction

The well-known flow problems of cohesive particulate solids in storage and transportation containers, conveyors or process apparatuses leads to bridging, channelling, oscillating mass flow rates and particle characteristics with feeding and dosing problems as well as undesired effects like widely spread residence time distribution, fertiliser time consolidation process or deformation rate $v<1\,\text{m/s}$, therefore shear stress $\tau_v<\rho\cdot v^2/2\approx 1\,\text{kPa}$, pressure $\sigma=0.1-100\,\text{kPa}$) was explained by acting adhesion forces in particle contacts, Molerus (11).

On principle, there are four essential mechanical deformation effects in particle surface contacts and their physical behaviour – influenced namely by mechanical, optical and electromagnetic material characteristics of bulk phase – can be distinguished as follows:

1) reversible elastic (Hertz (15), Huber (16), Mindlin (17), Dahneke (19), DMT theory from Derjagin et al. (20) and JKR theory from Johnson et al. (21, 22), Thornton (27)) without deformation rate and consolidation time effects, valid for all particulate solids;

2) irreversible plastic (Krupp (18), Schubert (23), Molerus theory (11, 12), Maugis (26) and Thornton (28)), deformation rate and consolidation time invariant, e.g. mineral powders;

3) reversible viscoelastic (Hsuin (24) and Rumpf et al. (25)), deformation rate and consolidation time variable, e.g. cut post-consumer waste particles;

4) irreversible viscoplastic (Rumpf et al. (25)), deformation rate and consolidation time variable, e.g. fine particle fusion.

If an external compressive normal force $F_N$ is acting on a soft contact of two isotropic, stiff, linear elastic, mono-disperse spherical particles the previous contact point is deformed to a contact area and the adhesion force between these two partners is increasing, see Fig. 1.
First of all, Rumpf et al. (25) have developed a constitutive model approach for describing the linear increasing of adhesion force $F_H$, mainly for plastic contact deformation:

$$F_H = \left(1 + \frac{p_{VDW}}{P_t}\right) F_{H0} + \frac{p_{VDW}}{P_t} F_N$$

With another prerequisite and derivation Molerus (12) obtained an equivalent formula:

$$F_H = F_{H0} + \frac{p_{VDW}}{P_t} F_N = F_{H0} + \kappa_p F_N$$

The adhesion force $F_{H0}$ without additional consolidation ($F_N=0$) can be approached as a single sphere-sphere-contact, see Fig. 1a). This soft particle contact is flattening by the external normal force $F_N$ to a plate-plate-contact, Fig. 1c). The coefficient $\kappa_p$ describes a dimensionless ratio of attractive Van Der Waals pressure $p_{VDW}$ for a plate-plate model to repulsive particle micro-hardness $P_t$ (resistance against plastic deformation)

$$\kappa_p = \frac{p_{VDW}}{P_t} = \frac{C_{H,sls}}{6 \cdot \pi \cdot a^2_{F,F(F)}}$$

and is to be called here as a plastic repulsion coefficient. The characteristic adhesion separation distance lies in a molecular scale (atomic centre to centre distance) and amounts to about $a = a_{F,F} = 0.3-0.4$ nm. It depends mainly on the properties of liquid-equivalent packed adsorbed layers and can be estimated for a molecular force equilibrium or interaction potential minimum $-dU/da = F = 0 = F_{attraction} + F_{repulsion}$. The Hamaker constant solid-liquid-solid $C_{H,sls}$ acc. to Lifschitz theory is related to continuous media, dependent on their permittivities (dielectric constants) and refractive indices, see Israelachvili (29).

If the contact radius $r$ is small compared with the particle size $d$, an elliptic pressure distribution $p_{rel}(r)$ is found, see Hertz (15). Schubert (23) has combined the elastic and plastic contact strain expressed with the annular elastic $A_{el}$ and circular plastic $A_{pl}$ contact area:

$$F_H = F_{H0} + \frac{p_{VDW}}{P_t \cdot (1 + 2 \cdot A_{el}/(3 \cdot A_{pl})} \cdot F_N$$

Taking into account these Eqs. (1), (2), (4) as well as the model from Thornton (28) and superposition
vided, the particle contact force equilibrium between attraction (−) and elastic as well as plastic repulsion or force response (+)

\[ \sum F = 0 = -F_{\text{H0}} - \text{p}_{\text{v0w}} \cdot \pi \cdot r_c^2 - F_N + \text{p}_{\text{r}} \cdot \pi \cdot r_d^2 + 2 \cdot \pi \int_{r_d}^{r_c} \text{p}_{\text{a}}(r) \cdot r \, dr \]  

(5)

delivers a very useful linear force displacement model for \( k_N = \text{const.} \) with the particle centre approach of both partners \( h_K \) (32), Fig. 2:

\[ F_N + F_{\text{H0}} = \frac{\pi}{4} \cdot d \cdot \text{p}_{\text{r}} \cdot [\kappa_0 - \kappa_p] \cdot h_K \]  

(6)

Thus, the contact stiffness decreases with smaller size especially for cohesive fine powders and nanoparticles, predominant plastic yielding behaviour provided (32):

\[ k_{N,p} = \frac{\partial F_N}{\partial h_K} = \frac{\pi}{4} \cdot d \cdot \text{p}_{\text{r}} \cdot (\kappa_0 - \kappa_p) \]  

(7)

But if we consider Eq.(9) a slightly nonlinear (progressively increasing) curve is obtained.

The dominant linear elastic-plastic deformation range between really tested yield loci levels YL1 to YL 4 is to be demonstrated in Fig. 2. The origin of this diagram \( h_K = 0 \) is equivalent to the characteristic adhesion separation for a direct contact (atomic centre to centre distance) and can be estimated for a molecular force equilibrium \( \alpha = \alpha_0 = 2r - 0 \). Provided that these molecular contacts are stiff enough compared with the soft mesoscopic particle contact behaviour influenced by mobile adsorption layers due to molecular rearrangement, this separation \( \alpha_{p,0} \) is assumed to be constant during loading and unloading in the interesting macroscopic pressure range of \( \sigma = 0.1 - 100 \) kPa. After loading \( 0 - Y \) the contact is elastically compacted with an approximated circular contact area, see Fig. 1b) and starts at the yield point \( Y \) at \( p_{\text{max}} = p_t \).

