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MICRO-STUDIES OF MINERAL PROCESSING FUNDAMENTALS

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Since many mineral processes rely on the manipulation of interfacial properties, such as hydrophobicity, surface tension and wettability, there is a great deal of interest in being able to relate the micro-scale properties of the solid/fluid species to the macro-scale behaviour of the processes themselves. In order to develop this understanding, it is necessary to be able to directly measure and observe such properties, a task which can be non-trivial due to the conditions that can be encountered on the plant scale. Over several years, a range of unique devices, collectively known as Micro-Force Balances (MFBs), have been designed and constructed in the author's laboratory, which can measure and observe micro-scale interactions between solid particles in both gaseous and liquid media, at ambient and high temperatures. Mineral processing applications such as granulation, flotation and spherical agglomeration have been the subjects of studies to date. This paper presents an overview of the MFB designs and the significant findings in relation to these applications.

Key words: micro-manipulation, flotation, granulation, spherical agglomeration, separation

INTRODUCTION

Mineral processing general involves the separation and/or collection of solid particles of different species, either suspended in liquids or agitated in gaseous media. The design and operation of such multi-tonne processes have changed little over the last few decades. Dwindling mineral resources and high energy costs have focussed attention on improvements in process efficiency and the recovery of much finer mineral grades. There is a growing realisation that significant advances can be made in process design and operation by developing understanding, on a fundamental basis, of the interactions between the solid particles and the surrounding medium, be it gas or liquid.

The fundamental understanding of particulate and surface interactions requires sensitive instrumentation to provide direct measurements and observations. Atomic

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Force Microscopy can be used to measure surface properties arising from molecular-scale interactions, such as electrostatic forces and surface asperities. However it can prove difficult to use such devices to monitor directly the behaviour between particulate species in suspension, particularly at high temperatures. It is for this reason that a novel device, termed a Micro-Force Balance, was developed at UCL in order to measure and observe the interaction between particles held together by liquid binder bridges. These studies were in relation to granulation and spherical agglomeration processes (Simons and Fairbrother 2000; Pepin et al 2000a,b; Pepin et al. 2001; Rossetti and Simons 2002). Subsequently, three further devices have been constructed, using state-of-the-art technology and with higher force-displacement resolution than the prototype version, to study particulate interactions in flotation froths (Spyridopoulos and Simons 2002), high temperature fluidisation (Pagliai and Simons 2002) and crystallisation (Pratola et al. 2001). In this paper, the experimental set-ups and main findings of the granulation, flotation and spherical agglomeration work will be discussed.

GRANULATION STUDIES

Despite the wide spread use of granulation in minerals processing, pharmaceuticals, detergents, fertilisers and foodstuffs, the fundamental mechanisms governing granule growth and subsequent strength are not fully understood. Hence, mineral processors have taken an empirical approach to the operation of granulators, whilst pharmaceutical companies have taken a product formulation approach. Over the last ten years this has started to change and currently there is a large amount of research being carried out on granulation which can be considered to be divided into two areas; (i) relating particle and formulation properties such as binder viscosity and interfacial energy, particle size distributions, friction and plasticity, to the bulk agglomeration behaviour, and (ii) the incorporation of these relationships into process scale simulations. The work reported here investigated microlevel phenomena in relation to particle and binder properties with the overall aim of improving the understanding and control of macroscopic granulation processes.

MEASURING LIQUID BRIDGE PROPERTIES

The objective of the work was to develop the fundamental understanding of the role of the liquid and solid properties in the growth, consolidation and strength of granules, from the initial contact between the liquid and particles to the resultant multi-particle bodies. The strength of liquid bridges was measured using a novel Micro-Force Balance (MFB) described in detail elsewhere (Simons and Fairbrother 2000). In brief, a pair of micro-pipettes holds the particles in position, of which one is highly flexible (see Fig.1). Once a drop of binding liquid has been administered between them (using a third pipette), the movement of the pipette base as the particles

are pulled apart is monitored by sensitive position transducers, providing a direct measure of the applied force. A unique optical follower with a resolution of order 1nm monitors the much smaller movement of the pipette tip. The pipette tips can be submerged in an optically clear dish so that the surface effects in liquid media can be investigated (see below).

