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## Measuring the induction time for particle–bubble attachment in flotation

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### ABSTRACT

Froth flotation is an exceedingly complex physicochemical process. The convenience of distilling much of the complexity of the bubble–particle interactions into a single parameter has led to the continuing popularity of the classical ‘induction time’ to quantify the threshold for bubble–particle attachment to occur. Despite this popularity and the simplicity of the concept, there is no single universal method of evaluating the induction period.

In this paper, we begin with a critical review of the available techniques for estimating the induction period. These are: back-calculation from experimental (micro)flotation tests; pushing a particle toward a stationary bubble (or *vice versa*) using an atomic force microscope (AFM); pushing a bubble toward a stationary bed of particles in the ‘*Induction Timer*’; pushing a bubble toward a stationary solid surface using the ‘integrated thin film drainage apparatus’ (ITFDA); and dropping particles onto a submerged stationary bubble using the ‘*Milli-Timer*’ device. Each one of these methods has advantages and disadvantages, and the best choice depends on the application.

In the experimental section, we present quantitative comparison of the induction periods estimated using two different techniques, namely the *Induction Timer* and the *Milli-Timer*. The same particles were tested in each device, under the same conditions. It was found that by tuning the operation of the particle pick-up device, similar estimates of induction period could be obtained to the estimates made by direct observation with the *Milli-Timer*. In the former device a bubble is driven toward a particle bed at a controlled rate, whereas in the latter a particle’s motion is governed by the hydrodynamics. The potential to match these presents an intriguing prospect for better understanding the bubble–particle interaction, and the possibility to ‘calibrate’ the simpler *Induction Timer* against direct observations.



## INTRODUCTION – QUANTIFYING THE TENDENCY TO ATTACH

For froth flotation to be successful, a sequence of several sub-processes needs to occur. For one thing, the chemistry needs to be such that at least one of the particle classes will favour adherence to the air bubbles introduced into the cell. For another thing, the hydrodynamics should be set so that the particles and bubbles collide frequently and vigorously enough to have opportunity to attach, but not so aggressively that particles simply bounce off the bubbles or that particle–bubble aggregates are ripped asunder after attachment. And then there are the important sub-processes taking place in the froth layer. These sub-processes are systematically covered *inter alia* by Nguyen & Schulze (2004) for the pulp and (Ata, 2012) for the froth phase.

With an understanding of the complexity of the interactions, for the practical task of operating a flotation cell there is a benefit to distilling information from the underlying mechanisms into a single parameter. This is a function served by the **induction period** (or induction time),  $\tau$ . The induction period is a measure of the time required to form an attachment between a particle and a bubble (Sven-Nilsson, 1934; Nguyen & Schulze, 2004: 257f.), which may depend on *e.g.* the surface chemistry, particle shape, particle and bubble sizes, bubble and particle trajectories, and their relative velocities (Albjanic *et al.*, 2010; Verrelli, Koh & Nguyen, 2011; Verrelli *et al.*, 2014).

That induction period is compared against the time available to form an attachment. In the case of a head-on collision between particle and bubble (Nguyen & Schulze, 2004: 257f.), this would be the ‘**dwelling time**’, during which the two objects are in close proximity, with a narrow, liquid-filled gap between them. Considering the geometries involved, and the swirling nature of flow within the pulp of a real flotation cell, glancing encounters between particles and bubbles are likely to be more common; in this case the particle is seen to ‘slide’ over the bubble’s surface before either attaching or withdrawing (Verrelli, Koh & Nguyen, 2011). Here the period available for attachment is called the ‘**sliding time**’. For simplicity we will refer to both dwell and sliding times herein as dwell times.

The basic concept is that  $\tau$  is intrinsic to a given class of particles, while the dwell time is characteristic of a specific operating condition of a flotation cell (or a region within the cell) – or the operation of a given laboratory device. It turns out that the reality is somewhat more complicated than this.

