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# **AUTOMATED CONTACT TIME APPARATUS AND MEASUREMENT PROCEDURE FOR BUBBLE-PARTICLE INTERACTION ANALYSIS**

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## **ABSTRACT**

The novel Automated Contact Time Apparatus (ACTA) presented in this paper serves as a diagnostic tool that allows the detection of changes in bubble-particle attachment probability and therefore floatability caused by alterations in the chemical environment and particle properties.

The apparatus consists of six identical capillaries where bubbles with defined size are produced simultaneously in a measurement chamber. The bubbles at the needle tips are placed in contact with the submerged particle bed for specific time periods, controlled with the help of automatic actuators. The advantage of the instrument is that hundreds of bubble-particle contacts can be measured automatically within a short time period. Microscopy pictures of each measured bubble are taken while recording the movement of the bubble before, during and after contact with the solid particles. The recorded pictures can be used to determine the actual bubble size and its corresponding deviation, and to detect the attachment of particles. The attached particles are collected in a detachable chamber for subsequent characterization. Furthermore, the device is portable and can be taken to the mineral processing plants for quick evaluation of particle-bubble attachment efficiency with particles and process water sampled directly from real processes.

**KEYWORDS:** Bubble-particle induction, flotation, statistical analysis, induction time

## 1 INTRODUCTION

The attachment between hydrophobic particles and gas bubbles is an important phenomenon in froth flotation, which is a widely-used separation technique applied not only in mining industry but also on e.g. in pulp and paper industry. The attachment efficiency, together with the collision and the stability efficiencies form the overall efficiency of the flotation process. To understand and optimize flotation processes in any system or field of industry, reliable data on bubble-particle interaction needs to be gathered on multiple different parameters. The concept of bubble induction time, introduced by Sven-Nilsson (Sven-Nilsson, 1934), has been observed to be a reliable indicator for flotation efficiency (Jowett, 1980). The most widely used attachment time measurement device was developed by Glembotsky (1953) and has been used by many authors (Gu et al., 2003; Ye et al., 1989; Yoon and Yordan, 1991a) to study different minerals and oil processing related systems.

Previously published works dealing with induction time measurements in various bubble-particle-gas systems revealed that the induction time is a sensitive function of numerous factors such as bubble size (Wang et al., 2005; Yoon and Luttrell, 1989), bubble movement (Gu et al., 2003), temperature (Yoon and Yordan, 1991b) in addition to the intrinsic physicochemical properties of the particles and the liquid. For this reason, a reliable standard for measuring bubble-particle induction time upon which flotation parameter optimization can be based is relevant.

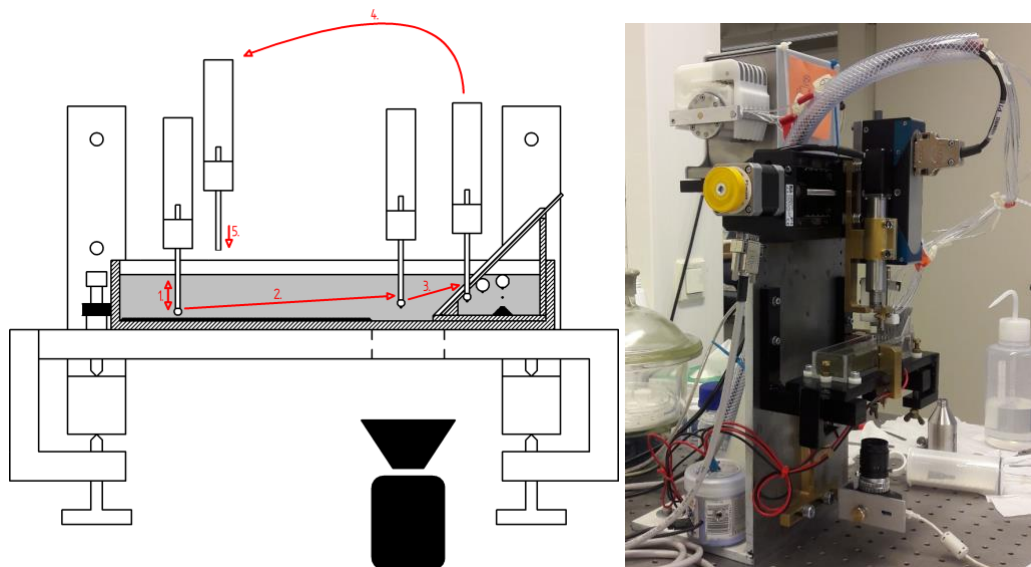
Particle-bubble attachment efficiency in flotation is influenced by many factors, water quality being one of the most important of them. In flotation research experiments are often performed in the laboratory, in artificially created, “synthetic plant water” systems (Biçak et al., 2012; Corin and Wiese, 2014; Manono et al., 2013). However, experiments done in laboratory conditions commonly do not reproduce all the effects occurring in process waters around real flotation plants. Also, experiments done in plant water taken from the plant into the laboratory do not correspond to the actual process water, as microbiological action and continuing, kinetically-controlled reactions of dissolved species alter the quality of these waters within time periods as short as a few hours (Levay et al., 2001; Levay and Schumann, 2006). Therefore, reliable flotation efficiency evaluation demands a tool that allow quick diagnostic testing of the effect of water quality on particle-bubble attachment efficiency at flotation plants with freshly taken process waters.

