

Adhesion of Dry Powders to Process Equipment Surfaces
IFPRI Research Project Proposal

Primary investigator:

Dr. Jan Henrik Finke

Institute for Particle Technology, Technical University Braunschweig
Center of Pharmaceutical Engineering, Technical University Braunschweig
Fraunhofer Institute for Surface Engineering and thin films IST, Braunschweig

1. State of research

Particle adhesion to process equipment is mostly unwanted and may become a critical problem in many process industries with pharmaceuticals and foods being only two prominent examples. It may happen under different stress conditions, by autogenic stress of powder beds in hoppers, by high normal stresses in compaction processes, or by shear stresses in feeder screws or other shear-intensive devices. In the interaction, the properties of both the particle and the substrate as well as the process and environmental conditions influence the outcome of the contact resulting in adhesion or detachment. If sufficiently strong adhesive forces keep one particle adhered to equipment surfaces, it depends on the interaction of the particle with its surrounding particles and on the stresses in the process, whether further material builds up or the formerly adhered particle is detached due to high enough (cohesive or in mixtures of particles also adhesive) particle-particle interactions. This interplay also determines whether sticking continuously grows over the number of compression cycles and when and at which level it reaches a steady state (Paul et al., 2017a). Material properties influencing particle adhesive forces comprise its chemical nature, crystal form and habitus, particle size (Sanioccki, 2014) and shape, as well as its deformation behavior, rendering plastic materials more sticky (Paul et al., 2017b). In formulations of different materials, also the weight fractions or surface area fractions play an important role. Process parameters include the magnitude of the stress in the particle-surface contact, its direction and nature as well as its kinetics. In the literature, adhesion phenomena, such as sticking, were mostly studied on an empirical basis, which makes many results hard to transfer or to apply in a generalized way.

The adhesive forces of particles in contacts to other particles or surfaces are physico-chemically mainly determined by van der Waals, electrostatic, and hydrogen-bonding forces (Shah et al., 2017). Inverse gas chromatography and sessile drop tensiometry are frequently applied to measure surface energies of solids and to differentiate polar and dispersive contributions. Additionally, a lower melting point was identified as a risk factor towards adhesion (Roberts et al., 2004). Towards particle deformation, contact mechanics models such as the model of Johnson, Kendall, and Roberts (Johnson et al., 1971) are applied to calculate the contact deformation and by that the bond contact area. However, connecting data from literature models to directly measured adhesive forces of real powders frequently leads to pronounced differences. This may be traced back to I) the complexity of particle-surface interactions due to the roughness of particles and surfaces causing a low number of point contact, II) the spatial distribution of adhesion-relevant properties on surfaces, and III) the changes in surface properties (mainly of the particles) due to their (contact) deformation under stress (Waknis et al., 2014).

The role of **environmental conditions** in adhesion processes is crucial by different mechanisms. I) **Humidity** may change the composition of the solid by means of plasticization, altering its thermal behavior and mechanical properties, II) surface-adsorbed water may enhance polar interaction, also facilitating bridging hydrogen-bonds over longer distances (Price et al., 2002), III) reduce electrostatic interactions (LaMarche et al., 2010), and IV) condensed surface water may facilitate capillary forces in the contacts. This may lead to higher sticking in certain ranges of material moisture resulting from environmental conditions (Danjo et al., 1997).

In application-oriented studies, the sticking propensity should be derived by measuring surrogate parameter directly in production processes. A prominent example is the measurement of take-off or scraping force, determining the peak force necessary to detach a tablet from the lower punch by tangential stressing, which results in **shear stress** in the interface between tablet and punch