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**Fig. 2** Recalculated characteristic contact deformation acc. to Fig. 1 of very cohesive titania particles, median particle size \( d_{50,1} = 610 \) nm, moisture \( X_w = 0.4 \).
with plastic yielding, see Fig. 1c). Now, the combined elastic-plastic yield boundary of the plate-plate contact is achieved, see Eq. (6). This displacement is to be expressed with the annular elastic $A_{el}$ (thickness $r_{K,el}$) and circular plastic $A_{pl}$ (radius $r_{K,pl}$) contact area, see Fig. 1c). For comparison, a dashed line for the flowability limit $h_{KL}=2$ (very cohesive acc. to Jenike (2)) is plotted additionally (32), see Fig. 2.

After unloading $U-E$ the contact recovers elastically in the compression mode and remains with a perfect plastic displacement $h_{K,el}$. Below point E left the tension mode begins. Between $U-E-A$ the contact recovers probably elastically along a supplemented Hertzian parabolic curvature up to a displacement $h_{K,A}$. Along $A-U$ the contact could be reloaded. If we apply a certain pull-off force $F_{Nh.z}=-F_{H,A}$ here negative, the adhesion (failure) boundary at point A is reached and the contact plates are failing and removing with the increasing distance $a=a_{F=0}+h_{K,A}-h_{K,el}$. This actual particle separation is to be considered for the calculation by means of a hyperbolic adhesion force curve $F_{Nh.z}=-F_{Ho}$ with the plate-plate model, see Eq. (3).

These new models in Fig. 2 follow Molerus’ (11), Schubert’s (23), Maugis’ (26) and especially Thornton’s (28) example.

The slopes of plastic curves are a measure of irreversible particle contact stiffness or softness, resp. Thus the dimensionless contact consolidation coefficient ($\kappa_{A}$) is read here as the slope of adhesion force, Fig. 3, influenced by predominant plastic contact failure.

$$\kappa = \frac{\kappa_{p}}{\kappa_{A} - \kappa_{p}}$$  \hspace{1cm} (8)

The elastic-plastic contact area coefficient $\kappa_{A}$ represents the ratio of plastic particle contact deformation area $A_{pl}$ to total contact deformation area $A_{K}=A_{pl}+A_{el}$ including a certain elastic displacement (32)

$$\kappa_{A} = \frac{2}{3} + \frac{1}{3} \frac{A_{pl}}{A_{K}} = 1 - \frac{1}{3} \frac{h_{K,el}}{h_{K}^{3}}$$  \hspace{1cm} (9)

with the centre approach $h_{K,el}$ for incipient yielding at $p_{el}(r=0)=p_{max}=p_{f}$:

$$h_{K,el} = d \left( \frac{\pi \cdot p_{f}}{2 \cdot E^{*}} \right)^{1/2}$$  \hspace{1cm} (10)

The pure elastic contact deformation $A_{el}=0$, $\kappa_{A}=2/3$, has no relevance for fine cohesive particles and should be excluded here. Commonly, for pure plastic contact deformation $A_{pl}=0$ or $A_{K}=A_{el}$, $\kappa_{A}=1$ is obtained. From Eq. (5), a linear model for the adhesion force $F_{H}$ as function of normal force $F_{N}$ is obtained again (32):

$$F_{H} = \frac{\kappa_{A}}{\kappa_{A} - \kappa_{p}} \cdot F_{Ho} + \frac{\kappa_{p}}{\kappa_{A} - \kappa_{p}} \cdot F_{N} = (1+\kappa) \cdot F_{Ho} + \kappa \cdot F_{N}$$  \hspace{1cm} (11)

The contact consolidation coefficient $\kappa$ is a measure of irreversible particle contact stiffness or softness as well, see Fig. 3. A small slope stands for low adhesion level $F_{H}=F_{Ho}$ because of stiff particle contacts, but a large inclination means soft contacts or consequently, a cohesive powder flow behaviour, see Fig. 6 as well.

This model considers additionally the flattening of soft particle contacts caused by acting an adhesion force $K \cdot F_{Ho}$. Herewith, the adhesion force $F_{Ho}$ for $F_{N}=0$ considers an essential characteristic micro roughness height $h_{r}<d$ particle size (Schubert (30)):

$$F_{Ho} = C_{H,sls} \cdot \left( \frac{2 \cdot h_{r}}{a_{F=0}^{2}} \right)^{1} \left[ 1 + \frac{d/h_{r}}{2 \cdot (1+h_{r}/a_{F=0})^{3/2}} \right] \approx C_{H,sls} \cdot h_{r} / 12 \cdot a_{F=0}^{2}$$  \hspace{1cm} (12)

The intersection of function (11) with abscissa ($F_{H}=0$) in the negative extension range of consolidation force $F_{N}$, Fig. 3, is surprisingly independent of the Hamaker constant $C_{H,sls}$:

$$F_{N,Z} = -\frac{\pi}{2} \cdot a_{F=0}^{2} \cdot h_{r} \cdot p_{f} \cdot \kappa_{A} \left[ 1 + \frac{d/h_{r}}{2 \cdot (1+h_{r}/a_{F=0})^{3/2}} \right]$$

$$= -\frac{\pi}{2} \cdot a_{F=0}^{2} \cdot h_{r} \cdot p_{f}$$  \hspace{1cm} (13)

Considering the model prerequisites for cohesive powders, this minimum normal (tensile) force limit $F_{N,Z}$ combines the influences of a particle contact hardness or micro-yield strength $p_{f}=3\cdot \sigma_{f}$ (yield strength in tension) and particle separation distance distribution characterised by a particle roughness height $h_{r}$ as well as molecular centre separation distance $a_{F=0}$. Obviously, this value characterises the contact softness with respect to a small asperity height $h_{r}$ as well, compare Eq.(7). It corresponds to an
A term for deformation rate influence on adhesion force in a particle contact is to be inserted

$$F_{Ht} = k_{t} \cdot F_{H0} + k_{t} \cdot F_{N}$$  \hspace{1cm} (14)

with a viscoplastic contact consolidation coefficient $k_{t} = \text{attraction/repulsion force ratio}$. A dimensionless combination of attractive contact strength $\sigma_{a} = \rho_{vdw}$ and repulsive particle contact viscosity $\eta_{v}/t = \rho_{1}$, i.e. viscoplastic stiffness, analogous to plastic deformation, Fig. 3.

$$k_{t} = \frac{\sigma_{a}}{\eta_{v} \cdot \dot{\varepsilon}_{v}} = \frac{\sigma_{a} \cdot d \cdot dt}{\eta_{v} \cdot d r_{st}}$$  \hspace{1cm} (15)

For example, the driving potential in the main case of viscoplastic contact deformation or particle fusion resp., is given by means of free surface energy $\sigma_{ss}$.