The induction period can be important in determining the eventual flotation grade and recovery in industrial operations. It has also been demonstrated through computational modelling that the ultimate grade and recovery is likely to become most sensitive to induction time for cases involving ‘borderline’ materials, *i.e.* particles that are difficult (not impossible) to float (Koh & Verrelli, 2014).

Several alternative experimental means have been used to estimate  $\tau$ . Each one has its own unique advantages and disadvantages, which we critically review and summarise in the following. What is also missing in the literature is a quantitative comparison of estimates across different devices. Here for the first time we present measurements on the same sample with the *Induction Timer* and the *Milli-Timer*.



## SURVEY OF EXPERIMENTAL TECHNIQUES

As mentioned above, there are several experimental techniques available to estimate  $\tau$ . Before discussing the individual techniques, let us consider the 'ideal' measurement. In this ideal measurement:

- the time period would be unambiguously defined, and directly measured;
- the test procedure would be consistently implemented, it would be fast, and not require expensive instruments or highly-specialised skills to operate;
- the test conditions would replicate the dominant (controlling) sub-mechanisms from a flotation cell, and the results would be relevant to industrial operation

While this perfect technique does not exist, the available techniques are still useful. There are five main options.

### Back-calculation from batch flotation tests

As the induction period relates exclusively to particle–bubble attachment, it has been estimated from microflotation tests which eliminate effects from the froth layer and pulp entrainment. These involve passing a slow stream of individual bubbles through a dilute, fluidised bed of particles, as in a Hallimond tube (Hallimond, 1944) — or, more likely, a modified version thereof (Kitchener, 1984). The collection efficiency is then measured.

To estimate  $\tau$  from the collection efficiency requires some approximation of the governing relation. One formula that has been prominently reported in the literature is the so-called Generalised Sutherland Equation (GSE) (Dai *et al.*, 1998). The proponents of this method report “good agreement” between the theory and empirical results for the dependence of collision efficiency upon particle size (Dai *et al.*, 1998).

### Atomic force microscopy

The atomic force microscope (AFM) can be used to drive particles of several micrometres in size (or smaller) against a submerged stationary bubble (Butt, 1994; Ducker, Xu & Israelachvili, 1994). Alternatively, a bubble could be driven towards a solid surface — usually flat (Manor *et al.*, 2008). The output of an AFM consists of a transient force signal. Ordinarily this output would be mapped against the gap between particle and bubble, but the ability of a bubble to deform means that the gap cannot be directly estimated with accuracy\*. The motion of the driven object is constrained to be axial — no lateral motion or rotation is allowed — and the interactions are almost invariably configured to be head-on (*i.e.* axisymmetric).

The induction period is then defined as the interval from when hydrodynamic forces due to proximity of the particle and bubble's surfaces become significant or perceptible, until the moment of attachment when a portion of the particle is rapidly engulfed, resulting in the so-called 'jump-into-contact' (Snyder, Aston & Berg, 1997).

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\* For some simple configurations — *e.g.*, head-on collisions of a bubble and a flat plate or perfect sphere — numerical implementation of the Stokes–Reynolds–Young–Laplace (SRYL) model can simulate the process quite well (Wang *et al.*, 2013), providing a 'corrected' estimate of the gap as a function of time.



## Induction Timer device

The *Induction Timer* (IT) has been developed based on the pioneering experiments of Cooke & Digre (1949), who measured attachments from a bed of particles resting at the bottom of a liquid-filled tank as a bubble was pushed down against the bed. Subsequently the concept was refined (Ye, Khandrika & Miller, 1989; Yoon & Jordan, 1991) and eventually commercialised with software and hardware to control the motion of the bubble and to aid in visualising the outcome of the interaction. The induction period is then defined as the dwell time for which 50 % of the interactions<sup>†</sup> result in attachment (Ye, Khandrika & Miller, 1989).