(Danjo et al., 1997). When trying to compare such pragmatic measures with sophisticated direct analyses of the adhesion force by atomic force microscopy (AFM) on single particle level, contradicting results were reported (Saniocki et al., 2013; Wang et al., 2004; Wang et al., 2003). This may be based on the fact that **the particle-particle-wall interactions of neighboring particles are not sufficiently taken into account and differentiated** in this setup (Talebi et al., 2025). Other practical approaches try and measure the detachment force of the upper punch from the tablet, which presents a **normal tensile** detachment setup (Waimer et al., 1999), challenging the sensitivity of the measurement. These different stress states leading to detachment may result in different interfacial strengths in shear and normal direction. Further, but less practical tests to quantify the work of adhesion of single particles are centrifugal and drop tests (Ermis et al., 2011; Talebi et al., 2025; Zafar et al., 2014), in which the particles are detached by their gravitational or inertial force. A simplistic lab approach was recently presented applying a stainless steel mixer mill setup to determine material adhesion to the grinding ball as a surrogate for sticking in tableting (Brocca et al., 2025). Differences between single-particle and tableting sticking results may be explainable by the different **failure modes**, which may in the latter case result in the detachment of the tablet or a breakage inside a plane of the tablet, leaving sticking material on the punches, if the adhesive forces outweigh the cohesive forces in the tablet (Saniocki et al., 2013).

Finally, some authors defined different sticking indices as probable measures to predict the sticking propensity of materials by measurements apart from sticking itself. A high sticking index of Saddick and Davé predicts a high sticking propensity, introducing the quotient of the particle-wall interactions (measured as the wall friction angle) divided by the particle-particle interactions (measured as the kinematic angle of internal friction) (Saddik and Dave, 2021). On the contrary, the sticking index of Capece more focuses on the formulation and takes the difference of the tensile strengths of formulations containing an API and the tensile strength of pure API tablets divided by the surface area fraction of the API in the formulation into account (Capece, 2019). In his approach, however, a high sticking index correlates with a low sticking propensity.

2. Objectives

The first 3 years of this project will focus on the adhesion of **particles, especially in ensembles**, under **high, mainly uniaxial stress** conditions in the particle contacts. Tablet compression and the formation of sticking to stainless steel tools will be focused on.

According to the state of research, knowledge on the effect of **balances between particle-particle** (of the same and different materials in formulations) and **particle-wall forces** needs to be extended to elucidate and **quantitate their influences** on the sticking propensity. In this, especially the **anisotropy of both the bond formation and the stress states during detachment** scenarios must be studied in detail. This aims at finally deriving **process models** that can predict sticking propensity and its location based on defined and possibly simple lab tests.

To facilitate this evaluation, a systematic method and study design will be performed to measure the forces and determine and **differentiate the process stresses and product strengths in all directions necessary**. A new **steel coupon compression test** will be established to produce specimens that can be stressed as directly as possible with **pure normal tensile stresses** or **tangential shear stresses** acting in the interface between particles and steel. Additionally, the bulk level studies are combined with **single-particle compression and adhesion** experiments