On the one hand, the influence of the pre-consolidation normal force $F_{N}$ on the contact circle radius ratio $r_{vis}/d$ was derived from Rumpf et al. (25):

$$\left( \frac{r_{vis}}{d} \right)^{2} = \frac{2 \cdot t}{5 \cdot \eta_{s}} \cdot \frac{2 \cdot \sigma_{ss} \cdot \eta_{s}}{d} \cdot \frac{1}{\pi} \cdot \frac{F_{x}}{d^{2}}$$  \hspace{1cm} (16)

The adhesion force $F_{Ht}$ is set proportional to the tensile strength of flowing material. This is created either by means of liquid-equivalent adsorption layer bridges with Van Der Waals and hydrogen bonds or by Van Der Waals solid bridges $\sigma_{ss} = \sigma_{a}$ and $\eta_{s} = \eta_{v}$, with small melting point $(0.75-0.9) \cdot T_{m}$ for particle fusion in the contact zone:

$$F_{Ht} = \pi \cdot r_{st}^{2} \cdot \sigma_{2s} = \frac{4 \cdot \pi \cdot \sigma_{2s} \cdot \sigma_{ss} \cdot d \cdot t}{5 \cdot \eta_{s}} \cdot \frac{2 \cdot \sigma_{2s} \cdot t}{5 \cdot \eta_{s}} \cdot F_{N}$$

$$= k_{t} \cdot F_{H0} + k_{t} \cdot F_{N}$$  \hspace{1cm} (17)

Generally, this corresponds to linear particle contact constitutive model, Eq.(14), with the viscoplastic repulsion or contact consolidation coefficient $k_{t}$ as a slope of adhesion force when a normal force $F_{N}$ acts in the deformed contact (33, 34, 35, 36):

$$k_{t} = \frac{2 \cdot \sigma_{2s} \cdot t}{5 \cdot \eta_{s}}$$  \hspace{1cm} (18)

The particle viscosity $\eta_{s}$ decreases with temperature increasing. This is to be described with a typical thermal kinetic expression for particle fusion with 3 parameters $(E_{f}, \text{molar fusion activation energy for shear rate gradient t}, T_{c} = (200..400) \text{K temperature parameter for polymers with } 0K<T_{c}<T_{G} \text{ glass transition point, R universal gas constant})$. The viscosity parameter $\eta_{s} = \eta_{s, \text{min}}$ for $T >> T_{m}$ depends here on molecular mass, chain length and number of cross-links for polymers (31).

$$\eta_{s} = \eta_{s, \text{min}} \cdot \exp \left[ \frac{E_{f}}{R \cdot (T-T_{c})} \right]$$  \hspace{1cm} (19)

The median adhesion forces $F_{H0}$ and $F_{H1}$, see Eq.(12), of a direct spherical contact

$$F_{H1,t} = 2 \cdot \pi \cdot \sigma_{ss} \cdot \eta_{v} \cdot t = \rho_{1} \cdot k_{t}(t) \hspace{1cm} \text{and}$$  \hspace{1cm} (20)

$$F_{H0} = 2 \cdot \pi \cdot \sigma_{ss} \cdot d = F_{H0}$$  \hspace{1cm} (21)

correspond each other if the so-called Derjaguin approximation (29) is valid $(aF_{o} << d)$

$$\sigma_{ss} = \frac{1}{2 \cdot A_{C}} \cdot \int F_{H0}(a) da = - \frac{1}{2} \cdot \int \rho_{vdw}(a) da$$

$$= \frac{C_{H,svs}}{24 \cdot \pi \cdot a_{0}^{2}}$$  \hspace{1cm} (22)

and the particle bondings are comparatively weak, e.g. Van Der Waals interaction $(C_{H,svs} \text{ Hamaker constant solid-vacuum-solid})$. But, these bonds are strong enough to disturb essentially reliable flow of particulate solids.

In opposition to time invariable plastic contact deformation, all parameters depend on time. Therefore superposition provided, the total adhesion force $F_{H tot}$ consists of a instantaneous $F_{H}$ and a time influenced component $F_{Ht}$, Fig. 3:

$$F_{H tot} = F_{H} + F_{Ht} = (1 + \kappa + \kappa_{t}) \cdot F_{H0} + (\kappa + \kappa_{t}) \cdot F_{N}$$  \hspace{1cm} (23)

In this context, the total adhesion force $F_{H tot}$ is a function of adhesion force $F_{H0}$ without any deformation or pre-consolidation in a very closed sphere-sphere contact plus a consolidation term $(\kappa + \kappa_{t}) \cdot F_{N}$ with a normal force in the flattened plate-plate particle contact. Finally, this Eq.(23) can be interpreted as a general linear particle contact constitutive model, i.e. linear in forces and stresses, but non-linear concerning material characteristics, Table 1. Generally, this adhesion force level, see Fig. 3, amounts up to $10^{5} - 10^{6}$ fold of particle weight for very cohesive fine particles. Additionally, for moist powders, the liquid bridge bonding forces are strength determined (33, 38, 39).