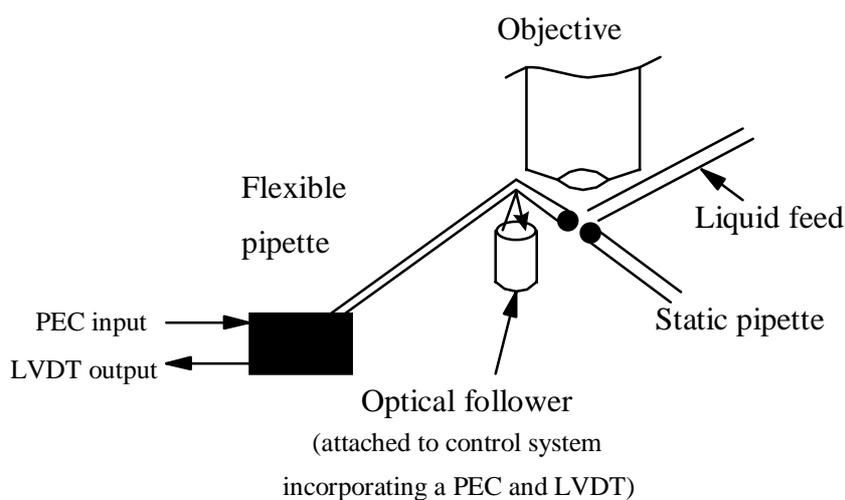


Fig. 1. Schematic of the micro-manipulation stage

The entire apparatus is PC controlled and, coupled with image analysis software, leads to accurate determination of liquid bridge dimensions and forces. For dynamic measurements, events separated in time by 0.1ms can be distinguished. In addition, the particles can be brought together, then separated, under (computer) controlled rates, by varying the voltage sent to the piezoelectric crystals in the position transducers. In this way, the dynamic nature of agglomeration processes can be studied.

In the work reported here, images of liquid bridges were sampled from a video recording of the rupture sequence. From the images, interparticle forces, separation velocity and interparticle distances were measured. When applicable, liquid volumes and liquid surfaces were also calculated from the 2D images using the parabolic approximations of the liquid bridge profiles (Pepin et al. 2000a,b). Such calculations are only valid for spherical particles.

GRANULE STRENGTH

Pellets of 18 mm diameter and 17 mm length were made from various spherical and non-spherical powders and moistened with silicon oils of increasing viscosity. The powders were: Sugar beads, Suglets 30-35 (SB1) and Suglets 250-355 (SB2), used as

a comparison to granules made of glass ballotini (GB) reported in (Iveson et al. 2001), a lactose DCL11 (L1), separated in three fractions (L1A), (L1B), (L1C), with 90 and 180- μm -mesh sieves, a lactose EFK sieved into coarse (L2) and fine (L3) fractions with a 100- μm -mesh sieve and a lactose 150 mesh (L4) (Pepin et al. 2001). Finally, a crystalline drug powder (DP) and Magnesium stearate NF-VG-1-726 (MGST), which exhibits low interparticle friction were also used. The liquid binders were: Silicone oils of increasing viscosity 9, 96, 971, 996 and 97920 mPa.s, water, a 0.2% w/w sodium dodecyl sulphate aqueous solution and aqueous solutions of hydroxypropylmethylcellulose and polyvinylpyrrolidone of increasing viscosity and varying surface tension.

The pellets were carefully retrieved from the die on a pre-tared microscope slide, weighed and then deformed radial to the cylinder axis between the two flat punches of an TAXt2® texture analyser (Stable Micro Systems). The upper punch was lowered onto the pellets at speeds of between 0.1 mm/s and 10 mm/s. From the weight of the pellet and a knowledge of its composition, the pellet solid and liquid volume fractions and porosity could be calculated. The slope of the force versus contact area curve was taken as the mass hardness.

RELATING LIQUID BRIDGE PROPERTIES TO GRANULE STRENGTH

The first step was to provide a more rigorous understanding of the effect of the liquid and solid properties on the shape of the liquid bridges and, hence, the strength. Liquid bridges were created on glass spheres and lactose using the silicone oils.