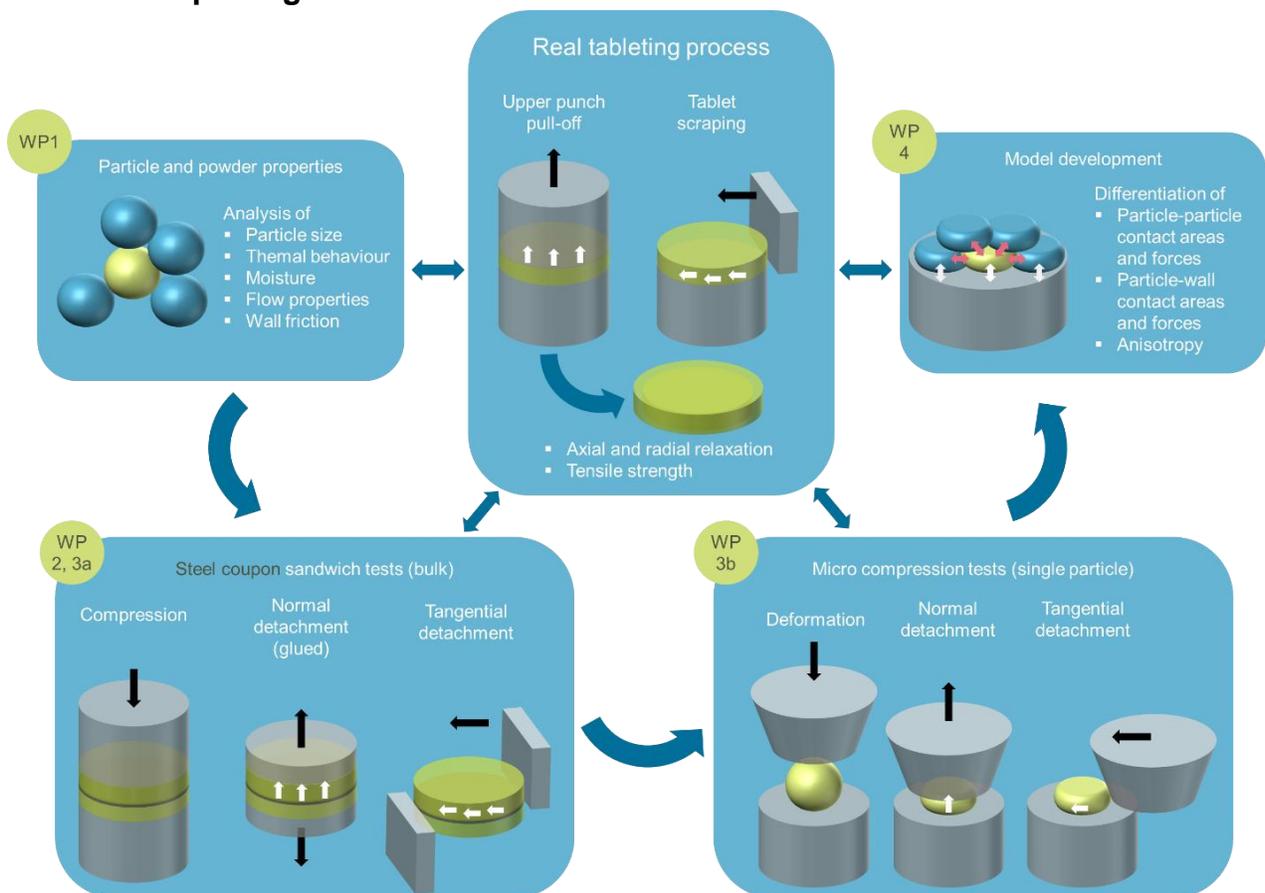
applying a nanoindenter and microscopy to study the effect of **particle deformation, contact area formation, and resulting detachment forces** in sufficient detail.

It is hypothesized that the different stress states during detachment may provide information on the **anisotropy of inter-particulate and particle-surface interactions** and the **role of surface roughness in adhesion** and mechanical interlocking in inter-particulate interactions.

Objectives are

1. Development of a coupon test for tensile and shear testing of the material-steel interface
2. Facilitation of the temperature and humidity control of a compaction simulator
3. Systematic study design to capture all formulation and process parameters influences
4. Performance of comparative studies of micro-compression and detachment trials of single particles (nanoindenter) and bulk/tableting studies on a compaction simulator
5. Elucidation of the effect of anisotropic behavior and stress distribution on sticking
6. Derivation of models for the prediction of sticking propensity based on material properties and lab trials, including the determination of minimal trial setups for reliable results

3. Work packages



WP1 Material characterization

To provide profound understanding of material properties and environmental conditions on the adhesion and sticking of particles, different types of materials are selected, comprising organic crystalline (e.g. ibuprofen, mannitol, lactose), organic amorphous (e.g. HPMCAS (hydroxypropyl

methylcellulose acetate succinate), PVP (polyvinyl pyrrolidone), Eudragit® E PO), and inorganic materials (e.g. limestone). To capture the effect of humidity exposure, all materials are tested after conditioning to different humidities ranging from 10 to 80 % relative humidity. In order to enable clear correlation of the structures and sticking behaviour to material properties, the (conditioned) starting materials are examined in detail. Particle size and shape are determined using laser diffraction, dynamic image analysis and SEM. The thermal and moisture properties of the materials, in particular their change under altered humidity conditions, are examined using thermogravimetry and differential scanning calorimetry as well as a thermobalance, water activity meter, and Karl Fischer titration. To test sticking index approaches from the literature (Capece, 2019; Saddik and Dave, 2021) and to use these data for further evaluation, power flowability will be tested with a ring shear tester and a powder rheometer and wall friction tests will be performed.