### 3. Particle contact Failure and cohesive Powder Flow criteria

Regarding the formulation of failure conditions at the particle contacts, we can obviously follow the Molerus theory (14), but here with a general supplement for the particle contact constitutive models Eqs.(11), (14) and (23), see in detail (32). The inclina-
tion of radius and centre contact force components \(F_R, F_M\) and normal vectors are described with an angle \(\alpha\), see contact failure conditions in Table 1. It should be noted that the stressing pre-history of a cohesive powder flow is stationary (steady-state) and delivers significantly a cohesive stationary yield locus in radius – centre-stress coordinates,

\[
\sigma_{R,M} = \sin \varphi_{it} \left( \sigma_{M,it} + \sigma_o \right)
\]

(24)

with the isostatic tensile strength \(\sigma_0\) obtained from the adhesion force \(F_{H_0}\), see Eq. (12).

\[
\sigma_0 = \frac{1 - \varepsilon_0}{\varepsilon_0} \cdot \frac{F_{H_0}}{d^2}
\]

(25)

From it, the stress dependent effective angle of internal friction \(\varphi_e\) acc. to Jenike as a slope of cohesionless effective yield locus follows obviously, see Fig. 4:

\[
\sin \varphi_e = \sin \varphi_{it} \left( \frac{\sigma_1 + \sigma_0}{\sigma_1 - \sin \varphi_{it} - \sigma_0} \right)
\]

(26)

If the major principal stress \(\sigma_1\) reaches the stationary uniaxial compressive strength \(\sigma_{c, it}\),

\[
\sigma_1 = \sigma_{c, it} = 2 \cdot \sin \varphi_{it} \cdot \sigma_0
\]

(27)

the effective angle of internal friction amounts to \(\varphi_e = 90^\circ\) and for \(\sigma_1 \rightarrow \infty\) follows \(\varphi_e \rightarrow \varphi_{it}\), see Fig. 4.

For the relation between the angle of internal friction \(\varphi_i\) (slope of instantaneous yield locus) and the stationary angle of internal friction \(\varphi_{it}\) following relation is used (12):

\[
\tan \varphi_{it} = (1 + \kappa) \cdot \tan \varphi_i
\]

(28)

The softer the particle contacts, the larger the difference between these friction angles are and consequently the more cohesive the powder behave. Therefore considering Eq. (23), the new relation between the time dependent angle of internal friction \(\varphi_{it}\) (slope of time yield locus) and the time invariable stationary angle of internal friction \(\varphi_{it}\) (slope of stationary yield locus) is defined as Fig. 5:

\[
\tan \varphi_{it} = (1 + \kappa + \kappa_i) \cdot \tan \varphi_i = \text{const.} \neq f(t)
\]

(29)

Now, the angle of internal friction of a time consolidation \(\varphi_{it}\) is to be expressed (33):

\[
\tan \varphi_{it} = \frac{\tan \varphi_i}{1 + \kappa_i \cdot \tan \varphi_i} = \frac{\tan \varphi_i}{2 \cdot \sigma_{st} \cdot \tan \varphi_i \cdot t}
\]

(30)

First of it, with this Eq. (30) following predictions are possible (33, 34, 35, 36, 37), Fig. 5:

(1) If no time consolidation occurs \(t=0\), both friction angles are equivalent \(\varphi_{it} = \varphi_i\). The non-linear and linear instantaneous yield loci in radius-centre-coordinates are obtained:

\[
\sigma_R = \sin \varphi_i \left( \sigma_{M,it} + \sigma_0 \right) \left( \frac{\tan \varphi_{it} - \sigma_{M,it} - \sigma_0}{\tan \varphi_i} \right)^2
\]

\[
- \frac{\tan^2 \varphi_{it} \cdot \sin^2 (\varphi_{it} - \varphi_i)}{\tan^2 \varphi_i} \cdot \tan \varphi_{it} \cdot \sin (\varphi_{it} - \varphi_i)
\]

(31)

\[
\sigma_2 = \sin \varphi_i \left( \sigma_M + \frac{\sin \varphi_{it}}{\sin \varphi_i} \cdot \frac{\sigma_{M,it} + \sigma_{M, it}}{\sigma_{M, it} + \sigma_0} \cdot \frac{\sin \varphi_{it}}{\sin \varphi_i} \right)
\]

(32)

(2) But if \(t>0\) the angle of internal friction during time consolidation decreases \(\varphi_{it} < \varphi_i\) and the non-linear and linear time yield loci in radius-centre-coordinates are:

\[
\sigma_{R, it} = \sin \varphi_{it} \left( \sigma_{M,it} + \sigma_0 \right) \left( \frac{\tan \varphi_{it} - \sigma_{M, it} - \sigma_0}{\tan \varphi_{it} - \sigma_{M, it} + \sigma_0} \right)^2
\]

\[
- \frac{\tan^2 \varphi_{it} \cdot \sin^2 (\varphi_{it} - \varphi_i)}{\tan^2 \varphi_{it}} \cdot \tan \varphi_{it} \cdot \sin (\varphi_{it} - \varphi_i)
\]

(33)

\[
\sigma_{R, it} = \sin \varphi_{it} \left( \sigma_{M, it} + \frac{\sin \varphi_{it}}{\sin \varphi_{it}} \cdot \frac{\sigma_{M, it} + \sigma_{M, it}}{\sigma_{M, it} + \sigma_0} \cdot \frac{\sin \varphi_{it}}{\sin \varphi_{it}} \right)
\]

(34)
(3) For $t \to \infty$ follows $\phi_a \to 0$, that means, the time yield locus is a parallel line to the $\sigma-$axis, i.e. failure criterion of ideal plasticity by Tresca. The bulk material is hardening to a complete solid state with plastic failure conditions as a limitation.

The non-linear and linear yield loci in $\tau$-$\sigma$-coordinates are shown in Table 1 and Fig. 5. These instantaneous yield loci, complementary consolidation loci and the stationary yield locus are completely described only with three material parameters plus the characteristic pre-consolidation (average pressure) influence (32):

1. $\phi_i$ - incipient particle friction of failing contacts, i.e. Coulomb friction;
2. $\phi_a$ - steady-state particle friction of failing contacts, increasing adhesion by means of contact flattening expressed with the contact consolidation coefficient $\kappa$, or by friction angles $\sin(\phi_a - \phi_i)$ and $(\sin \phi_a - \sin \phi_i)$ see Eqs.(31), (32) and (36). The softer the particle contacts, the larger the difference between these friction angles are and consequently the more cohesive the powder behave;
3. $\sigma_0$ - isostatic tensile strength of unconsolidated particle contacts, characteristic cohesion force in an unconsolidated powder, see Eq.(37);
4. $\sigma_{M,tt}$ - previous consolidation influence of an additional normal force in a particle contact, characteristic centre stress of Mohr circle of pre-consolidation state directly related to powder bulk density. This average pressure influences the increasing isostatic tensile strength of yield loci by means of cohesive steady-state flow as the stressing pre-history of a powder.