As is reported in (Pepin et al. 2000a), the investigations have shown that the rupture of liquid bridges occurs spontaneously at a certain interparticle distance and that two cases exist: One where the volume of liquid is fixed, that is when the three-phase contact line is observable on the particle surfaces during elongation, the second where there is no observable three-phase contact line and liquid covers the entire particle surfaces and the surrounding objects, creating liquid reservoirs outside the particles. The second case is observed when the bridging liquid perfectly wets the solid particles (advancing contact angle, $\theta_a =$ receding contact angle, $\theta_r = 0$). In this case, depending on the bridge elongation speed and viscosity of liquid binder, some liquid can move from the liquid reservoirs to the inter-particle gap and the liquid volume of the bridge can increase. In the former case, there is generally a noticeable contact angle hysteresis and the shape the bridge adopts depends on the wetting behaviour and the value of the three-phase contact angles (Pepin et al. 2000b). In Figure 2, two examples are shown. In Fig. 2(a), the liquid does not de-wet the particle surfaces during bridge elongation, the three-phase contact line is fixed. The shape then adjusts as the contact angles change with separation (in order for the constant volume condition to be met and $\theta_a > \theta > \theta_r$). In Fig. 2(b), the liquid de-wets the silanised particle and the contact angle on this particle remains relatively constant during separation ($\theta = \theta_a = \theta_r$) whereas the contact angle on the unsilanised particle, which

has a fixed three-phase contact line, does change ($\theta_a > \theta > \theta_r$). In the case where there is no observable three-phase contact line, the liquid perfectly wets the solid and the bridge shape remains torroidal throughout separation.

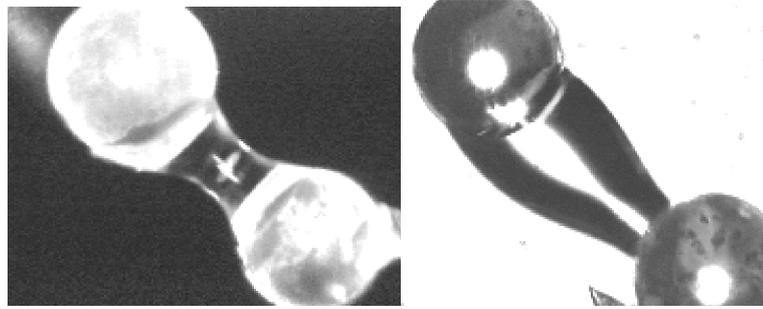


Fig. 2. Glycerol liquid bridges showing three-phase contact lines with glass spheres; (a) two non-silanised spheres (left, 126 μ m; right, 111 μ m), (b) left, unsilanised (118 μ m), right, silanised (125 μ m)

A parabolic model has been developed that predicts well the evolution of bridge shape with separation distance (Pepin et al. 2000a,b). The separation distance where rupture occurs can then be predicted by assuming that this occurs through the bridge's thinnest neck, y_{min} . This gives rise to an empirical expression for the bridge volume and the reduced hydrostatic pressure in the bridge caused by the bridge curvature. A three-term equation can then be derived for liquid bridge forces from contact through to rupture. With the assumption of glass spheres hard enough to sustain the surface tension and pressure forces of the liquid bridge, the normal force can be expressed as:

$$F_N = 2\pi y_{min} \gamma_{LV} + \Delta P_{neck} \pi y_{min}^2 + \frac{3\pi \eta v_i \bar{R}^2}{2H} \quad (1)$$

The first term is the capillary contribution, the second is that due to the reduced hydrostatic pressure, whilst the third is due to interparticle friction. H is the separation distance between the particles of average radius \bar{R} , v_i , the particle separation velocity, and η , the dynamic viscosity of the bridge liquid. This expression was shown to provide accurate predictions of the force when compared to those measured directly by the MFB, for both quasi-static and dynamic separations. The viscosity term becomes more dominant as the separation speed increases.

From the individual bridge strength model, an expression was derived, relating the hardness of granules to their porosity and liquid content (Simons et al. 2003):

$$\Omega_{calc} = \frac{1}{2} \left[\left(3\pi \bar{c} \eta v_i \times \frac{\epsilon}{\phi_L} \right) \times \left(\frac{1}{v} \right)^{2/3} + \left(2\pi y_{min} \gamma_{LV} + \gamma_{LV} (\cos\theta + 1) \times \frac{A_{ab}}{H} \right) \times \left(\frac{c}{v} \right)^{2/3} \right] \quad (2)$$

