

ADHESION FORCE MEASUREMENTS BETWEEN PARTICLES UNDER VARIATION OF RELATIVE HUMIDITY

Felix Belter^{*1}, Viktor Brandt², Harald Kruggel-Emden², Eberhard Schmidt¹

¹Institute of Particle Technology, University of Wuppertal, Wuppertal, Germany

²MVTA, Technische Universität Berlin, Germany

ABSTRACT

The processing of granular materials is often associated with dust emissions that arise from the mechanical or fluid-induced detachment of microscale dust particles adhering to macroscale bulk particles. The dustiness of granular materials depends, among other factors, on the adhesive forces between individual particles. Moisture content, which is governed by the ambient relative humidity, influences these interparticle adhesive forces and thus affects dust emission behavior. For the numerical prediction of dust emissions, a quantitative determination of interparticle forces is therefore essential. Consequently, investigating the influence of moisture content on adhesion forces between particles in bulk materials is crucial for understanding and predicting dust emissions. One method for measuring adhesive forces at the particle scale is atomic force microscopy (AFM).

This study presents an experimental setup designed to determine the adhesive forces between microscale and macroscale particles. The measurements are carried out in a custom-built climate chamber equipped with integrated humidification and dehumidification units, ensuring a defined relative humidity during the measurement process. Force-distance curves are recorded using an AFM and subsequently analyzed to quantify the adhesion forces. By systematically varying the relative humidity in each measurement series, its influence can be investigated in a controlled manner.

The study reports initial measurement results, identifies key challenges, and outlines corresponding solution approaches.

KEYWORDS

Bulk solids, atomic force microscopy, adhesive force measurements,
relative humidity, dustiness

1 Introduction

The processing of granular materials is associated with dust emissions. These emissions result from the mechanical or fluid-induced detachment of microscopic dust particles adhering to macroscopic bulk particles [1]. The dust generation of granules depends, among other factors, on the adhesive forces between individual particles [1]. A key factor influencing these interparticle adhesive forces – and thus determining dust emission behavior – is the material moisture content, which is influenced by the surrounding relative humidity [2]. For numerical and model-based prediction of dust emissions, a quantitative determination of interparticle forces is essential [3]. Therefore, investigating the influence of relative humidity on the adhesive forces between particles in bulk materials is crucial for understanding and predicting dust emissions.

Atomic force microscopy (AFM) has become an established tool for the direct measurement of adhesive forces at the particle level [4, 5]. However, standard AFM systems are often optimized for high-resolution topography scans on surfaces and offer limited flexibility for studying particle-particle interactions under variable environmental conditions. As a result, integration into a climate chamber and the simultaneous observation of macroscopic particles and the micrometer-sized AFM cantilever pose a challenge.

The objective of this work is therefore to develop and validate a specially adapted experimental setup that enables the direct measurement of adhesive forces between individual microparticles and a macroscopic particle surface under controlled relative humidity. To meet the specific geometric and experimental requirements, a modular and cost-effective measurement system was implemented. This system combines an educational atomic force microscope with a custom-built digital light microscope, allowing direct visual positioning of the AFM cantilever on the surface of the macroscopic particle. The entire setup is integrated into a custom-designed climate chamber, which permits systematic variation of the relative humidity.

This study presents the design and functionality of this new type of measurement setup. Initial measurements of adhesive force as a function of relative humidity are presented to demonstrate the system's capabilities. Furthermore, central challenges – such as sample preparation and force measurement – are identified, and potential solutions are discussed. The insights gained here are intended to lay the foundation for a systematic quantification of moisture-dependent adhesive forces, thereby contributing to a better physical understanding and improved predictability of the dust propensity of bulk materials.