With the derivation of time yield locus the uniaxial compressive strength (unconfined yield strength) $\sigma_{ct}$ is found as function of the major principal stress $\sigma_1$, comparable with a linear bulk material constitutive model, Table 1:

$$
\sigma_{ct} = \frac{2 \cdot (\sin \phi_a - \sin \phi_i)}{(1 + \sin \phi_a) \cdot (1 - \sin \phi_i)} \cdot \sigma_1 \\
+ \frac{2 \cdot \sin \phi_a \cdot (1 + \sin \phi_a)}{(1 + \sin \phi_a) \cdot (1 - \sin \phi_i)} \cdot \sigma_0 = a_{1,t} \cdot \sigma_1 + \sigma_{ct,0}
$$

The slope $a_{1,t}$ and the intersection of $\sigma_1-$axis $\sigma_{ct,0}$ are time dependent, Eq.(30). The abscissa intersection $\sigma_{1,t}$ of linear consolidation constitutive function $\sigma_i(\sigma_1)$, corresponds to the $F_{s,z}$ value of contact consolidation function acc. to Eq.(13) and Fig. 3.

Again, the following predictions are to be advanced:

1. If no time consolidation occurs, both angles are equivalent $\phi_a = \phi_i$ and the linear constitutive model for plastic contact deformation is obtained $\sigma_i = a_{1,t} \cdot \sigma_1 + \sigma_{i,0}$

$$
\sigma_i = \frac{2 \cdot (\sin \phi_a - \sin \phi_i)}{(1 + \sin \phi_a) \cdot (1 - \sin \phi_i)} \cdot \sigma_1 \\
+ \frac{2 \cdot \sin \phi_a \cdot (1 + \sin \phi_a)}{(1 + \sin \phi_a) \cdot (1 - \sin \phi_i)} \cdot \sigma_0
$$
Table 1 Yield characteristics from Particle Mechanics point of view (37)

<table>
<thead>
<tr>
<th></th>
<th>instantaneous consolidation</th>
<th>time consolidation</th>
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</thead>
<tbody>
<tr>
<td><strong>adhesion force</strong></td>
<td>( F_h = (1 + \kappa) F_{H0} + \kappa F_n )</td>
<td>( F_h = (1 + \kappa + \kappa_o) F_{H0} + (\kappa + \kappa_o) F_n )</td>
</tr>
<tr>
<td><strong>contact consolidation coefficient</strong></td>
<td>( \kappa = \frac{\kappa_0}{\kappa_0 - \kappa_\gamma} = \frac{p_{\gamma\gamma}/p_\gamma}{2 + 2 \phi \gamma / (\phi \gamma - 1)} )</td>
<td>( \kappa = \frac{\phi \gamma / \eta \gamma}{\phi \gamma / \eta \gamma + \phi \gamma - 1} = \frac{2 \phi \gamma}{\eta \gamma + \phi \gamma} )</td>
</tr>
<tr>
<td><strong>contact failure condition for stationary flow</strong></td>
<td>( F_h \sin 2 \alpha \leq \tan \phi_i )</td>
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<tr>
<td><strong>stationary angle of internal friction</strong></td>
<td>( \tan \phi_i = (1 + \kappa) \cdot \tan \phi )</td>
<td>( \tan \phi_i = (1 + \kappa + \kappa_o) \cdot \tan \phi )</td>
</tr>
<tr>
<td><strong>effective angle of internal friction</strong></td>
<td>( \sin \phi_i = \sin \phi_o \cdot (\phi_i + \phi_o) / (\phi_i - \sin \phi_o \cdot \phi_o) )</td>
<td>( \sin \phi_i = \sin \phi_o \cdot (\phi_i + \phi_o) / (\phi_i - \sin \phi_o \cdot \phi_o) )</td>
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<tr>
<td><strong>stationary yield locus</strong></td>
<td>( \tau_s = \tan \phi_i \cdot (\phi_i + \phi_o) )</td>
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</tr>
<tr>
<td><strong>particle contact failure condition</strong></td>
<td>( F_h \cdot [\sin \phi_i - (1 + \kappa) \cdot F_{H0} / F_h] \cdot \tan \phi_i \cdot \cos 2 \alpha \leq \tan \phi_i )</td>
<td>( F_h \cdot [\sin \phi_i - (1 + \kappa + \kappa_o) \cdot F_{H0} / F_h] \cdot \tan \phi_i \cdot \cos 2 \alpha \leq \tan \phi_i )</td>
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<tr>
<td><strong>failure angle</strong></td>
<td>( \alpha = \kappa / (1 + \kappa) \cdot \tan \phi_i )</td>
<td>( \alpha = \kappa / (1 + \kappa + \kappa_o) \cdot \tan \phi_i )</td>
</tr>
<tr>
<td><strong>angle of internal friction</strong></td>
<td>( \tan \phi_i = \text{const.} )</td>
<td>( \tan \phi_i = \text{const} )</td>
</tr>
<tr>
<td><strong>non-linear yield locus</strong></td>
<td>( \tau_s = \tan \phi_i \cdot (\phi_i + \phi_o) )</td>
<td>( \tau_s = \tan \phi_i \cdot (\phi_i + \phi_o) )</td>
</tr>
<tr>
<td><strong>linear yield locus</strong></td>
<td>( \tau_s = \tan \phi_i \cdot \sin \phi_o \cdot \sin \phi_i )</td>
<td>( \tau_s = \tan \phi_i \cdot \sin \phi_o \cdot \sin \phi_i )</td>
</tr>
<tr>
<td><strong>uniaxial compressive strength</strong></td>
<td>( \sigma_o = \frac{2 \cdot \sin \phi_o \cdot \sin \phi_i}{(1 + \sin \phi_o) \cdot (1 - \sin \phi_i)} \cdot \sigma_i + \frac{2 \cdot \sin \phi_o \cdot (1 - \sin \phi_i)}{(1 + \sin \phi_o) \cdot (1 - \sin \phi_i)} \cdot \sigma_o )</td>
<td>( \sigma_o = \frac{2 \cdot \sin \phi_o \cdot (1 + \sin \phi_o)}{(1 + \sin \phi_o) \cdot (1 - \sin \phi_i)} \cdot \sigma_i + \frac{2 \cdot \sin \phi_o \cdot (1 + \sin \phi_o)}{(1 + \sin \phi_o) \cdot (1 - \sin \phi_i)} \cdot \sigma_o )</td>
</tr>
</tbody>
</table>