WP2 Method development

a) Coupon-based characterization of interfacial strengths

A coupon test is developed based on stainless steel coupons (flat, round discs with the size of the die and tablet) with defined surface properties regarding roughness or coating. These are used as the lower or the mid layer in bi- or tri-layer compaction, respectively. This facilitates to measure the interfacial strength outside the compaction simulator with higher precision in the low force range on material testing machines. To this end, test rigs are developed that apply direct normal tensile stress against the compression direction or shear stress perpendicular to the compression direction and tangential to the interface. It will be necessary to evaluate adhesives/glues for the setup to measure direct tensile stress to avoid artificial alterations of the specimens. The statistical deviations of the setup are analyzed to determine the necessary number of samples and tests.

b) Conditioning of compression devices

To study the crucial effect of humidity and temperature on the sticking propensity, both setups of powder bulk and single particle compression are extended to become conditioned (temperature 10-60 °C, relative humidity 10-80 %). To this end, a setup for air conditioning of the respective process chambers is developed. It should be based on the tempering, mixing, and flow controlling of a dry and a wet gas stream to achieve the set humidity and temperature. Temperature and humidity sensors will be applied to control the conditioning state. On the compaction simulator, the temperature in the compression zone can directly be manipulated by a temperature-controlled die. In the case of single particle stressing on the nanoindenter, it may be necessary to passively condition the sample chamber by setting up according salt solutions and allow for sufficient equilibration times. Additional to the conditioning of the measuring and production devices, the powders are stored and conditioned under controlled environment in climate chambers.

WP3 Systematic study of adhesive properties on bulk and single-particle scale

a) Coupon-based compression tests of powder bulks

Systematic studies of the interfacial properties between particle ensemble (tablet) and steel surface are performed by producing coupon-based specimens under different compression stresses and kinetics on a compaction simulator. Sticking studies without coupons are additionally performed with punches with removable tips to quantify the sticking via weighing. During these trials, the in-die data including radial die-wall stress are analyzed to provide information on the axial-radial stress distribution. Furthermore the deformation properties are evaluated by compressibility analysis and application of according models (**Wünsch et al., 2019**). Tablet scraping forces are measured with an instrumented scraper and stresses are calculated. As the

products of these trials, the tablets are analyzed towards their weight, dimensions, and breaking force to derive porosity and tensile strength (by Brazilian test evaluated according to Fell and Newton (Fell and Newton, 1970)). Additionally, breakage tests by tablet band indentation to cause horizontal breakage (such as delamination (Castrati et al., 2016)) are performed to differentiate the strength in vertical and horizontal direction and to derive a measure for the structural anisotropy along these different breakage plains. To assess the local porosity on tablet surfaces, which may also provide information on the superficially local particle deformation, mercury porosimetry of whole tablets is performed for selected samples. Additionally, the elastic recovery of the tablets is studied in detail in axial and radial direction. All this is to collect and derive information on the bond formation to differentiate the normal and the radial particle deformation.

b) Micro-compression and detachment tests of single particles

Single particles of the materials are subjected to micro-compression using a 100 or 500 μm flat punch, preferably made of stainless steel, in a nanoindenter on a stainless steel substrate. During the detachment phase, the normal adhesive force is measured. Particles are assessed via microscopy before and after the compression and detachment to detect the displacement (particle sticks to the flat punch) or the residual deformation (particle sticks to the substrate). By coating the flat punch with an anti-adhesive coating (cooperation with Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig, Germany), particles remain attached to the substrate after decompression. In this case, the indenter tip will be used to stress the particle parallel to the substrate surface to cause and measure lateral detachment under shear stresses.

