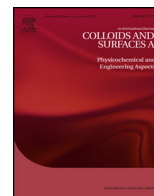




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Investigating the removal of particles from the air/water-interface – Modelling detachment forces using an energetic approach

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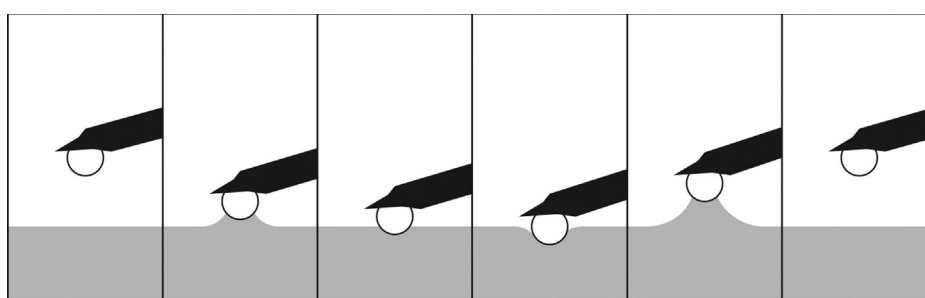
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HIGHLIGHTS

- CP-AFM measurements of the detachment force of colloidal particles from liquid interfaces.
- Spring model for calculating the detachment force by respecting interfacial deformation.
- Comparing the measured and calculated forces with the capillary force model.

GRAPHICAL ABSTRACT



1) Attachment 2) Snap-in 3) Equilibrium 4) Retracing 5) Deformation 6) Detachment

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ABSTRACT

For solid particles it is energetically favorable being situated at the interface of fluid phases and removing a particle from an interface requires a high force. The capillary force effects the particle and causes an interfacial deformation during detachment. In this study the detachment force of hydrophilic and hydrophobic particles is measured via CP-AFM. In order to calculate the detachment force, a simple analytical model is developed and compared with the classical capillary force model. The new model is grounded on an energetic approach in compliance to the interfacial deformation and wetting of the particle. A model spring is assumed for both sub-processes and the force affects onto these two springs. The calculated force in the new model referred to goes better with experimental values than the capillary force model which does not consider interfacial deformation.

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Abbreviations: AFM, atomic force microscopy; A-AW, air to air-water-interface; TPC, three phase contact; W-WA, water to water-air-interface.

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1. Introduction

Interfacial processes play a key role in many industrial processes. Especially the attachment and detachment of particles at fluid interfaces strongly affect the overall performance of processes like agglomeration [1], froth flotation [2], and filtration [3]. Froth flotation uses the selective attachment of hydrophobized particles to the gas-liquid-interface of the bubble. In agglomeration, liquid-bridges are applied between solid surfaces to generate high

attractive forces, e.g. interaction forces control the adhesion of particles at the filter surface of deep bed filters. In the case of deep bed filtration of metal melts, the collection of non-wetted particles in porous ceramic foam filters is increased if the filter surface has the same surface properties. The higher the wetting angle and the surface energy of the solid surfaces, the higher the probability for the presence of nano-bubbles [4]. These create attractive gas bridges which lead to agglomeration of dispersed particles [5] or the collection of dispersed particles onto the inner filter surface [3]. Capillary interactions are also important in the flotation process and have as well been studied with colloidal probe atomic force microscopy [6,7].

From a thermodynamic point of view, it is energetically favorable for particles to be placed at the interface of two fluid phases. Compared to molecular surface-active substances like surfactants the adsorption of particles is not a reversible process. Therefore the adsorption of particles cannot be described by an adsorption equilibrium for small particles. This is due to the magnitude of the energy of adhesion, which is much larger than the thermally induced kinetic energy $k \cdot T$ [8,9]. Thus once a particle is trapped in an interface, it is difficult to remove it again.

Many authors have measured and reported capillary forces acting on microspheres which are in contact with a fluid interface, either if the particle has been moved from the gas phase (air) into an air–water interface (drop, system A-AW) [10–12] or in the opposite direction from a water phase into a water–air interface (bubble, system W-WA) [10,12–19]. Also, investigations using particles with sharp edges can be found in literature [20]. All these measurements provide information on the behavior of particles at fluid interfaces, the forces acting on the particle during attachment and detachment and the surface properties of the particle. Numerical models for calculating the process are reported as well [21,22]. In this paper, we report on the measurements of the capillary forces between hydrophilic and hydrophobic particles and drops. Furthermore, a simple spring model based on energetic considerations, is presented to calculate these forces. This model describes the physics of the process much better than the simple capillary models used so far. Finally, a comparison between measured and modelled forces is given.