2 Methods and Experimental Procedure

2.1 Fundamental Concept

The objective of the present experimental study is to conduct reproducible adhesion force measurements between microscaled and macroscaled particles. The device employed for this purpose is an Educational AFM by THORLABS. It should be noted that, as the device is designed for educational purposes, its resolution is not comparable to the standards used in research. However, the open design of the device allows for the deliberate consideration of environmental conditions, such as relative humidity in this case, and provides visual insight into the operational processes of the device. This configuration enables in situ sample preparation using colloidal probe techniques [4]. Immediately thereafter, measurements can be initiated without the need for sample transfer.

The functional principle of the atomic force microscope for recording force-distance curves is schematically illustrated in Figure 1. Laser light from a laser source is focused onto the cantilever via an optical fiber and a lens. The reflected light is directed onto a four-quadrant photodiode. The laser light is recorded as a measurement signal and converted into a voltage signal. When the cantilever deflects, the position of the laser spot on the photodiode shifts, consequently altering the voltage signal. This results in a deflection-dependent voltage signal [6].

A sample stage located beneath the cantilever is equipped with a piezoelectric membrane, enabling precise movements of the stage. The movement of the sample stage is controlled via a corresponding computer.

In addition to the conventional setup, a custom-built digital microscope has been integrated into the AFM. This microscope is aligned with the cantilever and allows visual observation of the sample preparation and measurement process.

The AFM is installed in a climate chamber. Within this chamber, the relative humidity can be adjusted in the range of 40% to 90% to operate the device under defined environmental conditions.

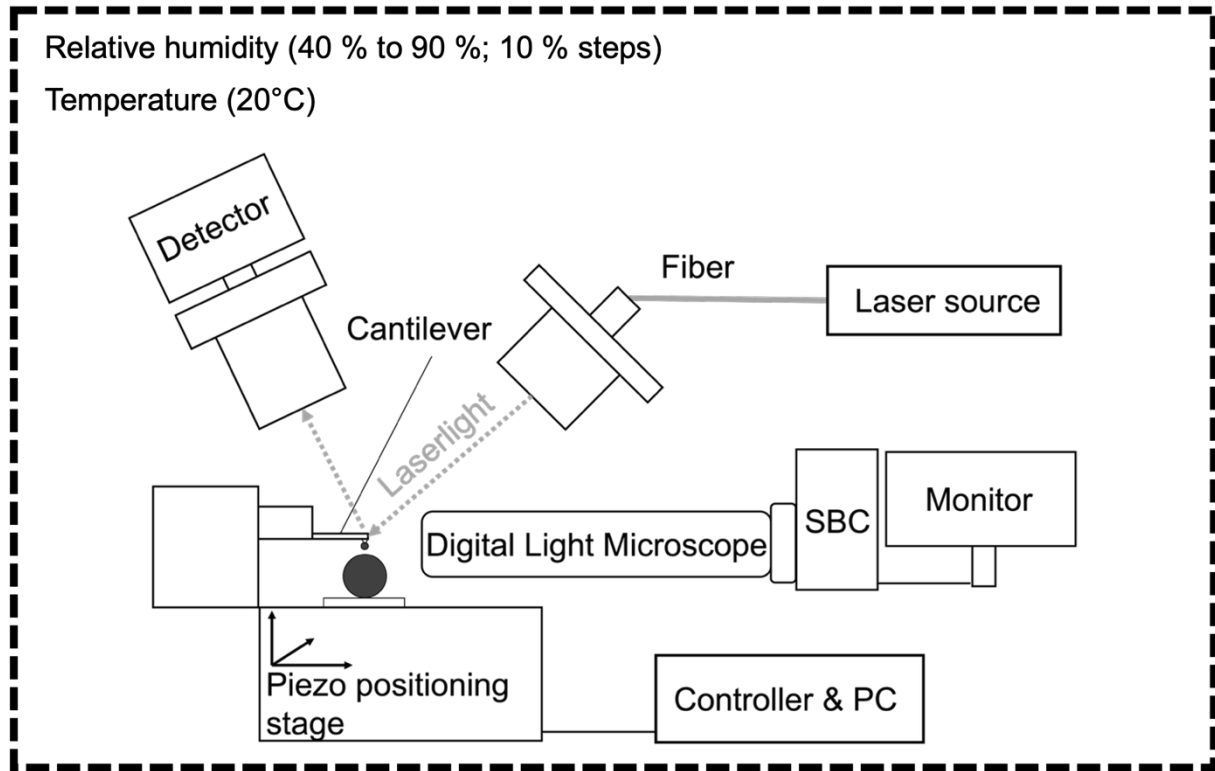
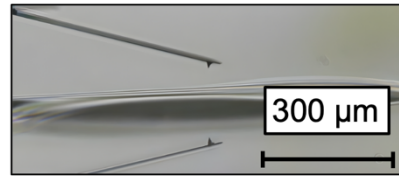