where \bar{v} is the average particle volume, A_{AB} is the average solid-solid contact area between two solid particles in the wet agglomerate, ϕ_L is the liquid volume fraction, c is the co-ordination number (for spheres) and ϵ is the agglomerate voidage. The calculated hardness was plotted against the hardness measured for granules made up of the range of solid particles and liquids indicated above. From figure 3, it can be seen that agglomerates made from L1, L2, L3, L4, SB1, SB2, and DP exhibit measured hardness which can be predicted from the above equation. The hardness of agglomerates made from glass beads (Iveson et al. 2001) are also well predicted below 10^5 N.m^{-2} . Above this limit, which corresponds to capillary numbers (a measure of the strain rate suffered by the agglomerate) which exceed 7×10^{-2} , the mass hardness is over estimated. Further work is required to address this anomaly, probably caused by the fact that dynamic forces are transmitted between layers throughout the agglomerate and, hence, liquid bridges cannot be considered in isolation.

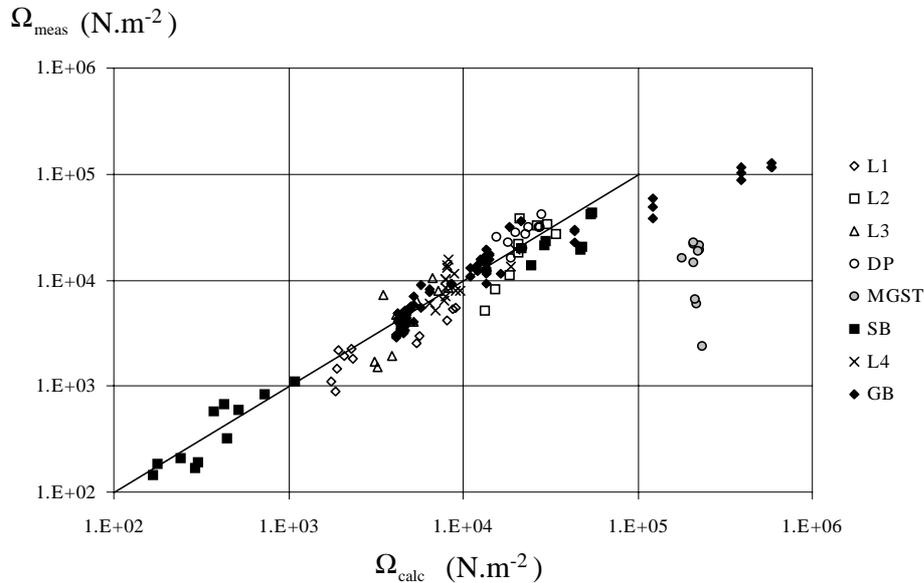


Fig.3. Measured versus calculated mass hardness (Simons et al. 2003)

The hardness of agglomerates made with magnesium stearate (MGST) is over estimated. The wet masses made from magnesium stearate had a low porosity and the liquid saturation of the pores created by the solid particles was close to 80%. These granules were therefore actually in the funicular saturation state, with the particles not in contact, but rather suspended in liquid with some air present. Hence, the model is not applicable. Further investigations on the measured mass hardness can be made by individually neglecting the capillary, friction and viscous elements of the three-term equation (Pepin et al. 2001).

SPHERICAL AGGLOMERATION STUDIES

Spherical agglomeration is an industrial process traditionally used to separate or recover fine solids dispersed in a liquid suspension through the addition of a second immiscible liquid (binder) which presents an affinity for the solids and is capable of forming small liquid bridges that hold the particles together. Under appropriate physicochemical conditions the desired particles can be selectively agglomerated and removed from the slurry.

More recently, the spherical agglomeration technique has been used for the manufacture of high value products, such as crystalline pharmaceutical drugs, and is attracting increasing attention in the bioprocessing area. However, spherical agglomeration has yet to reach widespread commercialisation beyond the minerals industry. This is despite the simplicity of the process, the low cost of installation required and the possibility of agglomerating particles down to a few microns in size and is probably due to the lack in understanding of the controlling mechanisms involved. Even in the minerals industry, there is scope for improvement in understanding of the process, particularly as the requirement to recover very fine particles increases due to economic considerations. Viewing the process as a production, rather than separation, technique may also hold new opportunities for minerals applications.

In the work reported here, a micro-scale approach has been taken to investigate the mechanisms that lead to spherical agglomeration. The geometry and the strength of liquid bridges formed between pairs of particles with diameters in the range 70 to 130 μm , submerged in a second liquid, have been analysed and compared with values predicted by theory. The effects of different surface properties, created by the addition of surfactants and electrolyte, on the bridge behaviour and the forces developed between the particles, have also been investigated.