In this paper, we present a novel experimental setup named Automated Contact Time Apparatus (ACTA). The device has been developed in order to create an automated system, that allows the collection of large amount of data for statistical analysis within a relatively short time period. The instrument is transportable and can be taken to mineral processing plants to serve as diagnostic tool. Therefore, measuring the effect of different process water qualities on the particle-bubble attachment efficiency in freshly taken and altered process waters is feasible.

## 2 EXPERIMENTAL

### 2.1 Description and operation of the experimental setup

The operating principle of the ACTA in some respects resembles the one presented by Glembotsky (1953), however the mechanical setup of the instrument differs notably from the previous arrangements. Furthermore, multiple novel features has been developed improving the functionality as well as the diversity of the data produced. Figure 1 presents schematics the operation principle of ACTA, together with a picture of the actual device.



*Figure 1 - Experimental steps: 1- bubbles are formed at the tips of the needle array and pushed against the particle bed; 2 - bubble-particle aggregates are transported above the digital camera for imaging; 3 - the bubbles are taken to the sample collector and detached from the needle; 4 - the needles are lifted above the surface and moved horizontally then lowered into the liquid above an untouched spot on the particle bed for the next measurement cycle.*

The steps presented in figure 1 are performed automatically and the measurement cycles are repeated until the entire length of the particle bed has been sampled. Using, for example, a 1 mm step distance between the bubble-particle bed contact points, 65 measurement cycles or 390 measured bubbles are collected in a single measurement run.

The novel features of the instrument involve the array of six needles connected to a two-directional motor assembly, an automated plough for particle bed levelling, the particle collecting mechanism, fully programmable bubble movement during wave function and an automated image capturing and analysis system for the bubble size measurement and detection of particle-bubble attachments. The detailed operation of the instrument and the details about its mechanical construction are described below.

#### 2.1.1 Particle bed preparation

The first step in the experimental procedure is the preparation of the particle bed. After the solid particles are conditioned in the solution of interest, part of the slurry is filtered in order to produce a clear liquid where the experiments can be done without

compromising the image quality of the photos taken for bubble size evaluation. The particles are introduced into the cell with the help of a large pipette and the cell is filled up with the filtered experimental solution. In order to produce reliable measurement data, a flat and even particle bed is crucial. However, manual levelling and smoothing was found to be an extremely cumbersome and tedious task. Therefore, an automated plough attached to a MP-20 Micro Pusher (Physik Instrumente (PI) GmbH & Co. KG) with PI SMC pollux controller was designed and built. The reference position of the plough is set by the position of the tips of the needle array for improved accuracy. Once a completely flat particle bed is formed, the plough can move over the bed several times to improve packing of solid particles at the surface.

