



The dependency of the critical contact angle for flotation on particle size – Modelling the limits of fine particle flotation

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ABSTRACT

The flotation behaviour of fine particles is studied in this work. Fine methylated quartz particles within the size range from 0.2 to 50 μm , and with varying contact angles, were floated in a mechanical flotation cell. Results indicate that particles of a given size need to possess a minimum critical contact angle, which increases in value as particle size decreases, for flotation to be initiated. As a consequence, a non-floating component exists within a given size fraction. This is interpreted as a fraction consisting of particles below the critical contact angle for flotation for that size. The critical contact angle for flotation is explained in terms of the existence of an energy barrier for bubble–particle attachment. The flotation results are interpreted by means of Scheludko et al. (1976) and Drelich and Miller (1992) models for the floatability of fine particles. The experimental data compared very well with calculations using the Drelich and Miller equation, allowing extension to the prediction of the critical contact angle for flotation down to particle sizes well below the previous limits investigated, bridging the gap existing in the literature.

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1. Introduction

In flotation, fine particles show lower flotation rate, resulting in low flotation recovery. The recovery of particles by air bubbles during flotation occurs after three consecutive sub-processes of collision, attachment and stability have taken place to form a bubble–particle aggregate. The whole process, termed collection, may be expressed as (Dai et al., 1998):

$$E = E_c \cdot E_a \cdot E_s \quad (1)$$

where E_c , E_a and E_s are the collision, attachment and stability efficiencies respectively. Collision is mainly controlled by hydrodynamics (e.g., bubble rise velocity, bubble size) in the flotation cell, while attachment is dominated by interfacial behaviour (surface forces) between bubble and particle. For fine particles, which possess low inertia, the stability efficiency is essentially unity (Dai et al., 1998). Particle recovery by flotation is sensitive to both particle size and contact angle (Crawford and Ralston, 1988), with very fine particles (<10 μm) and coarse particles (>100 μm) floating poorly, but for different reasons. Coarse particles have low flotation rate due to detachment problems associated with disruptive forces in the flotation cell (Pyke et al., 2003), whereas fine particles' poor floatability emanates from low collision efficiency between bubbles

and particles. Decreasing particle size results in a decrease in collision efficiency as particles are not able to deviate from fluid streamlines to collide with bubbles (Weber and Paddock, 1983; Yoon and Luttrell, 1989). The bubble–particle collision frequency is controlled by cell hydrodynamics and kinetics, making the problem of fine particle flotation partly a kinetic one. This implies that particles should eventually float if given sufficient residence time in the flotation environment. However, flotation of fine particles may be hindered by the existence of an energy barrier that prevents successful bubble–particle attachment after collision, thus making the problem of fine particle floatability also a thermodynamic one. Put differently, fine particles may possess insufficient kinetic energy to displace the intervening liquid layer between the colliding particle and bubble (Hewitt et al., 1995).

The energy barrier manifests as a critical contact angle, which represents the amount of activation energy that must be overcome before bubble–particle attachment can occur. Theoretical considerations and experimental data published to date support the existence of a critical contact angle below which flotation does not occur (Crawford, 1986; Gontijo et al., 2007; Miettinen, 2007), but validation of the experimental data for fine particles with theoretical predictions has not yielded satisfactory agreement. This disparity is discussed further below.

In their theoretical analysis of the floatability of fine particles, Scheludko et al. (1976) proposed that the kinetic energy of particles must be larger than the energy needed to overcome the

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resistance to the formation and expansion of the three phase contact line for successful particle–bubble attachment to occur. This energy must be supplied by the kinetic energy of the particle. At equilibrium, the kinetic energy balances the energy needed to form and expand the three phase line of contact, from which a minimum particle diameter, $D_{min,K}$, for flotation may be estimated (Scheludko et al., 1976):

$$D_{min,K} = 2 \left[\frac{3\gamma_{slv}^2}{V_t^2 \Delta\rho \gamma_{lv} (1 - \cos \theta)} \right]^{\frac{1}{3}} \quad (2)$$

where θ is the receding contact angle, γ_{slv} is the line tension (which is the reversible work which must be done to isothermally expand the unit length of the three phase contact line), γ_{lv} is the liquid–vapour surface tension, $\Delta\rho$ is the difference between the particle and fluid densities and V_t is the bubble–particle approach velocity. To date, this theoretical model has not been satisfactorily validated with experimental data due to the uncertainty in the value of the line tension and the lack of data for very fine particles. There is no consensus on the line tension value used in Eq. (2) (Mingins and Scheludko, 1979; Drelich, 1996; Amirfazli and Neumann, 2004; Lin et al., 1993; Li et al., 1992), since the experimental values of line tension in the literature are quite varied, ranging from 10^{-12} to 10^{-5} N, while theoretically determined values range from 10^{-12} to 10^{-10} N (Drelich, 1996). A recent review of the theoretical and experimental data on line tension concluded that sometimes the values of line tension are inappropriately compared for dissimilar systems which may not be reasonably expected to have the same value even from a theoretical perspective (Amirfazli and Neumann, 2004). The values of line tension quoted by Scheludko et al. (1976) were estimated for particles of a given size (about 2 μm) and contact angle (between 20° and 40°), and thus cannot be expected to fit experimental data for substantially larger particles (about 10 μm or larger).