WP4 Data evaluation and model derivation

Data collected on single-particle and bulk level are used to differentiate the role of lateral confinement and contact deformation on the development of particle adhesion and surface sticking in this specific case. To this end, the detachment stresses measured on both scales are compared by applying the combination of the JKR model with the model of Rumpf in case of tablets, as we proposed in (Wünsch et al., 2021). This model combination must, however, be extended to also take the anisotropy in normal and radial direction into account, which should be correlated to tablet strength measurements with different failure plains (WP3a). Furthermore, it needs to be extended to the particle-surface case as the sticking-relevant interface.

The modification of material properties by the sorption of water from the environment will be evaluated via the moisture content and changes in material properties (WP1) as well as the effect on the glass transition temperature of amorphous materials (by DSC). The applicability of the concept of homologous temperature will be evaluated.

The above mentioned correlations and derived knowledge will be integrated into process models that aim to trace correlations back to physical and chemical principles to link the effects of material and process parameters to adhesion/sticking propensity. It will be evaluated, which separate and possibly simple tests suffice to predict the sticking propensity with necessary precision.

As a practical guide for applications, a regime map to rank the sticking propensity and possibly attribute mechanistic sticking causes will be developed. It is assumed that the scaling parameter will include material properties (e.g. homologous temperature, deformation resistance, axial-radial stress distribution, moisture) on the one, and process parameters (e.g. compression stress, compression speed) on the other hand.

Second 3 year period:

In the second phase, an extension of the test setups and models to shear stress-mediated particle-surface contact and adhesion such as in feeder or extruder screws is planned. It is envisioned that the establishment of a shear zone in a two or three roller mill with differential speed of the rollers may be applied to provoke a force-controlled sticking to the faster roll, which can be quantified by image analysis and by scraping and collecting material.

4. Support from IFPRI members

The project would benefit from support and input of interested IFPRI members towards the choice on model materials to study and the further definition of parameter ranges, if applicable. The surface properties of the steel could be a further topic for support by the IFPRI members. Any input on the relevance and specificities of industrially occurring sticking and background knowledge is additionally highly welcome to be discussed.

5. Synergies in the scientific environment and to other projects

The project will be performed in the Center of Pharmaceutical Engineering at the Technical University Braunschweig, providing a transdisciplinary research environment, where the topic of this proposal perfectly fits in. The proposal will extend the research area of particle adhesion and sticking in the Division of Pharma and Bioparticle Technology at the Institute for Particle Technology, which I am leading. It will synergistically cooperate with a current industrial PhD project with Roche on sticking and picking, which focuses on the detection of sticking to provide a practical approach to evaluate the sticking propensity in industrial applications. This IFPRI project proposal in contrast focuses more on fundamental research, sophisticated mechanical analyses, and single particle experiments, which are not carried out in the Roche project. Additionally, the cooperation and my additional position at the Fraunhofer Institute for Surface Engineering and thin films IST facilitates the testing and tailoring of surface coating against sticking. In this cooperation, also the adhesion control for inhalable particles on inhaler surfaces and on milling equipment are studied in addition to a long lasting track record in coating development of tableting punches. A joint project with Fraunhofer IST is in preparation to focus on modifying and analyzing machine surface coating properties such as chemical nature and surface topography in the topic of adhesion in milling, tableting, and melt processing. This cooperation may facilitate the application of new coatings also in the proposed IFPRI project. Finally, I am preparing a fundamental research funding proposal for submission to the German Research Foundation in Q1/2026, focusing on the analysis and modelling of the effect of humidity on the tableting behavior and the kinetics-dependence of pharmaceutical materials, which may synergistically complement this proposal towards the study and interpretation of humidity effects.

6. Recourses

At the Institute for Particle Technology and the Center of Pharmaceutical Engineering, we have access to the infrastructure necessary for this project: Compaction simulator with temperature-controlled die, climate chambers, material testing machines, tablet tester, atomic force microscope, nanoindenter, differential scanning calorimetry, powder rheometer, ring shear tester, loss-on-drying balance, water activity meter, Karl Fischer titration, and Raman microscope, if necessary.

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