### ***2.1.2 Bubble formation and particle-bubble contacting***

After the particle bed is prepared, the actual measurement can be started. First, air bubbles with a set size are produced at the end of each needle in the six-needle array and the bubbles are then pressed against the particle bed (Figure 1, step 1). With this arrangement, notably more data can be collected in a single measurement cycle. An Ismatec™ Reglo Digital peristaltic pump has been applied to pump the air through Tygon® LMT-55 hoses (0.25 mm ID) into the needles to create the bubbles. The pumping time for the bubble formation is set as constant for a single measurement run. The warming of the hoses during pumping has been observed to have impact on the variation in the bubble size. For this reason, careful warm up and the lowest possible pumping time is used during the experimental run to create bubbles with equal size. The peristaltic pump is used in this setup because, in addition to bubble formation, the detachment of the bubbles from the end of the needles and the drying of the needles by purging air through them between the measurement cycles can be done with it.

A V-273 PIMag® Voice Coil Linear Actuator attached to C-413 PIMag® Motion Controller is applied for moving the needle array vertically. The maximum velocity of the V-273 PIMag® actuator moving the needle array vertically is 200 mm/s and the maximum acceleration of the needle array is 100 m/s<sup>2</sup> setting the physical limitations for the induction time measurement. The movement is fully programmable and any shape of wave function is possible within the given operational limitations characteristic of the actuator. The horizontal movement is produced by a PLS-85 Precision Linear Stage actuator and a PI SMC Pollux controller. All the actuators and control electronics are produced by Physik Instrumente (PI) GmbH & Co.

In addition to contact time ( $T_c$ ), the parameters controlled and recorded during the measurements with ACTA are the approach- ( $V_a$ ) and detachment velocity ( $V_d$ ) of the bubble to/from the particle bed, the approach- ( $D_a$ ) and the retreat distance ( $D_d$ ), and bubble diameter ( $D_D$ ) are (Figure 2).

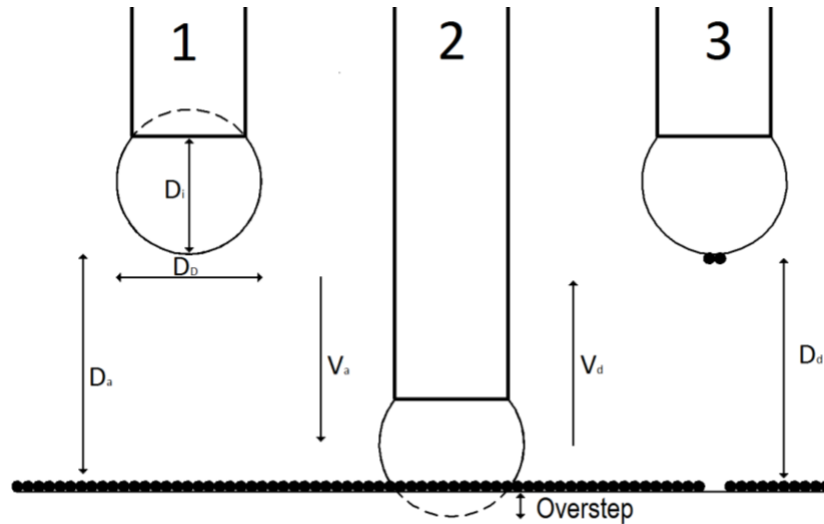


Figure 2 - The controllable and recorded parameters of the bubble approach, contact and detachment from the particle bed: Approach distance ( $D_a$ ), approach velocity ( $V_a$ ), Detachment velocity ( $V_d$ ), retreat distance ( $D_d$ ), bubble diameter ( $D_b$ ) and overstep.

### 2.1.3 Bubble imaging, bubble size measurement and particle detection

As the flotation efficiency is sensitive to the diameter of the bubble (Wang et al., 2005), the precise value for the of the bubble diameters needs to be measured. Furthermore, as represented schematically in Figure 2, the real approach distance ( $D_a$ ) depends on the fraction of the bubble that remains inside the needles and the distance of immersion into the particle bed (i.e., overstep) both of which are in turn functions of the bubble size. Consequently, the measurement of the diameter of each individual bubble is crucial for the reliability of the experiment.