LIQUID PHASE MEASUREMENT OF BRIDGE PROPERTIES

For measurements of liquid bridge forces between particles suspended in liquid media, the pipette shapes shown previously must be adapted so that their tips can be submerged in an optically clear dish under the view of a water objective (Fig.4). For such cases, it was not possible to monitor the movement of the bend using the optical follower. Hence, a manual technique, taking the distance measurements directly from the image sequences, was used for the liquid phase work. Next generation MFBs, have done away with the optical follower and instead measure the amount of bend of a pre-calibrated flexible strip using a linear variable differential transducer of much higher resolution (Pratola et al. 2001) (see below).

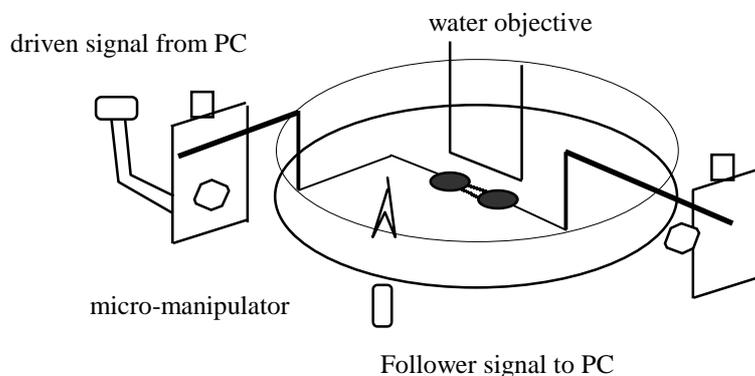


Fig.4. Schematic of the set up used to form a liquid bridge in a second immiscible liquid

LIQUID BRIDGE STRENGTH IN SPHERICAL AGGLOMERATES

Experiments were conducted to determine the effects on bridge geometry and strength caused by having a liquid phase continuous medium under a range of physico-chemical conditions. Glass ballotini spheres of diameter in the range of 70-130 μm were used as the solid particles, with silicon oil 100cSt (BDH) and water ("Analar" BDH) as the bridging and suspending liquid respectively. One electrolyte (NaCl -AnalaR) and two surfactants (sodium dodecyl sulphate, SDS-specially pure, cetyltrimethylammonium, CTAB-biochemical grade) were used to modify zeta potentials and interfacial energies. The pH was adjusted with either KOH or HCl (0.1M), both supplied by Fisher Scientific.

A rupture sequence of a silicon oil bridge in water is shown in Figure 5. From such images the advancing and receding contact angles can be measured and the liquid bridge profiles determined using the geometrical approximation developed during the gas phase work. It was observed that the variation in contact angle hysteresis between aqueous solutions at different pH, electrolyte and surfactant concentration was directly related to the zeta potential on both the glass and oil droplet surfaces and, hence, the double layer interaction between the two (Rossetti and Simons 2001). For instance, at pH ~ 3 , the potentials of glass and silicon oil have opposite signs, leading to attraction and, hence, a more stable glass-oil interface. This is reflected in a lower value of the receding contact angle corresponding to a higher degree of hydrophobicity of the glass. As the pH of the solution is increased, both the glass and the oil potential drop to negative values with an increase of the receding angle and a diminution of the hydrophobicity.

To validate this postulation, a hybrid Atomic Force Microscope (AFM), formerly used to measure interactions between ink particles and air bubbles in liquids in relation to paper pulp flotation (Berg et al. 1998), was adapted to provide quantitative

data on the double layer interaction. Measurements were made between a glass sphere attached to the tip of the cantilever and an oil droplet formed on a glass slide submerged in various aqueous solutions. This device has a higher force and distance resolution than the MFB. This work is currently being analysed and has not yet been published, but current indications are that the postulation is correct.