Fig. 1: Schematic representation of the atomic force microscope used, including the integrated digital light microscope.

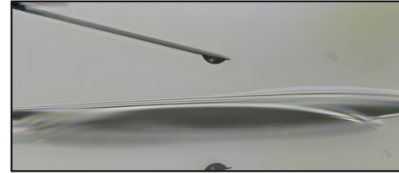
2.2 Experimental Procedure – Sample Preparation

This work describes sample preparation as the process of attaching a microscopic calcium carbonate particle to the tip of the cantilever. The preparation was carried out directly in the atomic force microscope (AFM). Additionally, a custom-built digital Light microscope was designed and constructed, which was then coupled to the AFM. The digital light microscope comprises a 12-megapixel HD camera, a single-board computer, and an objective lens (KEYENCE VH-Z20R). The integrated digital light microscope provides visual insight during the preparation process.

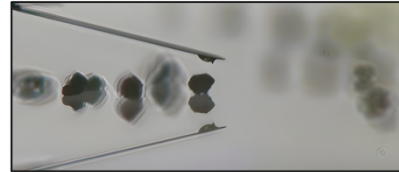
1. application of UV-curing adhesive onto a coverslip



2. application of UV-curing adhesive to the cantilever tip



3. attachment of a CaCO_3 particle to the adhesive-coated tip



4. curing of the adhesive by UV light

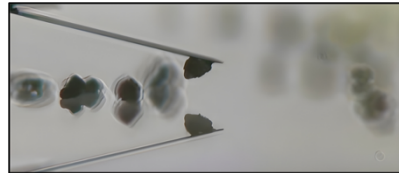


Fig. 2: Steps for sample preparation to reproducibly create a colloidal probe for recording force-distance curves

In the first step, a UV-curing adhesive was applied to a cover slip using tweezers and positioned on the sample stage (see Fig. 2, step 1). The sample stage was then manually approached toward the cantilever tip using adjustable screws. Once the tip immersed into the adhesive, it was retracted, leaving a droplet adhered to the tip (see Fig. 2, step 2). In the third step, calcium carbonate particles (Eskal 45 by KSL Staubtechnik) were dispersed onto a cover slip and placed on the sample stage. Suitable particles were visually identified using a digital light microscope. The particles had to be as spherical as possible to ensure comparability with Discrete Element Method (DEM) simulations and facilitate subsequent theoretical calculations. Additionally, they had to be isolated to guarantee that only a single particle adhered to the cantilever. The depth of field was used to manually determine whether the particle was precisely located beneath the cantilever (see Fig. 2, step 3). If this was the case, the particle was approached toward the cantilever tip until it adhered to the adhesive (see Fig. 2, step 4). In the final preparation step, the adhesive was cured by exposing it to a UV lamp for 20 seconds in accordance with the manufacturer's specifications.

2.3 Experimental Procedure – Recording Force-Distance-Curves

After sample preparation, force-distance curves were recorded in a climate-controlled chamber. An air conditioning system maintained a constant room temperature of 20 °C, while the relative humidity was regulated using a humidifier and dehumidifier unit.