For each cycle, the needle arrays with the bubble-particle aggregates are imaged. A digital camera is located under a transparent floor of the pool (Figure 1). Automated light emitting diode (LED) lights are used to increase the contrast for bubble diameter detection. To detect the possibly attached particles, optical fibers have been placed inside the needles for backlighting. Therefore, two pictures are taken during each measurement cycle: one with bright white side/backlight for bubble size measurement and a second one with backlighting with optical fibers from inside the needles (Figure 3).



Figure 3- Photos of bubbles taken from a single measurement cycle: a typical picture from the needle array taken for automated bubble size measurement (top), and a second picture from the same bubble array aided by optical fiber backlighting for automated particle detection (bottom)

The attached particles can thus be detected automatically and the pictures are saved for manual evaluation, if necessary. However, as the particles commonly get attached in clusters, the number of attached particles is, at the moment, not possible to evaluate from these pictures.

All pictures taken are saved in case manual examination is necessary. The collected pictures are used to determine the actual  $D_D$ ,  $D_a$ ,  $D_d$  and overstep separately for each bubble. The variation in the bubble size is, however, relatively small. For example, for 1.5 sec pumping and with 95% confidence limits, the bubble diameter was  $2.1098 \pm 0.0268$  mm calculated from 1295 measured bubbles.

It is worth noting that approach and detachment distances ( $D_a$  and  $D_d$ ) and overstep depends also on the vertical height of the bubble ( $D_i$ ), which is impossible to accurately predict without experimental investigation, as small bubbles have the tendency to remain partially inside the needles and large bubbles deviate from an ideal spherical shape. For this reason, a set of pictures was taken of bubbles with differing sizes from the bottom to determine the horizontal radius of the bubble and laterally to determine the vertical height ( $D_i$ ) of the bubble corresponding each horizontal diameter ( $D_D$ ). A linear least-square function of  $D_i$  as a function of  $D_D$  was generated from the obtained data to describe the vertical height of the bubble as function of the lateral diameter. This function was used to calculate the actual movement parameters of each bubble during the experimental runs.

#### ***2.1.4 Mass flow measurement and other particle property analyses***

In the original setup by Glembotsky (1953), the only results that is determined is whether attachment occurred or not at the set contact time, but the effect of particle properties (surface oxidation, surface coverage by reagent, particle shape, etc.) cannot be quantified directly, as the attached particles are not collected for further analysis. The experimental setup developed in this work allows the collection of particles in sufficient quantities for subsequent analysis, e.g., SEM or XRD. As presented in Figure 1, the particles attached to the bubbles are collected into a removable particle collector box. The particles trapped inside the collector box can be recovered for example, with the aid of a filter. In such case, by measuring the mass change of the filter paper, the total mass of all collected particles during the measurement run can be determined. The measured mass can be used as a supplement for the determination of attachment probability.

#### ***2.1.5 Contact time and bubble movement***

As every movement of the bubble during the wave function is crucial for the induction time (Gu et al., 2003), the vertical movement of the needle array is controlled by a proportional-integral-derivative (PID) controller (V-273 PIMag®) and the movement data of the inbuilt precise optical linear encoder is recorded. The wave function controlling the movement of the needles is used to define  $T_C$ , by programming the amount of time at which the needles are at their lowest position. Using the recorded data from the movement of the actuators, the values for the actual  $T_C$  for each bubble (herein defined as the time in which bubbles are in contact with the particle bed), overstep,  $V_a$  and  $V_d$  can be calculated. An example of the recorded movement data and the obtained parameters are presented in