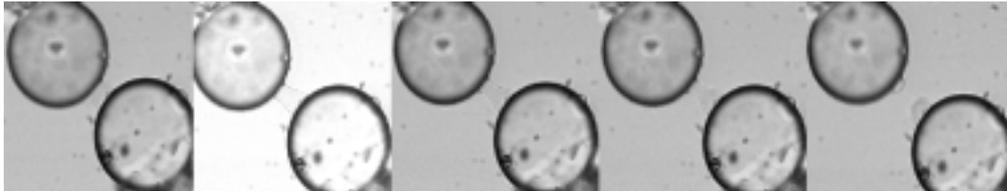


Fig.5. Rupture sequence of a 100cSt silicon oil bridge between two glass spheres, 43 μm (left) and 45 μm (right) diameter, separated at $1\mu\text{m/s}$. The volume of the bridge is $\sim 5500 \mu\text{m}^3$

Force measurements obtained using the MFB indicate the importance of selecting the appropriate conditions for successful agglomeration (Fig. 6). The geometrical model was seen to hold for the liquid phase experiments. Optimum conditions for agglomerating glass spheres with silicon oil rely on high interfacial tension between the particle and the suspending medium coupled with low zeta potential on the glass surface (e.g. by using CTAB as the surfactant).

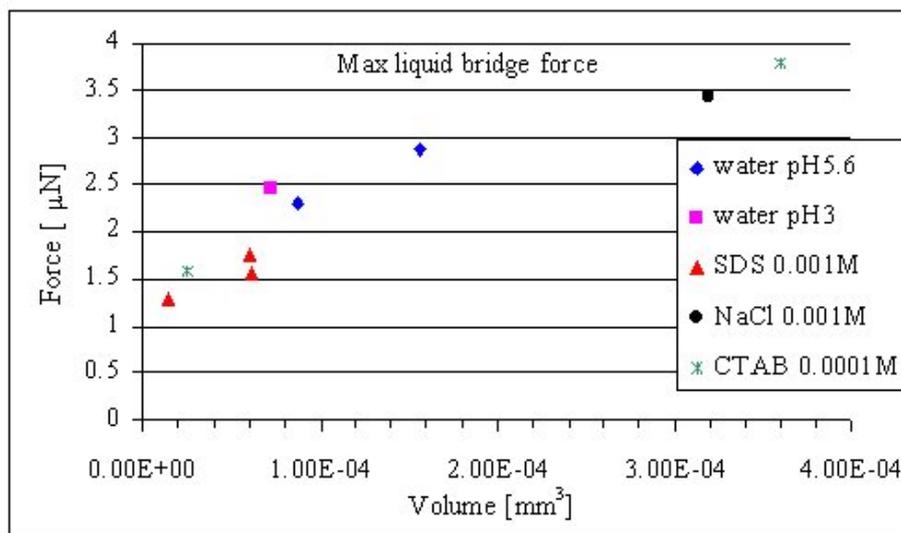


Fig.6. Max. liquid bridge force versus volume for a 100 cS silicon oil bridge in an aqueous solution

FLOTATION STUDIES

Flotation processes utilise air bubbles to capture solid particles from the bulk liquid gangue and subsequently rise to the surface, where they form a froth layer. The froth is then removed, either to recover or discharge the solid matter. A great deal of work has been published attempting to establish the influence of various parameters on froth formation and stability, such as the characteristics of the surfactants, electrolytes and solid particles. As interest increases on the application of dissolved air flotation (DAF) to the recovery of fine mineral particles, a fundamental understanding of the effect of various operational conditions on both froth formation and stability is vital for process optimisation and control, since the efficiency of the process depends heavily on these phenomena.

MEASUREMENT OF BUBBLE-PARTICLE INTERACTIONS

A novel apparatus has been developed to execute a wide range of interparticulate interactions, such as bubble-bubble and bubble-solid particle interactions. It is based on the earlier device described above and comprises the MFB where the interparticulate interactions take place, a sophisticated visual system, and a computer to control the electronic components of the set-up.

The MFB is shown in Fig.7 (Spyridopoulos and Simons 2002). At its centre is an optical-clear glass cell, which contains the liquid in which the interactions take place. A 3-axis micromanipulator is located above the cell, used either to hold directly the upper micropipette, at the tip of which either a bubble or particle is held, or the force measurement device. On the right of the cell, there is a piezo-driven branch, which holds a second micropipette. The piezo-driven branch can move the micropipette in very small steps, down to 0.05 μm . A visual system monitors and records the interactions in the cell.