Drelich and Miller (1992) revised Scheludko et al. (1976) approach and proposed that a pseudo-line tension, which takes into account surface heterogeneities and roughness, will give a better correlation with experimental data compared to the line tension. Using the same theoretical basis used to derive Eq. (2), Drelich and Miller (1992) recognised that:

$$\gamma_{slv} = \gamma_{lv} \cdot (1 - \cos \theta) \cdot r_c \quad (3)$$

where r_c is the critical radius of wetting, i.e. the bubble drop radius below which there is no effective attachment between the solid and dispersed phases. Substituting Eq. (3) into Eq. (2), an alternative expression for the minimum particle size, $D_{min,K}$, was proposed:

$$D_{min,K} = 2 \left[\frac{3r_c^2 \gamma_{lv} (1 - \cos \theta)}{V_t^2 \Delta\rho} \right]^{\frac{1}{3}} \quad (4)$$

Drelich and Miller (1992) determined experimental values for the critical radius of wetting, r_c , as a function of contact angle, θ . The r_c values were estimated by extrapolating a plot of θ versus r to $\theta = 0$, for methylated quartz plates in distilled water at pH 5.6.

In this paper, the flotation behaviour of fine methylated quartz of different particle size and contact angle was studied. The existence of a critical contact angle for flotation, which is particle size dependent, is shown. Further, to investigate contact angle heterogeneity within individual classes of particles, the contact angle of feed and tails from selected tests was measured by means of film flotation, which uses the critical surface tension of wetting (Zisman, 1964). Other methods, such as Washburn technique (Washburn, 1921), were considered not reliable for fine particles (<10 μm), because fine particles tend to form aggregates when dry, biasing the measurement. The critical surface tension of wet-

ting was determined in film flotation at 0% floating (Yarar and Kao, 1984), as opposed to 50% floating which is also suggested in the literature (Fuerstenau et al., 1991). This is because for very fine quartz particles the first process to occur when particles are placed on the liquid surface is capillary penetration, resulting in particles sinking as aggregates. At 0% floating (or 100% sinking) it is ensured that both aggregates (which sink due to gravity at higher surface tension values compared to individual particles) and individual particles sink, eliminating any ambiguity in the value of the surface tension of wetting. In essence, this approach gives the minimum value of the surface tension of wetting from a range of possible values. Film flotation experiments were carried out on samples of feed and tailings, and differences in contact angle interpreted to be a result of segregation in flotation of particles having different size and contact angle values.

Finally, the flotation results are validated against the Scheludko model (Scheludko et al., 1976) and its variation (Drelich and Miller, 1992), based on theoretical considerations in regards to the limits of fine particle flotation.

2. Materials and methods

All glassware was cleaned by immersion in concentrated sodium hydroxide of pH above 13 and sonication for at least 30 min to remove any organic contamination. The glassware was then thoroughly rinsed with de-ionised water to remove all the sodium hydroxide and finally dried overnight in a clean oven at 110 °C. All chemicals used in this work were of analytical reagent quality and were used without further purification.

2.1. Materials preparation

Microcrystalline quartz of particle size less than 10 μm was obtained from Sigma Aldrich. These particles are designated as *Sigma* quartz. Other quartz size fractions were obtained by wet grinding lumpy crystalline quartz in a laboratory stainless steel ball mill and wet sieving at 38 μm . The $-38 \mu\text{m}$ size fraction was passed through a 1" cyclone (precyclone) with the overflow discarded and the underflow classified in a Warman cyclosizer that split the particles into six different size fractions (C1, C2, C3, C4, C5, and C6). Selected size fractions (C3, C5, C6 and Sigma) were subsequently cleaned separately to remove any organic and inorganic contaminants (such as iron from the grinding media) before subsequent manipulation to alter the contact angle values.

2.2. Cleaning quartz particles

Hot aqua regia and piranha solution were used to clean each of the selected quartz size fractions of any surface impurities. The reaction time was varied from 6 to 10 h depending on the particle size and the reagent used. The acid was washed out by rinsing the particles with de-ionised water until the pH became neutral. A centrifuge was used to accelerate particle settling for the finest size fractions (Sigma and C6). The particles were dried overnight in a clean oven at 110 °C in a covered clean beaker and subsequently stored in clean and sealable glass containers.

2.3. Methylation of silica particles

The contact angle of clean silica particles was manipulated to different degrees of hydrophobicity by using trimethylchlorosilane (TMCS) (Blake and Ralston, 1985). Because TMCS reacts readily with water in the atmosphere, the methylation procedure was carried out in a glove box under dry nitrogen. A known mass of cleaned quartz for each size fraction was placed in a 1 l laboratory

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