Macroscale particles consisted of calcium carbonate with a grain size of approximately 1 mm. To remove adhering fine particles ($x < 100 \mu\text{m}$), the particles were wet-washed. This ensured that the measurement occurred between the macroscale particle and the previously prepared microscale particles on the cantilever, preventing fine particles adhering to the macroscale particle from attaching to the cantilever. Subsequently, the particles were dried in an oven at 105 °C for 24 hours. After brief cooling under standard laboratory conditions (20 °C and approximately 40 % relative humidity), the particles were fixed onto a cover slip using a carbon adhesive pad before being placed on the AFM sample stage.

The manual approach of the sample stage to the cantilever was performed using adjustment screws. Throughout the approach process, the distance between the microscale and macroscale particles was visually monitored (see Fig. 3). Shortly before contact between the particles, the manual approach was stopped, and the recording of a force-distance curve began. For this purpose, the piezo membrane expanded by 4 μm in the vertical direction to establish contact between the particles. The distance results from the piezo expansion factor and the corresponding applied voltage (see Chapter 3).

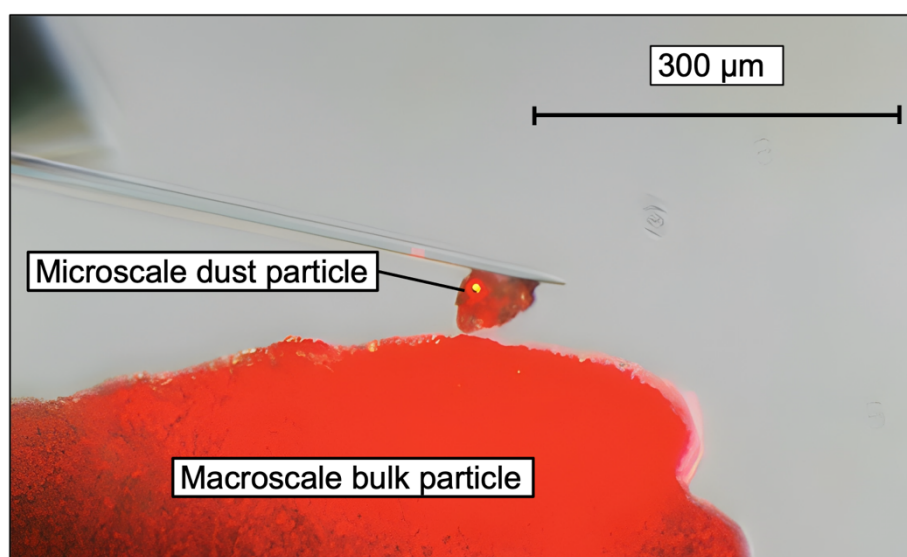


Fig. 3: Image of the prepared CaCO_3 particle (microscale dust particle) captured using a digital light microscope prior to contact with the other CaCO_3 particle (macroscale bulk particle).

The measurements comprised ten force-distance curves per relative humidity level. The measurement series started at 40 % relative humidity, which was incrementally increased in 10 % steps up to 90 %. The relative humidity was verified using a mobile hygrometer (Temtop M2000) in close proximity to the AFM measurement zone. According to the manufacturer's specifications, the fluctuation range of the relative humidity is ± 5 %. A conditioning time of 30 minutes was maintained between each step to allow the particles to adapt to the respective humidity level. Literature indicates that the adsorption of water on CaCO_3 surfaces occurs within a few seconds to minutes [7, 8], ensuring that the 30-minute waiting period was sufficient for establishing adsorption equilibrium.