The force-measurement device consists of two stainless-steel sheets connected vertically to rigid blocks. Any force between the particulates is transferred to the left-hand micropipette, which moves the lower block of the flexure-strip assembly. The latter carries the armature of an LVDT, which measures the displacement (resolution better than 0.1 μm), caused by the bending of the sheets. This depends on the force constant of the sheets which, for a 50 μm thickness is about 20 N/m. Lower force constants can be achieved, either by employing thinner strips, or by making holes at the centre of the sheets.

The visual system is comprised of two cameras and their respective displays; a high-speed camera capable of recording images between 30 and 3000 frames per second (fps) and a CCD camera placed at right angles to the high-speed camera. Each camera is coupled to a single-tube microscope fitted with a plan achromatic objective lens. The mechanism of the microscope permits the movement of the cameras in two dimensions.

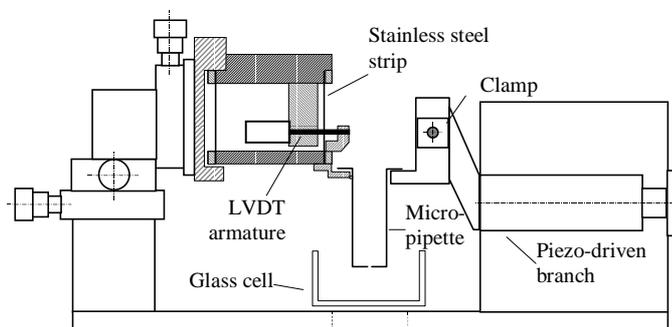


Fig.7. Schematic diagram of the MFB

The positioning of either two bubbles or a bubble and particle is achieved by using the 3-axis micromanipulator. The separation between the two bubbles is controlled by two means. Either a coarse adjustment can be made using the micromanipulator, or fine separation control can be obtained by moving the branch attached to the piezoelectric tube. Due to the inherent hysteresis of piezo tubes, a displacement sensor, LVDT, mounted in parallel with the piezo tube, follows and records the exact displacement. Instrument control and data acquisition is carried out via a computer.

BUBBLE COALESCENCE IN FLOTATION FROTHS

The effect of naturally occurring surfactants, known as humic substances (HS), on DAF froth formation and stability have been investigated by conducting bubble coalescence experiments. Bubbles of typical diameter 250 μm and 500 μm were grown on opposing nozzles inside the glass cell which was filled with an aquatic solution. The bubbles were brought together by the expansion of the piezoelectric tube. The rate of expansion and the resulting collisions were controlled by software, which controls the frequency and the value of the signals sent to a micromanipulator. The conditions under which the bubbles coalesce, e.g. coalescence frequency, type and concentration of HS in solution, and bubble size, have been observed. For the cases where coalescence did occur, the time elapsed for coalescence was measured using the high-speed video. After the formation of the bubbles, their vertical alignment and their positioning to a separation distance of about 10 μm , the piezoelectric crystal was expanded at a rate of 10 $\mu\text{m/s}$ and the event recorded using the high-speed camera. The event ended either with the coalescence of the bubbles, or when the piezoelectric tube had reached its maximum expansion, 100 μm . In the latter case, the bubbles were squeezed but did not coalesce. At least 10 repetitions were conducted for each case (bubble size, HS concentration and type). All the experiments and measurements took place at room temperature, 21-23 $^{\circ}\text{C}$.

Two samples of HS were tested: (a) a model polymaleic acid of molecular weight <500 Da, and (b) NOM taken from a sitka Spruce stand at Rumster in Caithness, UK.

Both of the samples were supplied from Thames Water Plc, UK, one of the major water companies in England and collaborators in this project.

HS solutions were prepared by dissolving a specific quantity of the sample in AnalaR-grade water (BDH, Dorset, UK). The pH of the aquatic solutions was in the acidic region (3.5-4.0) and a few drops of NaOH 1N (BDH, Dorset, UK) were required to adjust it to 5.25-5.5, the pH usually encountered in drinking-water treatment. The non-soluble matter was removed by passing the solutions through a filter paper with pore size of 0.2 μm .