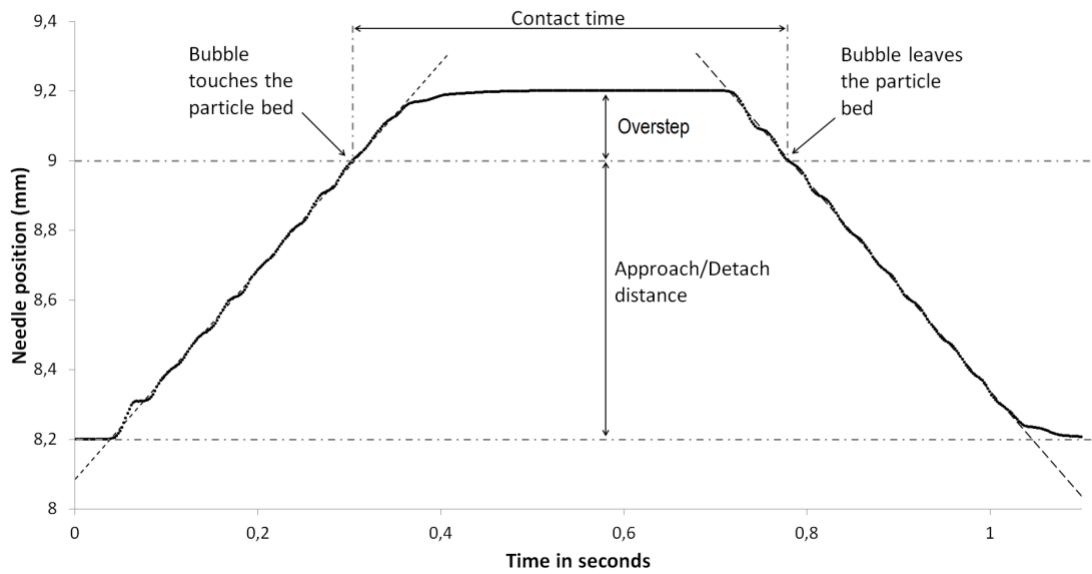


Figure .

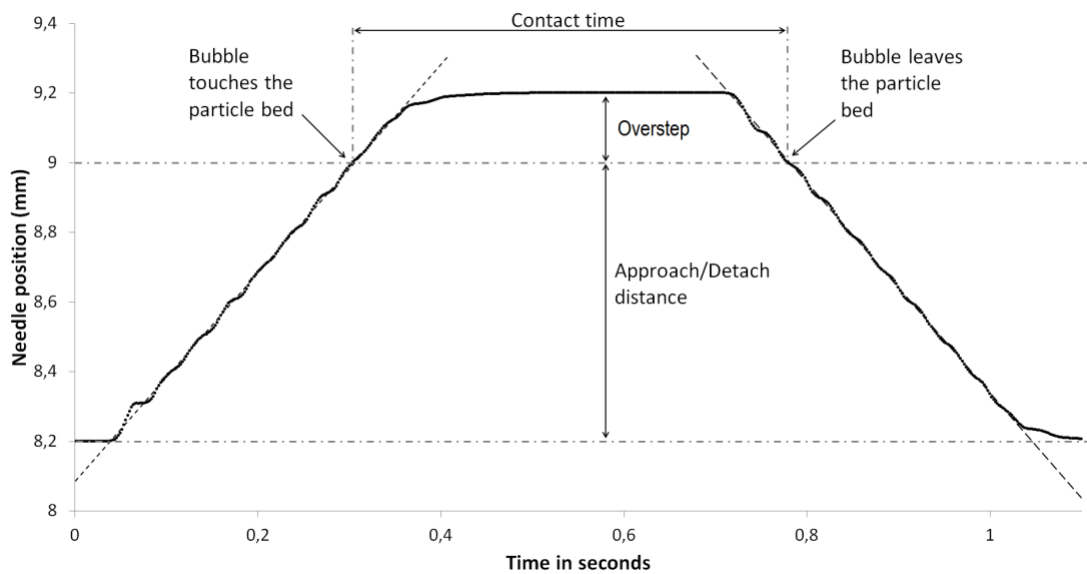


Figure 4 - An example of the recorded movement data of the needles during approach, contact and detachment from the particle bed

The standard deviation in the position of capillary array in between measurement cycles has been measured to be below  $5 \times 10^{-5}$  mm in all sections of the vertical movement. This means that the V-273 actuator consistently moves to near-exactly the same final position and therefore significant variation in contact time or bubble compression occurring due to the actuator movement is unlikely.



## **2.2 Materials and sample preparation**

In the testing phase of the instrument, three types of particles were used: 125-250  $\mu\text{m}$  quartz particles (Aldrich,  $\geq 99.995\%$ ), 106 - 125  $\mu\text{m}$  glass microspheres (Whitehouse Scientific Ltd) and 106 - 125  $\mu\text{m}$  chalcopyrite (from Durango, Mexico supplied by Ward's Science). The quartz particles were washed with purified water (MiliQ) before each experiment.