## Integrated thin film drainage apparatus

The ‘integrated thin film drainage apparatus’ (ITFDA) is like a hybrid of the IT and the AFM. The equipment controlling the bubble’s motion remains the same as the IT. The sedimented particle bed of the IT is replaced by a single solid ‘particle’ attached to a force sensor (Wang, 2013; Wang *et al.*, 2013). The “film drainage time”, or  $\tau$ , is measured as the time elapsed from detection of a minimal repulsion force until the moment of attachment, much like with an AFM.

## Milli-Timer apparatus

The *Milli-Timer* (MT) device involves the settling of a dilute swarm of particles onto a stationary, submerged bubble (Verrelli, Koh & Nguyen, 2011), which can both be of industrially realistic size. A feature of this technique is that the particles are able to move laterally or rotate. Hence, the particles tend to ‘slide’ over the bubble’s surface before either attaching or withdrawing. The moment that sliding of a given particle begins is determined by judging a discontinuity in the particle’s trajectory. By use of high-speed video recording and zoom lenses, it is possible to directly observe the moment of attachment as a sudden motion, or ‘jump in’, of the particle (Verrelli & Koh, 2010; Verrelli, Koh & Nguyen, 2011). These two times then directly yield  $\tau$ .

A limitation of the MT is that in the current design there is little scope to check whether more energetic encounters could promote attachment<sup>‡</sup>. We see an opportunity to enhance the MT by configuring the liquid in the cell to flow.

## Comparison

A comparison of the alternative techniques for estimating  $\tau$  is presented in **Table 1**. The criteria are based on the characteristics of an ideal measurement (*vide supra*). It is seen that technique has certain advantages and disadvantages, and the best choice depends upon the whether it is intended to undertake a fundamental measurement of absolute values, or whether a quick comparison is desired.

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<sup>†</sup> Sometimes 100 % is used as the criterion (Ozdemir *et al.*, 2009; Albjanic *et al.*, 2011).

<sup>‡</sup> Besides incidental, minor fluctuations in approach velocity due to particle swarm effects (the effects of slightly different particle shapes and sizes appears to be negligible in specimens studied to date) (Verrelli *et al.*, 2014).



**Table 1** Comparison of the alternative experimental techniques for estimating induction periods

Characteristic	Microflotation	AFM	Induction Timer	ITFDA	Milli-Timer
How is $\tau$ estimated?	Uses approximate model (e.g. GSE)	Inferred from transient force data	Inferred from nominal, user-specified dwell times	Inferred from transient force data	Directly observed sliding and attachment
Is $\tau$ unambiguously defined?	Unambiguous for a given model	Yes, although start of period requires judgement	Choice of 50 % or 100 % threshold yields different estimates	Yes, although start of period requires judgement	Yes, although start of period requires judgement
Consistently implemented?	Model should account for typical differences in implementation e.g. changes in bubble rise rate	$\tau$ will vary greatly depending on approach rate	$\tau$ will vary greatly depending on approach rate, nominal dwell time, etc. Statistical analysis inconsistent.	$\tau$ will vary greatly depending on approach rate	Consistently implemented while particles are introduced by free settling
Speed of test	~Hour	~Several hours	A few hours	~A few hours	Several hours
Speed of analysis <sup>a</sup>	A few hours	A few hours	A few hours	A few hours	Days
Equipment cost	Cheap	Expensive	Moderate	Moderate	Moderate
Operator/Analyst skill	Standard	Highly specialised	Standard	More specialised	More specialised
Type of motion	Natural motion of particles & bubbles, at low speeds	Highly-constrained motion, at arbitrary speeds	Somewhat constrained motion, at arbitrary speeds	Highly-constrained motion, at arbitrary speeds	Natural motion of particles onto stationary bubble in quiescent liquid
Range of motion	> 1 cm	< 0.02 mm	< 1 mm	< 1 mm	> 1 cm
Range of $\tau$	<1 ms to >10 ms	<0.1 ms to ~100 ms	~10 ms to ~5 s	~10 ms to ~5 s	~1 ms to ~100 ms
Multi-body effects?	Yes	No	Yes	No	Slight
Typical particle and bubble sizes	Industrially relevant	Micron or sub-micron scales	Industrially relevant	Millimetre scale	Industrially relevant
Good to estimate	Efficiency (and $\tau$ is inferred)	Close-range forces	Relative ease of attachment	Long-range forces	Induction period; particle trajectory
Suited for	Site and applied research laboratory	Fundamental science research laboratory	Site and applied research laboratory	Fundamental engineering research laboratory	Fundamental engineering research laboratory