3 Results

3.1 Interpretation of a Force-Distance Curve

In the following, the term “sample” refers to the macroscopic particle on the sample stage, while the “colloidal probe” denotes the microscopic particle adhered to the tip of the cantilever. To illustrate the interpretation, Figure 4 shows an ideal force-distance curve. Recording a force-distance curve is a cyclic process that can be divided into four phases:

Approach: The sample is moved toward the colloidal probe. At large distances, no measurable force is detected. Once the colloidal probe enters the range of attractive forces (e.g., van der Waals forces), it may suddenly snap onto the surface, an effect known as “jump-to-contact” [9]. This effect is observable as a small decrease in the voltage signal value at the photodiode as the cantilever is deflected.

Contact: The colloidal probe is in physical contact with the sample surface. As the sample is pressed further against the colloidal probe, the cantilever bends upward. Consequently, the voltage signal value at the photodiode increases

Retraction: The direction of the sample movement is reversed, and it is withdrawn from the colloidal probe. Due to adhesive forces, the tip remains attached to the surface. The cantilever now bends downward, due to the adhesion force between the sample and the colloidal probe. This adhesion can be caused by various mechanisms such as van der Waals forces, capillary forces, or chemical bonds [9].

Pull-off: As the sample continues to move away, the downward deflection of the cantilever increases. The restoring force of the cantilever acts against the adhesive force. At the point where the restoring force exceeds the maximum adhesive force, the colloidal probe abruptly snaps back to its neutral position [6, p. 91].

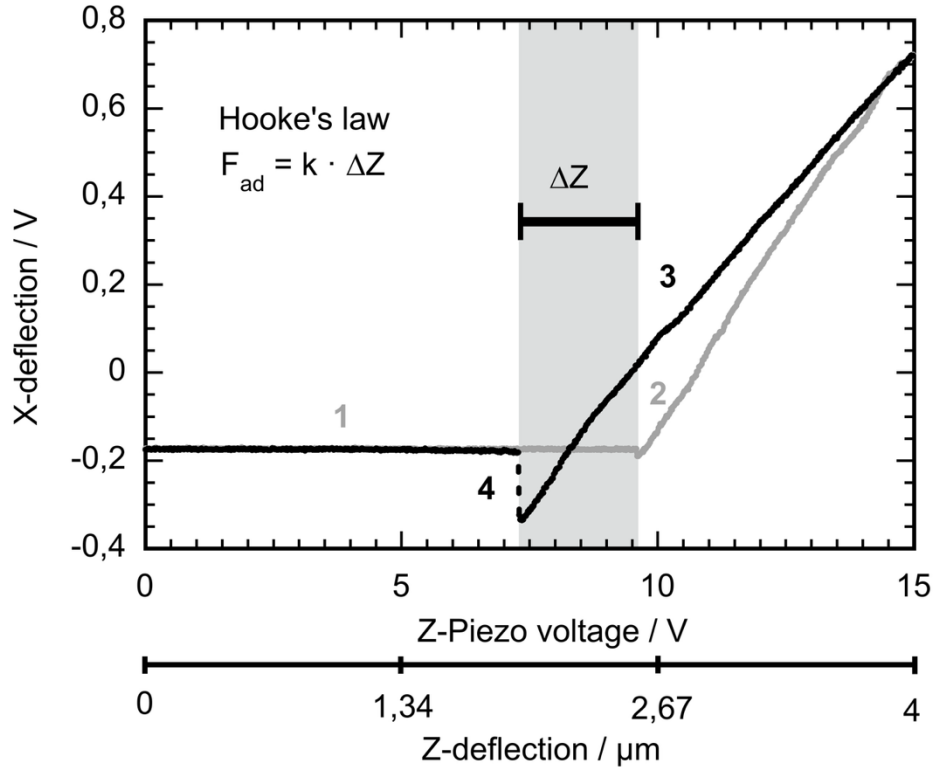


Fig. 4: Illustration of an ideal force-distance curve depicting the various phenomena: 1. approach, 2. contact, 3. retraction, 4. pull-off.