The glass microspheres were hydrophobised by silylation in trimethylchlorosilane vapour at  $\sim 70$  °C (Yakubov et al., 2000). A magnetic mixer was applied to mix the microspheres during the hydrophobization for even distribution of the trimethylchlorosilane on the surface of the microspheres. The hydrophobized particles were no more than 28 minutes submerged in purified water before the start of the experimental run to prevent degradation of the hydrophobic coating.

The chalcopyrite was first crushed with hammer, then pulverized in a ring mill and conditioned in 30 g/t potassium ethyl-xanthate (KEX) solution to render them hydrophobic.

Contact time experiments were carried out with pure, clean, hydrophilic quartz and mixture of 2/3 pure quartz and 1/3 hydrophobic glass microspheres, as well as KEX treated chalcopyrite particles.

The experiments hereby presented were carried out in purified water (MiliQ) with no additional control of pH.

## **3 RESULTS AND DISCUSSION**

### **3.1 Attachment probability of hydrophilic and hydrophobic particles alone and in mixture**

The general functionality of the developed instrument was tested by measuring the attachment probability of hydrophilic particles and a mixture of 2:1 hydrophilic and hydrophobic particles. Both populations were analyzed under identical experimental parameters. A total of 390 measured bubbles were collected in each experiment. The attachment probability data of the two experiments is visualized on Figure .

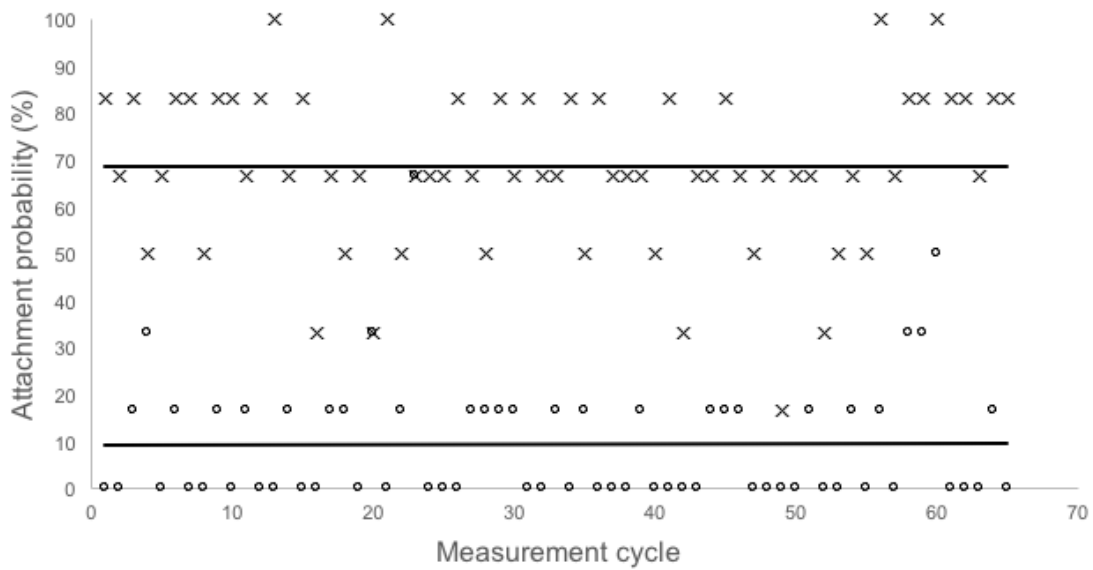


Figure 5 - The data obtained by measuring pure hydrophilic quartz (circle) and a mixture of 2/3 hydrophilic quartz and 1/3 hydrophobic quartz particles (cross).