<sup>a</sup> Assuming all models have already been set up.

## EXPERIMENTAL METHOD

Two of the techniques described above were employed to characterise  $\tau$  for the same particle samples: the *Induction Timer* (IT) and the *Milli-Timer* (MT).



Two particle samples were analysed, each consisting of cleaned borosilicate glass spheres (Mo-Sci Specialty Products, Rolla, USA) in narrow size fractions, rendered hydrophobic by methylating the surface to a specified degree of saturation according to the method detailed step-by-step by Verrelli *et al.* (2014). The two samples are distinguished as:

- Sample 1 = 90–106  $\mu\text{m}$ , 25 % methylated
- Sample 2 = 75–90  $\mu\text{m}$ , 50 % methylated

The percentage methylation can loosely be understood as the percentage of the particle surface that is covered (or shielded) by the methylating species, which in this case is the hydrocarbon branch  $-\text{Si}(\text{CH}_3)_3$  (Verrelli, 2008: 925ff.; Koh *et al.*, 2009). The higher the degree of methylation, the greater the hydrophobicity. Previous particle measurements indicated contact angles of  $\theta_a \sim 55^\circ$  at 50 % methylation, compared to  $\theta_a \sim 35^\circ$  at 25 % methylation (Koh *et al.*, 2009).

The particles were immersed in water purified by reverse osmosis and deionisation, allowing ~60 minutes for ‘equilibration’. Fresh bubbles were blown several seconds before each measurement from ambient air.

For the *Milli-Timer*, the method proceeded as described in the previous section, and also reported in (Verrelli, Koh & Nguyen, 2011). The bubbles in this case were 1.53 to 1.60 mm in diameter. From past experience we knew that the particles would approach the bubble with initial velocities of order 20 mm/s, but that the radial velocity would decrease to below one tenth of this near the bubble’s surface (Verrelli, Koh & Nguyen, 2011).

For the *Induction Timer*, the method proceeded as described in the previous section, and also reported in (Albijanic, Bradshaw & Nguyen, 2012). The bubble in this case was 1.5 mm in diameter. In general, the parameters controlling the bubble’s motion can be freely chosen. Given the prior results from the MT, through an iterative process, herein we chose parameters to give approximate agreement in induction periods between the two techniques. A consideration was to constrain approach velocities to those seen in the MT. The bubble retraction speed was consistently maintained at 3 mm/s. To attain the desired resolution in time, for a given sample the bubble was pressed down 20 times for each set of parameters, including dwell time. As a refinement over the usual analyses (*e.g.* Ozdemir *et al.*, 2009), or even more careful analyses (Burdukova *et al.*, 2010), the error bars on the attachment probability for each dwell time are 95 % confidence intervals that were rigorously evaluated from the formula for Wilson’s ‘score interval’ (Agresti & Coull, 1998; Brown, Cai & DasGupta, 2001a, b). We use a conventional definition of attachment time equal to the dwell time corresponding to 50 % efficiency of attachment.