![Fig. 6 Consolidation function of titania.](image)

and with Eqs. (12) and (21) the isostatic tensile strength is expressed \( \sigma_o = 1 - \rho_{0,0} / \rho_0 \):

\[
\sigma_o = \frac{1 - \varepsilon_0}{\varepsilon_0} \cdot \frac{F_{H0}}{d^2} = 2 \cdot \pi \cdot \frac{1 - \varepsilon_0}{\varepsilon_0} \cdot \frac{C_{H,H}}{E_0} \cdot \frac{h_r}{d} \cdot \left(1 + \frac{h_r}{2 \cdot a_{F=0}}\right)^2
\]

(37)

(2) But if \( t > 0 \) the angle of internal friction during time consolidation decreases \( \phi_i < \phi \), and the slope \( \alpha_{ia} \) increases.

(3) For \( t \to \infty \), \( \phi_i \to \phi \), that means, the slope follows \( \alpha_{ia} \to 1 \). This is the largest slope considering the model prerequisites of an only viscoplastic flow. If the first derivative is greater than one \( \alpha_{ia} = \sigma_{at}/\sigma_o > 0 \), a non-linear relation of particle contact deformation and consequently other physicochemical effects of irreversible bulk material consolidation should be considered.

(4) Notice that for \( t \to \infty \) the intersection of \( \sigma_{at} \)-axis \( \sigma_{at,0} \) achieves a upper limit, which is only dependent on surface energy \( \sigma_{at} \) and particle size and not from time and viscosity:

\[
\sigma_{at,0} = \frac{2 \cdot \sin \phi_i \cdot (1 + \sin \phi_o)}{(1 + \sin \phi_o) \cdot (1 - \sin \phi_i)} \cdot \sigma_0
\]

\[
= \left( \frac{4 \cdot \pi \cdot \varepsilon_o}{\varepsilon_0} \cdot \sin \phi_i \cdot \frac{\sigma_{at}}{d} \right)^{\frac{3}{2}}
\]

(38)
4. Powder Flowability and Compressibility

Assessing the flow behaviour of a powder, Eq. (35) shows that the flow function according to Jenike (2) is not constant and depends on the consolidation stress level \( \sigma_1 \):

\[
\frac{f_{c}}{f_{c1}} = \left( 1 + \sin \varphi_0 \right) \left( 1 - \sin \varphi_0 \right) \left( \sin \varphi_a + \sin \varphi_a \sin \varphi_\varepsilon - \sin \varphi_0 \right) \sigma_0 / \sigma_1
\]  

(39)

But roughly we can write for a small intersection with the ordinate \( \sigma_0 \), i.e. isostatic tensile strength near zero, the stationary angle of internal friction is equivalent to the effective angle \( \varphi_\varepsilon \) and Jenike’s (2) formula is obtained in order to demonstrate the general model validity:

\[
f_{c1} \approx \frac{1 + \sin \varphi_0}{2 \left( \sin \varphi_a - \sin \varphi_0 \right)}
\]  

(40)

Thus, the semi-empirical classification by means of the flow function introduced by Jenike (2) is adopted here with a certain particle mechanical sense completion, Table 2:

Table 2: Flowability assessment and elastic-plastic contact consolidation coefficient \( \kappa(\varphi_0=30^\circ) \)

<table>
<thead>
<tr>
<th>Flow function ( f_{c1} )</th>
<th>( \kappa ) values</th>
<th>( \varphi_0 ) in deg</th>
<th>Evaluation</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>100–100</td>
<td>0.01006–0.1068</td>
<td>30.3–33</td>
<td>free flowing</td>
<td>dry fine sand</td>
</tr>
<tr>
<td>4–10</td>
<td>0.1068–0.3</td>
<td>33–37</td>
<td>easy flowing</td>
<td>moist fine sand</td>
</tr>
<tr>
<td>2–4</td>
<td>0.3–0.77</td>
<td>37–46</td>
<td>cohesive</td>
<td>dry powder</td>
</tr>
<tr>
<td>1–2</td>
<td>0.77–∞</td>
<td>46–90</td>
<td>very cohesive</td>
<td>moist powder</td>
</tr>
<tr>
<td>&lt;1</td>
<td>∞</td>
<td>–</td>
<td>non-flowing hardened (( f_{c1} ))</td>
<td>moist powder, hydrated cement</td>
</tr>
</tbody>
</table>

A characteristic value \( \kappa = 0.77 \) for \( \varphi_0 = 30^\circ \) of a very cohesive powder is included in the force displacement, Fig. 2, as well as adhesion force diagram, Fig. 3, and shows directly the correlation between strength and force enhancement with pre-consolidation, Table 2. Due to the consolidation function in Fig. 6, a small slope stands for a free flowing particulate solid with very low adhesion level because of stiff particle contacts, but a large inclination means a very cohesive powder flow behaviour because of soft particle contacts, Fig. 3.