2. Materials and methods

An atomic force microscope (AFM) XE-100 from Park Systems is used throughout this investigation. An external data recording system, which extends the standard system of the AFM, records the force acting on the cantilever while it is being moved by the stepper motor instead of the piezo-system. This is necessary to reach moving distances far above $10 \mu\text{m}$, which is the largest operation distance of the piezo element of the z-scanner. The stepper motor was calibrated via time measurements while moving the z-stage a certain distance several times. At the reverse point of movement, a short idle time passed, till the cantilever moved in the opposite direction. The appropriate measured points were cut out from the force distance curve and afterwards the curves of trace and retrace were merged again. Tipless cantilevers (ACL-TL from AppNano) with a high spring constant of 65 N/m are being used. The spring constant of the cantilever has been determined via the method of Cleveland et al. [23]. Therefore the resonance frequency was measured before the colloidal probe had been added to the cantilever. Thus, the spring constant was calculated via the geometry of the cantilever. After gluing the colloidal probe on the cantilever, 16 force distance curves were measured on a smooth silicon surface. The slope of the resulting repulsive part of the force distance curve represents the spring constant of the cantilever. The piezo setup was adjusted till the slope fitted the calculated spring constant. The

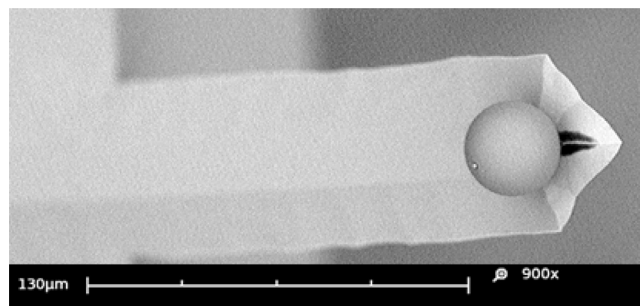


Fig. 1. SEM image of a used ACL-TL cantilever with a silica colloidal probe ($R = 15 \mu\text{m}$).

standard deviation of the slope is 1.5 N/m . It is assumed that the silica particle is not deformable. As a result, the slope represents the true spring constant of the cantilever. The fluid interface is generated in the corresponding liquid cell. The air–water interface is created with an aqueous drop, which is placed onto a hydrophobic surface. For all measurements, the volume of the drop is set constant. A spherical silica particle with a radius of $15 \mu\text{m}$ is used as the hydrophilic probe (Fig. 1). In order to vary the surface properties, i.e. wettability, a second spherical silica particle ($R = 20 \mu\text{m}$) was coated with Dynasylan® F8261 from Evonik (Essen, Germany). This coating has a teflon-like functional fluoroalkyl group as a hydrophobic part. For coating of the particles, a silanol solution was prepared, i.e. isopropanol (95.9 wt.%), distilled water (3 wt.%) and 37% HCl (0.1 wt.%) were mixed together and at least 1 wt.% Dynasilan F8261 was added to the solution. This mixture was stirred for 5 h before immersion of particles. The 40 wt.% of solid particles were added to the solution. After stirring the suspension for 24 h, the particles were sedimented through gravitational force and the supernatant was removed manually using a syringe. The residual particles were dried for 1 h at 60°C and furthermore for 1 h at 120°C to ensure a crosslinking of the silane molecules.

In order to vary the interfacial energy of the fluid, ethanol is added to the liquid phase from 1 to 15 wt.%. This directly affects the wetting behavior of the particle. The ethanol content and the resulting surface energy can be correlated to the measured interaction forces. The surface energy derives from a calculation using the surface tension of water and water–ethanol mixtures, which are taken from literature [24].

The measuring procedure is presented in Fig. 2 and will be discussed subsequently.

At first, the cantilever is moved to the interface with a constant velocity of $5 \mu\text{m/s}$. The distance between cantilever and interface is large enough that no forces are acting on the particle (position 1). At a certain proximity to the interface, the particle will suddenly come in contact with the interface (position 2), so the cantilever is deflected. An attractive capillary force can be measured acting nearly instantaneously on the particle due to the fast wetting process and the deformation of the interface. This phenomenon is called snap-in. The dynamics of the snap-in were investigated by Chen et al. [25] and McGuiggan et al. [26]. While the cantilever moves further with constant velocity the deformation of the interface is reduced (position 3) until the particle passes a position where the forces acting on the particle are zero (position 4). This equilibrium position is energetically favorable and depends on the surface energies γ_{sl} , γ_{sg} and γ_{lg} at the three-phase-contact-line (TPC). A common method to quantify the surface energies is the method of van Oss et al. [27] via drop shape analysis. A method to determine the dynamic hysteresis of contact angles on a single particle via AFM is described by Ecke et al. [10]. The advancing contact angle, which is needed for the force calculation, was calculated from the measured snap-in distance during attachment to a

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