## EXPERIMENTAL RESULTS AND DISCUSSION

The results from the MT are presented in **Figure 1**. As reported previously, values of  $\tau$  were found to depend upon the horizontal offset of a particle’s initial position from the vertical axis through the centre of the stationary bubble. (Initial velocities were 15 to 19 mm/s for the smaller particles, and 23 to 36 mm/s for the larger particles.) This variation makes it more difficult to define a single representative value of  $\tau$ . For efficiency calculations the procedure is to integrate over the projected area of the bubble (plus a small increment to account for the particle radius) (*cf.* Yoon & Luttrell, 1989). That formula cannot be applied here, because grazing trajectories will have infinite values of  $\tau$ . Various alternatives exist, such as

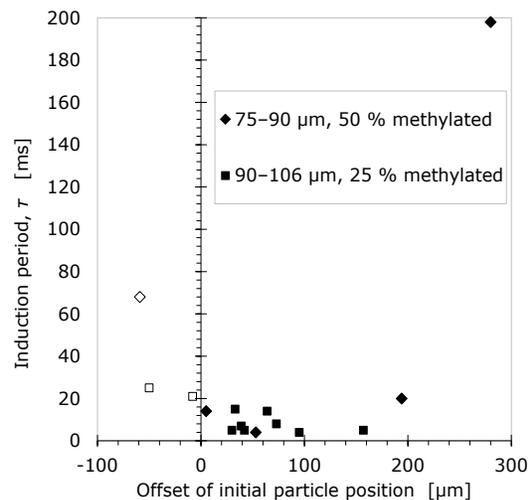


averaging  $1/\tau$ , but the optimal approach depends on the mathematical model in which it is to be used. For the present illustrative purposes, and noting the large stochastic variations, it is sufficient to take 20 ms as a representative value of  $\tau$  for both specimens. (The lower methylation of the second specimen may have been counterbalanced by its larger diameter and hence approach velocity.)

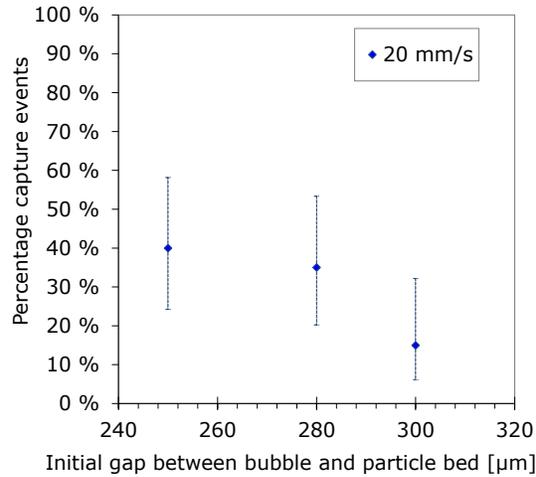
For the IT the first step was to choose an initial particle–bubble gap. As per **Figure 2**, at a representative  $v_a$  of 20 mm/s the target  $\tau$  was approximately indicated for an initial gap of 250  $\mu\text{m}$  with Sample 1, so this was set for the subsequent tests. With Sample 1 three  $v_a$  values were tested, namely 2, 20, and 50 mm/s, as shown in **Figure 3**. Clearly the slower approaches suggest larger  $\tau$ , consistent with Gu *et al.* (2003). To match our nominal target of  $\tau = 20$  ms, the closest match would be for  $v_a = 50$  mm/s. This is a somewhat surprising result, as it corresponds more closely to the *initial* velocity of particles in the MT (far from the bubble), rather than the velocity shortly before attachment. Another interpretation might be that more energy (force) is required to press the bubble against a particle bed, as the hydrodynamic drainage resistance is much larger than when only a single particle is involved. In any case, the result suggests the possibility to **calibrate** the IT, using ‘absolute’ results from another source, such as data from the MT.

Unfortunately for Sample 2 we were unable to find a value of  $v_a$  that would suggest  $\tau$  to be larger than 10 ms, as in **Figure 4**. (Shorter dwell times cannot be reliably implemented on the IT.) To get the desired magnitude of  $\tau$  here would require either much slower  $v_a$ , or else a larger initial gap. The reason for this is so far unclear.