The attachment probabilities presented in Figure 5 were calculated by dividing the amount of bubbles with attached particles by the total number of bubbles (i.e., 6 in a single measurement cycle). After the experimental run with a mixed particle bed, the attached particles were examined with an optical microscope. Since the jagged quartz particles and the spherical glass beads present distinctive shapes, it was possible to identify the presence of hydrophobic and hydrophilic particles in the sample holder. The collected particles showed that the clear majority of collected particles were hydrophobic glass beads, but a small fraction of hydrophilic quartz particles was also found. The presence of such hydrophilic particles in the sample collector reflects phenomena such as particle entrapment, a well-documented phenomenon in real flotation practice. This is a further advantage of the method hereby proposed as this type of effects would be overlooked in other methods, such as single particle-bubble attachment timers.

The impact of hydrophobic particles is clearly visible in the obtained data. The attachment probability in a particle bed of solely hydrophilic quartz particles is 9.5 % and the median amount of bubbles carrying particles in a single measurement run was zero. On the other hand, for the quartz mixture with hydrophobic beads, the corresponding values are 68.8 % and 4. This clearly shows the functionality and potential of the developed concept.

Secondly, the linear trend lines describing the attachment probabilities are almost horizontal indicating that the results can be perceived comparable regardless of the number of the measurement cycle. In the other words, the attachment probability does not vary significantly during the measurement run, at least within the time frame used in these examples. As seen in Figure , a linear fit of the data shows an insignificant slope in both particle samples, however such a small slope can be easily associated to the scattering of the data.

The third observation from the gathered attachment probability data is that the scattering of data seems to be constant throughout the experimental run in both cases. This, together with the observation that the attachment probability was approximately constant during the measurement implies a successful preparation of a flat and even particle bed and reflects the overall reliability of the experimental setup and method.

### **3.2 Attachment probability and collected mass as functions of contact time**

One of the novel elements of the instruments is the particle collector box that facilitates the collection of particles attached to the air bubbles for subsequent analysis. Because the attachment probability does not take into consideration the amount of particles attached to the air bubbles (i.e., attachment is considered equally successful whether one or more particles are attached), the attachment probability distribution is skewed.

Especially when measuring bubble-particle interaction with heterogeneous particle beds, the agglomeration tendency of submerged hydrophobic particles may result in the formation of clusters at the surface of the particle bed and eventual segregation of hydrophobic particles. This risk of this occurrence is even higher if the particle bed was not carefully prepared. If the measurements are performed neglecting the clustered surface, there are two possibilities: i) the particle-bubble attachments may not take place, as bubbles form an interface only with hydrophilic particles surrounding the clusters, or ii) when the bubble is in contact with a hydrophobic cluster, it picks up multiple particles but is still considered a single attachment event. For this reason, if only attachment probability as function of contact time is considered, the particle bed preparation may have a direct influence on the attachment probability results, as a bed containing hydrophobic clusters will result in a decreased number of bubbles with attached particles, but conversely an increased probability of bubbles with multiple attachments. In the end however, the number of total attached particles during the complete measurement run may not be significantly affected in case of clusters.

Therefore, in case of particle beds containing heterogeneous particles, a more repeatable and robust way to quantify the attachment formation performance is by evaluating the mass of the attached particles. This has been demonstrated in Figure 6, where the mass transfer data as well as attachments probability data are plotted together as function of contact time from two experiments with hydrophobic chalcopyrite, prepared as detailed in Section 2.2. Details of the experimental procedure can be found elsewhere (Bellers, 2017). The experiments labeled I and II in Figure 6 were prepared in different batches, but were made using the same ore. It should be kept in mind that these are natural ore samples and thus, they may have some slight differences in the content of impurities.