3.2 Adhesion Force

To determine the adhesion forces from the recorded force-distance curves, the deflection of the piezoelectric membrane in the vertical direction is utilized. This deflection, denoted as ΔZ , is calculated using the piezo-specific expansion coefficient c_p , which specifies the height change of the piezoelectric membrane per volt, and the corresponding voltage difference of the piezoelectric membrane between the contact and pull-off events in the force-distance curve [6, p. 131–132] (see Fig. 4).

$$c_p = 0,26 \mu\text{m} \cdot \text{V}^{-1}$$

$$\Delta Z (Z_p) = c_p \cdot \Delta Z_p$$

The adhesion force can subsequently be approximated using Hooke's Law, based on the spring constant k (as provided by the manufacturer) of the cantilever and ΔZ .

$$F_{ad} = k \cdot \Delta Z$$

Figure 5 illustrates the arithmetic mean values of the calculated adhesion forces for each relative humidity level ($N = 10$) and the empirical standard deviation. The measurement results indicate that adhesion forces are influenced by the increase in relative humidity. At 40 % relative humidity, the results exhibit a significantly lower adhesion

forces compared to higher humidity levels. In the range of 50% to 80%, the values do not differ significantly when considering the empirical standard deviation. Only the measurements at 90 % relative humidity show a clear increase of the adhesion force.

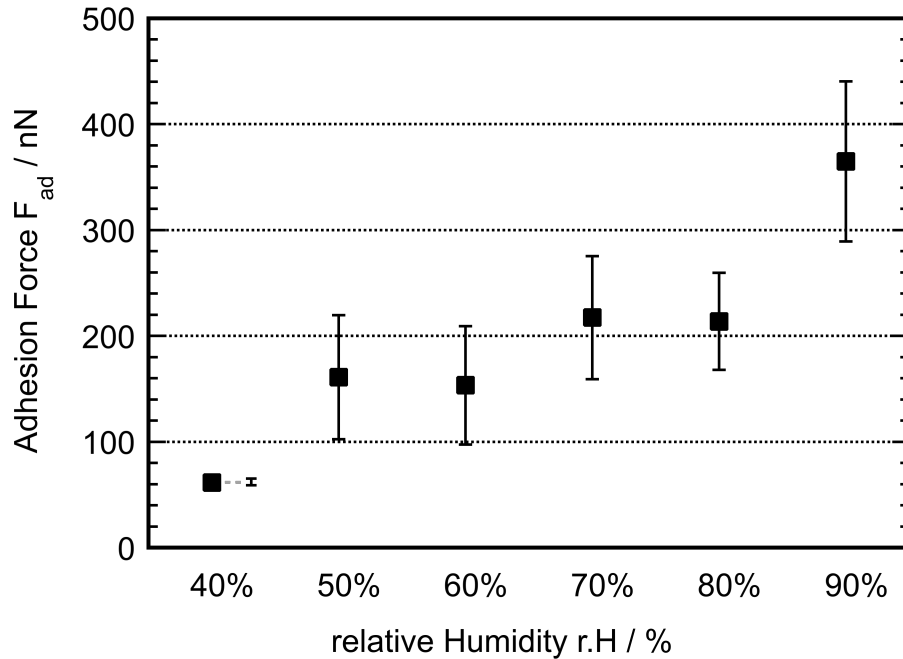


Fig. 5: Adhesion force values in dependence on the relative humidity based on force-distance-curves ($N = 10$)

4 Conclusion

This study investigates a methodological approach for the quantitative determination of adhesion forces between microscaled and macroscaled particles under targeted variation of relative humidity. The objective of this methodology was to conduct reproducible measurements of adhesion forces, including sample preparation using colloidal probe techniques. The aim was to strike a pragmatic balance between scientific standards and practical feasibility.

By systematically recording adhesion forces under different humidity conditions, trends in the progression of adhesion forces as a function of relative humidity were identified using calcium carbonate as the sample material. The results showed that adhesion forces increased with rising relative humidity, which aligns with findings reported in the literature [10]. It is notable that the adhesion force increases significantly at 90% relative humidity. In light of the existing literature, this substantial increase in force may indicate the formation of capillary condensation.