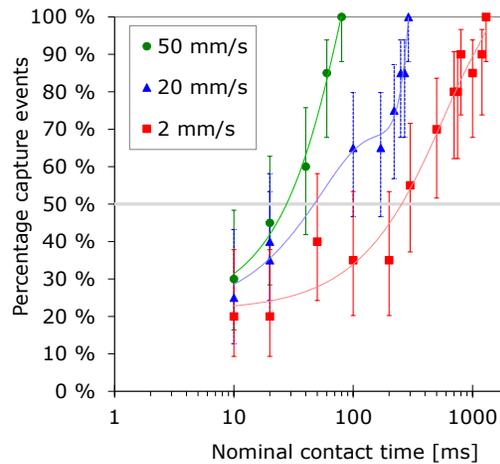
Finally, the error bars in **Figure 2**, **Figure 3** and **Figure 4** are seen to be substantial, even though each individual point used data from 20 pick-up trials. This might surprise some readers, and reinforces the need to conduct a large enough number of measurements and to employ the proper statistical analyses.



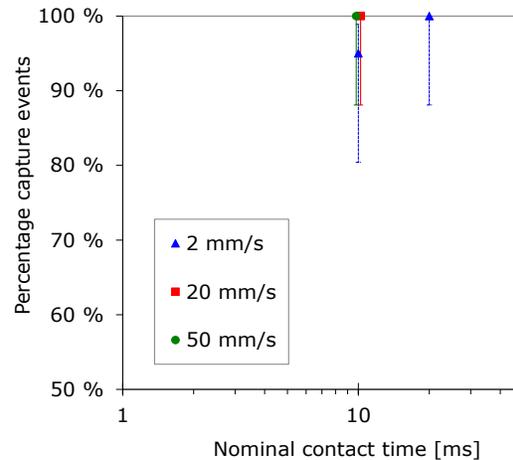
**Figure 1** Measured values of  $\tau$  from the MT, as a function of the particle’s initial position. Hollow points represent particles that crossed over the axis due to upstream interaction with other particles.



**Figure 2** Measured attachment probabilities from the *Induction Timer* for different initial gaps between the bubble and the particle bed, with nominal  $v_a = 20$  mm/s and a dwell time of 20 ms, for Sample 1



**Figure 3** Attachment probabilities for different dwell times in the IT, with different nominal bubble approach velocities, for Sample 1 (Initial gap = 250  $\mu\text{m}$ .) Curves have been added to guide the eye



**Figure 4** Attachment probabilities for different dwell times in the IT, with different nominal bubble approach velocities, for Sample 2 (Initial gap = 250  $\mu\text{m}$ .) Points at 10 ms are offset for clarity

As these results demonstrate, it is not necessarily meaningful to compare estimates of induction period from one technique with those from a totally different technique, unless some prior calibration (or correlation) has been undertaken. Certain 'trends' might be expected to be observed across all techniques, even in the absence of any specific calibration: for example, more hydrophobic particles yielding shorter induction periods. However, other factors such as particle size also play a role. Even if all other particle characteristics are held constant, the expected trends may still not be observed if the measurements are made at the limits of the instrument capabilities (in a given configuration): for example, the techniques discussed will not be able to accurately resolve sub-millisecond induction periods.

## CONCLUSIONS

After reviewing the alternative techniques for estimating  $\tau$ , each was found to have its own pros and cons. Several of the techniques are strongly dependent upon the experimental parameters, most notably the speed of approach. However, the method presented here may provide a way of calibrating a device. The calibration will not be needed if it is only desired to make a relative comparison ( $\tau_1 > \tau_2$ ). However, the calibration will be appropriate whenever it is desired either to make a quantitative comparison (e.g.  $\tau_1 = 7.5 \tau_2$ ) or to enter absolute values in a numerical simulation (e.g.  $\tau_1 = 90 \text{ ms}$ ,  $\tau_2 = 12 \text{ ms}$ ).

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