Obviously, the finer the particles the “softer” the contacts and the more cohesive the powder (33, 42). Köhler (43) has experimentally confirmed this thesis for alumina powders (\( \alpha\text{-Al}_2\text{O}_3 \)) down to the submicron range (\( \sigma_{c,0} = \text{const.} = 2 \text{ kPa} \), \( d_\text{50} \) median particle size in \( \mu\text{m} \)):

\[
f_{c1} \approx 2.2 \cdot d_{\text{50}}^{0.62}
\]  

(42)

Analogously to adiabatic gas law \( p \cdot V^{\kappa} = \text{const.} \), a differential equation for isentropic compressibility of a powder \( dS = 0 \), i.e. remaining stochastic homogeneous packing without a regular order in the continuum, is to be derived:

\[
\frac{dp_b}{\rho_b} = \frac{dp}{\rho} = \frac{n \cdot \sigma_{M,\text{st}}}{\sigma_{M,\text{st}} + \sigma_0}
\]  

(43)

The total pressure including particle interaction \( p = \sigma_{M,\text{st}} + \sigma_0 \) should be equivalent to a pressure term with molecular interaction \( (V_m \text{ molar volume}) \)

\[
(p + a_{\text{PV}}/V_m^2) \cdot (V_m - b) = R \cdot T
\]  

(44)

in Van Der Waals equation of state to be valid near gas condensation point. A loose powder packing is obtained \( \rho_b = \rho_{b,0} \) if only particles are interacting without an external consolidation stress \( \sigma_{M,\text{st}} = 0 \), e.g. particle weight compensation by a fluid drag, and Eq.(43) is solved:

\[
\frac{\rho_b}{\rho_{b,0}} = \left( \frac{\sigma_0 + \sigma_{M,\text{st}}}{\sigma_0} \right)^n
\]  

(45)

Therefore, this physically based compressibility index \( n = 1/\kappa_{\text{ad}} \) lies between \( n = 0 \), i.e. incompressible stiff bulk material and \( n = 1 \), i.e. ideal (gas) compressibility index, see Fig. 7 above.

For hopper design purposes in powder mechanics the major principle stress \( \sigma_1 \) during preconsolidation is used instead of the centre stress \( \sigma_{M,\text{st}} \). Hence we replace the total pressure \( p = \sigma_1 + \sigma_{M,\text{st}} \) with the new abscissa intersection \( \sigma_{1,2} \) in the negative tensile or pull-off range of the consolidation function \( \sigma_1 = f(\sigma_1) \), Eq.(36) and Fig. 7, and obtain this function:
5. Design Consequences for Reliable Flow

Mainly for functional silo design purposes in Mechanical Process Engineering, these fundamental models can be applied by means of a characteristic apparatus dimension function, here a minimum hopper opening width b_{min,t} avoiding cohesive bulk material arches (g gravitational acceleration, ff flow factor acc. to Jenike, m=1 conical hopper, m=0 wedge hopper, \( \varphi_w \) angle of wall friction, \( \theta \) hopper angle versus vertical, \( \rho_b \) bulk density):

1. If no time consolidation occurs \( t=0 \), both friction angles are equivalent \( \varphi_w=\varphi_t \), and the linear bulk material constitutive model for plastic contact deformation is obtained. Consequently it is:

\[
b_{min} = 2 \cdot (m+1) \cdot \sin^2 (\varphi_w + \theta) \cdot \sin \varphi_{st} \cdot \sigma_0 \rho_b \cdot g \cdot [1 - \sin \varphi_{st} \cdot \sin \varphi_{st}] \cdot (2 \cdot ff - 1) \]

2. For the general case \( t>0 \) the angle of internal friction during time consolidation decreases \( \varphi_{st}<\varphi_t \), the slope\( \alpha_{st} \), and \( b_{min,t} \) increase:

\[
b_{min,t} = 2 \cdot (m+1) \cdot \sin^2 (\varphi_w + \theta) \cdot (1 + \sin \varphi_{st}) \cdot \sin \varphi_{st} \cdot \sigma_0 \rho_b \cdot g \cdot [1 - \sin \varphi_{st} \cdot \sin \varphi_{st} - (\sin \varphi_{st} - \sin \varphi_{st}) \cdot (2 \cdot ff - 1)]
\]

This approach should express the enormous problems concerning reliable flow of powders which are tending to time consolidation, hardening and caking. Consequently, discharging aids should be applied in handling practice (33).

The essential consolidation functions necessary for reliable design are collected in Fig. 8. For the cohesive steady-state flow \( \sigma_1=\sigma_{c,st} \), Fig. 4 and Eq. (27), the flow factor is \( ff=1 \) and a minimum outlet width \( b_{min,at}<b_{min} \) (instantaneous flow) is obtained which prevents bridging during the stationary hopper operation.

\[
b_{min,at} = 2 \cdot (m+1) \cdot \sin^2 (\varphi_w + \theta) \cdot \sin \varphi_{at} \cdot \sigma_0 \rho_b \cdot g \cdot [1 - \sin \varphi_{at} \cdot (2 \cdot ff - 1)]
\]

As starting value, \( b_{min,at} \) should be used for numerical calculation (33) of both functions \( \varphi_e(\sigma_1) \) and \( \rho_b(\sigma_1) \) as
well as the flow factor \((1, 2)\) \(ff\). Before that the hopper angle \(\theta(\phi, \sigma_1, \phi_w)\) has properly to be selected \((1, 2, 4, 5, 13)\).

For the sub-micron titania powder \((d_{50}, 3 = 610\ \text{nm}, \sigma_0 = 0.327\ \text{kPa}, p_s = 800\ \text{kg/m}^3, \) friction angles \(\phi_1 = 33^\circ, \phi_{\min} = 54^\circ, \phi_w = 31^\circ, \theta = 11^\circ, \) \(ff = 1.3\) practically reasonable \(b_{\min, st} = 0.7\ \text{m}\) for steady-state flow, but \(b_{\min, t} = 1.52\ \text{m}\) for incipient yield and incredible \(b_{\min, t} = 8.4\ \text{m}\) for gravitational flow (without flow promotion) after time consolidation at rest \((t = 24\ \text{h}, \phi_w = 31^\circ)\) are determined for a conical hopper.

Both, shear test results – accurate measurements provided – evaluated with these combined particle and continuum mechanical approach, are used as constitutive functions for computer aided silo design for reliable flow \((37)\) on the one hand. On the other hand a supplemented slice-element standard method \((41, 42)\) is used for pressure calculations. Considering the reliable physical basis of the \(p_h(\sigma_1)\) and \(\phi_0(\sigma_1)\) functions for example, these can be suitably extrapolated using pressure calculations for large silos with more than \(1000\ \text{m}^3\) storage capacity \((44)\).

6. Conclusions

Taking into consideration all the different properties of cohesive to very cohesive powders tested (particle size distribution, moisture content, material properties etc.), the model fit can be characterised as satisfactory to good. Thus, the model has proved its effectiveness and can be accordingly applied in reliable silo design for flow and pressure calculation.