Continuous visual monitoring of the sample preparation and measurement process confirmed that the measurements indeed occurred between the microscaled and macroscaled particles – a confirmation that is still a challenge in studies using the same model of the device [1, p. 62–76].

However, the quantitative values of the adhesion forces are subject to critical consideration, as the atomic force microscope (AFM) used in this study does not achieve the resolutions typically employed in research. Furthermore, the determined adhesion force values exhibit standard deviations that do not allow a quantitatively reliable distinction in the relative humidity range of 50% to 80%. And finally, the rough surface of the Material and the non-spherical shape of the particles used complicates comparison with theoretical calculations or other studies, making it difficult to assess the significance of the results and, consequently, to evaluate the reliability of the methodology presented here.

Future work must further examine the methodological setup regarding the reproducibility and significance of the quantitative results. For instance, investigations using reference materials with smooth surfaces and ideal sphericity could be conducted to better contextualize the measurement results and to eliminate unknown influencing parameters.

In summary, this study provides a methodological foundation for performing sample preparation using colloidal probe techniques, followed by adhesion force measurements under continuous visual monitoring and variation of relative humidity with a minimalistic experimental setup. And although the results can be utilized for discrete element method (DEM) simulations and dust prognosis models, the quantitative results should be interpreted with caution due to the methodology being rudimentary compared to established technical research standards.

Literatur

- [1] Nadja Woschny, Ein Beitrag zur verbesserten Beschreibung der Schüttgutstaub freisetzung unter Verwendung eines Referenzmaterials, Wuppertaler Reihe zur Umweltsicherheit, Shaker Verlag, Düren, November 2025
- [2] H. Schubert, Handbuch der Mechanischen Verfahrenstechnik, Vol. 1, Wiley-VCH, Weinheim, 2003
- [3] Brandt V., et al., Benchmarking study on scaling options for simulating dust detachment as part of the discrete element method (DEM) in mechanically agitated systems. Powder Technology, 2026, <https://doi.org/10.1016/j.powtec.2026.1-22411>
- [4] Butt H., et al., Force measurements with the atomic force microscope: Technique, interpretation and applications. Surface Science Reports, 59, 1-152, 2005, <https://doi.org/10.1016/J.SURFREP.2005.08.003>
- [5] Kühn N., et al., Determination of interaction forces of (sub)-micron sized particles via the capillary rise method and colloidal probe atomic force microscopy: A combined approach, Journal of colloid and interface science, 680(Pt A), 696-713, 2024, <https://doi.org/10.1016/j.jcis.2024.10.187>
- [6] THORLABS GmbH, Hrsg. EDU-AFM1 EDU-AFM1/M Rasterkraftmikroskop Handbuch, THORLABS GmbH, 4. Dez. 2019.
- [7] Rahaman A., et al., Dynamics of Water Adsorption onto a Calcite Surface as a Function of Relative Humidity, Journal of Physical Chemistry C, 112, 2109-2115, 2008, <https://doi.org/10.1021/JP077594D>
- [8] Arjmandi M., Khayet M., Computational Prediction of the Complete Adsorption–Regeneration Cycle of Functionalized Metal–Organic Frameworks for Atmospheric Water Harvesting, ACS Applied Nano Materials, 9, 2284 – 2300, 2026, <https://doi.org/10.1021/acsanm.5c04879>
- [9] Mokgalapa N., et al., Graphite Particle Adhesion to Hastelloy X: Measurements of the Adhesive Force with an Atomic Force Microscope, Nuclear Technology, 2014, <https://doi.org/10.13182/NT13-9>
- [10] Zimon A. D., Adhesion of dust and powder, 1969, <https://doi.org/10.1007-978-1-4615-8576-3>