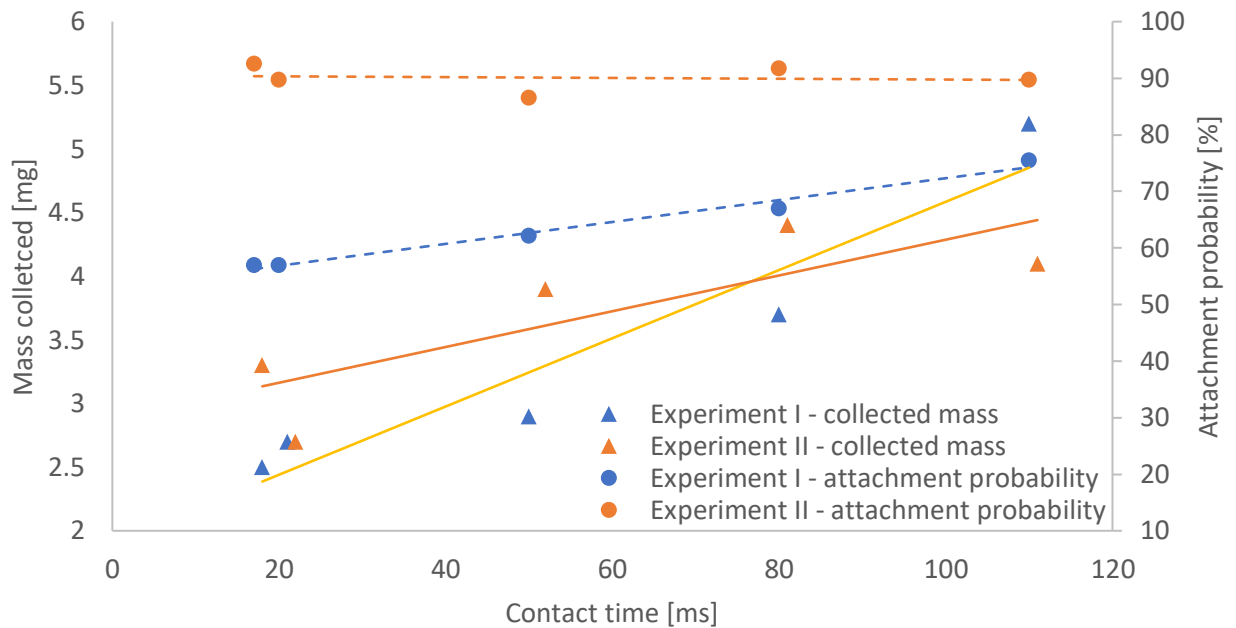


Figure 6 – Attachment probability and collected mass as a function of contact time in the tests conducted with chalcopyrite hydrophobised with 30 g/t KEX. The dashed linear trend lines present attachment probabilities as function of attachment time whereas solid trend lines present collected mass.

As seen in Figure 6, the variations on the surface of particle bed caused notable differences between the experiments in term of attachment probabilities. It is worth mentioning that the shortest  $T_C$  currently achievable with ACTA is ca. 20 ms. As the attachment probability of the hydrophobic quartz was over 50% at the shortest measurable  $T_C$ , these results do not allow the measurement of a classically defined attachment time. This is however a result of a high level of hydrophobicity in this specific system that is unlikely in real mineral flotation systems. As seen in Figure 6, the relative difference in the collected mass is clearly smaller compared with attachment probability. Therefore, small changes in particle bed with the same bulk sample material have a stronger impact on attachment probability than on collected mass. These findings further demonstrates the advantages in measuring mass transfer of collected particles, a unique feature of the device hereby introduced.

#### 4 CONCLUSIONS AND FUTURE WORK

A new method and device for bubble-particle attachment probability measurements has been introduced. The aim of the newly developed instrument is not only to improve reliability of the bubble induction measurements, but also to collect statistically significant, large data sets in a fast and reliable manner using fully automated measurement runs. The trials hereby presented demonstrate the functionality of the concept, since the attachment probability values obtained a high sensitivity of the developed method to differentiate between hydrophobic and hydrophilic particles. The compact size of the developed device allows it to be taken to the mineral processing plants for quick evaluation of particle-bubble

attachment efficiency with particles and process water sampled directly from real processes. The addition of the particle collector box opens the possibility for subsequent quantitative and qualitative analysis of the collected particles.

The presented instrument is a versatile tool that can be used in various ways. Some possible uses include the evaluation of particle-bubble attachment efficiency with particles and process water sampled directly from real processes in the field; measurement of induction time with differing gases; potential to examine induction time between liquid droplets and dry particles that opens the possibility for testing e.g. granulation; studies on entrapment of hydrophilic particles between hydrophobic ones. The removable sample collector box gathering the attached particles opens numerous opportunities for experimental research on the impact of different physical features of particles to the induction time.

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