Obviously, recommendations are to be elaborated with respect to the powder product design for processing, logistics, transportation, distribution and consumption, see Borho et al. \((45)\) as well.

7. Acknowledgements

The author would like to acknowledge his co-workers Dr. S. Aman, Dr. T. Gröger, S. Schubert, B. Reichmann and Th. Kollmann for their experimental contributions, relevant information and theoretical tips.

Indices

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td>separation</td>
</tr>
<tr>
<td>(a_1)</td>
<td>slope of (\sigma_1(\sigma_1)) consolidation function</td>
</tr>
<tr>
<td>(A)</td>
<td>area, particle contact area</td>
</tr>
<tr>
<td>(b)</td>
<td>outlet width</td>
</tr>
<tr>
<td>(C_H)</td>
<td>Hamaker constant</td>
</tr>
<tr>
<td>(d)</td>
<td>particle size</td>
</tr>
<tr>
<td>(E)</td>
<td>modulus of elasticity ([\text{kN/mm}^2])</td>
</tr>
<tr>
<td>(F)</td>
<td>force ([\text{N}])</td>
</tr>
<tr>
<td>(ff)</td>
<td>flow factor acc. to Jenike ([-] )</td>
</tr>
<tr>
<td>(f_f)</td>
<td>flow function acc. to Jenike ([-] )</td>
</tr>
<tr>
<td>(g)</td>
<td>gravity acceleration ([\text{m/s}^2])</td>
</tr>
<tr>
<td>(m)</td>
<td>mass, hopper shape factor ([\text{kg, } -])</td>
</tr>
<tr>
<td>(p)</td>
<td>pressure ([\text{kPa}])</td>
</tr>
<tr>
<td>(p_t)</td>
<td>plastic yield strength of particle contact ([\text{MPa}])</td>
</tr>
<tr>
<td>(r)</td>
<td>contact radius ([\mu\text{m}])</td>
</tr>
<tr>
<td>(\alpha)</td>
<td>failure angle ([\text{deg}])</td>
</tr>
<tr>
<td>(\beta)</td>
<td>auxiliary failure angle function ([\text{deg}])</td>
</tr>
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<td>(\dot{\gamma})</td>
<td>shear deformation rate gradient ([\text{s}^{-1}])</td>
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<tr>
<td>(\varepsilon)</td>
<td>porosity ([-] )</td>
</tr>
<tr>
<td>(\dot{\varepsilon})</td>
<td>deformation rate gradient ([\text{s}^{-1}])</td>
</tr>
<tr>
<td>(\eta)</td>
<td>viscosity ([\text{Pa-s}])</td>
</tr>
<tr>
<td>(\eta_{\text{V}})</td>
<td>viscoplastic yield strength of particle contact ([\text{Pa-s}])</td>
</tr>
<tr>
<td>(\kappa)</td>
<td>contact consolidation coefficient ([-] )</td>
</tr>
<tr>
<td>(\theta)</td>
<td>hopper angle ([\text{deg}])</td>
</tr>
<tr>
<td>(\phi)</td>
<td>angle of friction ([\text{deg}])</td>
</tr>
<tr>
<td>(\rho)</td>
<td>density ([\text{kg/m}^3])</td>
</tr>
<tr>
<td>(\sigma)</td>
<td>normal stress ([\text{kPa}])</td>
</tr>
<tr>
<td>(\sigma_a)</td>
<td>adhesion strength ([\text{kPa}])</td>
</tr>
<tr>
<td>(\sigma_1)</td>
<td>major principal stress ([\text{kPa}])</td>
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<tr>
<td>(\sigma_0)</td>
<td>isostatic tensile strength ([\text{kPa}])</td>
</tr>
<tr>
<td>(\tau)</td>
<td>shear stress ([\text{kPa}])</td>
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</table>

Symbols

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</tr>
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<td>Hamaker constant ([\text{J}])</td>
</tr>
<tr>
<td>(d)</td>
<td>particle size ([\mu\text{m}])</td>
</tr>
</tbody>
</table>

Indices

- \(a\) : adiabatic
- \(b\) : bulk
- \(c\) : compressive
- \(e\) : effective
- \(el\) : elastic
- \(H\) : adhesion (Haft-)
- \(i\) : internal
- \(K\) : total contact
- \(l\) : liquid
- \(m\) : molar
- \(M\) : centre of Mohr circle
- \(min\) : minimum
- \(N\) : normal
- \(p\) : pressure
- \(pl\) : plastic
- \(pre\) : pre-shear
- \(R\) : radius of Mohr circle
- \(s\) : solid
- \(st\) : stationary
- \(t\) : time dependent
- \(T\) : tangential
- \(V\) : volume
- \(VdW\) : Van Der Waals
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Author’s short biography

Jürgen Tomas

J. Tomas studied process engineering, special field system engineering, at the Institute of Technology (TH) Merseburg from 1971 to 1975. He finished his studies with Diploma-theses concerning crystallization and particle agglomeration in a liquid fluidized bed reactor. After his research work at the department of Mechanical Process Engineering and Mineral Processing of the Institute of Mining Technology (Bergakademie) Freiberg, he wrote his PhD-theses “Investigations on Flow Behaviour of Moist Soluble Bulk Materials” in 1982. R/D-activities followed this work, e.g. design of silo plants for power stations, cement mills, process industries as well as port installations for ores, consultant for trouble shouting in chemical plants. From 1988 to 1989, he taught as Assistant Professor at the Addis Abeba University (Ethiopia). In 1991 he wrote the ScD-theses (Habilitation) “Modelling the Flow Behavior of Bulk Materials on Particle Interaction Forces Basis and Application in Bunker Plant Design” at the Bergakademie Freiberg. After substituting a professorship in Mechanical Process Engineering at the Bergakademie Freiberg from 1992 to 1993, he was appointed professor at the School of Technology (HTWS) Zittau/Görlitz and, in 1994, full professor at the Otto-von-Guericke-University Magdeburg. From 1995 to 1997, he was chairman of the joint research field 385 (DFG Sonderforschungsbereich) “Building Material Recycling”. His principal research activities are in particle characterization, particle and powder mechanics, comminution, solid-solid separation, press filtration, bulk solids storage and handling.