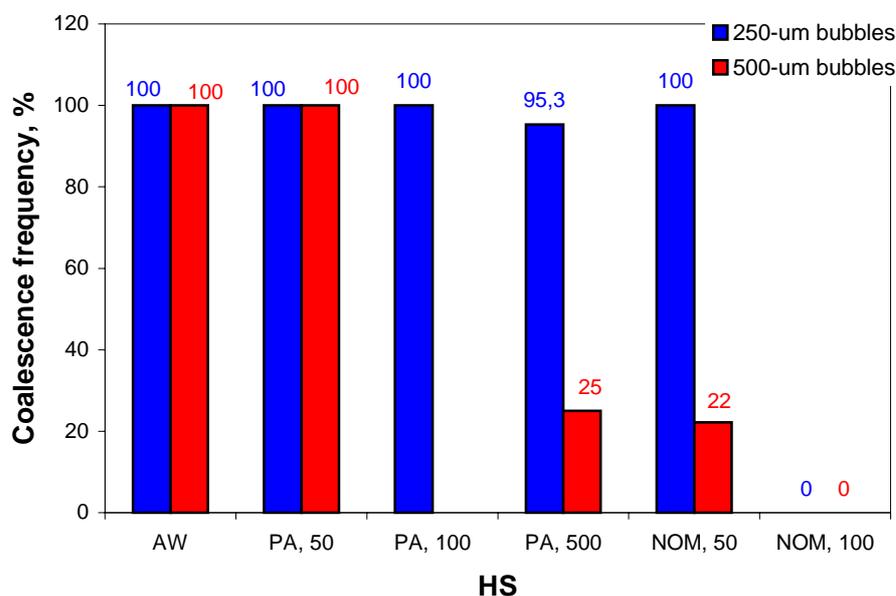


Fig.8. Coalescence frequency in HS solutions. AW: AnalaR water, PA: Polymaleic acid (MW <500 Da), NOM: Natural organic matter from Rumster – The numbers after commas denote the concentration in mg/l (Spyridopoulos and Simons 2002)

The results for the coalescence frequency versus HS concentration are shown in Figure 8. As was expected, there was always bubble coalescence in AnalaR water. The two HS tested in this work, however, showed different effects. The NOM reduced the coalescence frequency to 25%, even at the concentration of 50 mg/l. When its concentration was 100 mg/l, it inhibited the coalescence of bubbles completely. On the other hand, the polymaleic acid reduced the coalescence frequency only when its concentration was 500 mg/l. At lower concentrations, the coalescence frequency was 100%. These observations seem to be in accordance with the measurements of the surface tension of these HS, where the NOM appeared to be more surface-active than the polymaleic acid. From the same figure, also, it can be seen that the presence of HS affected the bubbles of 500 μm more than those of 250 μm .

The effect of the presence of solid particles on bubble coalescence has also been investigated. The experimental method and results have been reported elsewhere (Spyridopoulos and Simons 2001). In brief, coalescence depends not only on particle size, but also on the degree of hydrophobicity exhibited by the particle, measured through the 3-phase contact angle. High contact angles reduce coalescence.

CONCLUSIONS

A range of novel devices, known as Micro-Force Balances, have been developed that can be used to elucidate the fundamental mechanisms behind micro-scale interactions in mineral separation processes. It has been shown that direct measurement and observation of interactions between solid particles in the presence of liquids and bubbles is leading to important developments in understanding and modelling that, in turn, will lead to improvements in process design and operation. Further work is now underway in using such results to develop, for instance, computer simulations of flotation froths (Spyridopoulos et al. 2003).

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W szeregu procesach przeróbczych wykorzystuje się właściwości międzyfazowe takie jak hydrofobowość, napięcie powierzchniowe lub zwilżalność dla osiągnięcia zamierzonego celu. Jest interesujące znalezienie korelacji między właściwościami granic międzyfazowych w skali mikro, a realizacją procesów przeróbczych w skali makro. W celu lepszego poznania i zrozumienia tych zależności, konieczny jest bezpośredni pomiar oraz obserwacja tych właściwości. Szereg lat doświadczeń doprowadziły nas do zaprojektowania i budowy szeregu unikalnych aparatów o wspólnej nazwie Micro-Force Balance (MFB). Dzięki tym aparatom możemy mierzyć i obserwować oddziaływania między cząstkami ciała stałego w fazie gazowej i ciekłej, w temperaturze pokojowej oraz w podwyższonej. Przedmiotem badań z wykorzystaniem tych aparatów były takie procesy przeróbcze jak granulacja, flotacja, aglomeracja sferyczna. Przedstawiona praca zawiera opis działania aparatów oraz znaczące odkrycia dokonane w trakcie badań wyżej wzmiankowanych procesów.