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Flotation of fine particles from binary mixture by ionic microbubbles

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Abstract : This article reportsthe separation efficiency of fine particles by ionic microbubbles (IMB). IMBs were generated using a microbubble generator. Thediameter of the bubbles was in the range of 30-50 μm. Acationic surfactant Cetyl Trimethyl Ammonium Bromide (CTAB), has been used to separate zinc oxideparticle (2.2 μm particle diameter) from a mixture of zinc oxide and silica powder (11 μm particle diameter). Agglomeration between particles of different charge is a potential problem. However, the initial results indicate a good separation using a novel system. The results are compared with conventional batch scale flotation.

Keywords : Collision; Fine particle; Flotation; Ionic microbubble; Separation.

INTRODUCTION

Flotation is a process in which particles are removed selectively from water by attachment to rising air bubbles. It has a long history in the mineral beneficiation industry[1]. In recent years, it has become an interesting method of clarifying effluent or potable waters[2]. It has been employed to remove oil particles from water[3]. In conventional mineral flotation, the particles are typically 0.5 mm in diameter or larger; the bubbles are large, of order 1 mm; and the solids content in the pulp is normally high, of order 25% by weight. In effluent treatment, in contrast, the particles are typically small, < 20 μm in diameter and close to neutral buoyancy. The concentration of particles is diluted ~ 20 ppm. It is advantageous to use very small bubbles, often less than 100 μm in diameter. These are made either by a “dissolved air” process in which the liquid to be treated is saturated with air at about 3-4 atm. and then pressured is released to atmospheric pressure. When the air comes out of the solution, a swarm of fine bubbles are formed. In the “dispersed air” method, bubbles are formed mechanically in a mixer or sparger, or electrolytically[4]. Microbubbles have been used in separation and recovery of proteins[5-8], dye and pigment removal[9-10], removal of heavy metal ion from water[11] and fine particle recovery[12]. The effect of electrical double layer interactions and the charge on the particles and bubbles on the the rate of recovery of fine polystyrene particles has been studied previously[13]. Cilliers and Bradshaw[14] have studied the separation of pyrite and quartz mixtures at batch flotation scale, and found that the sulfur recovery increased through the use of stable dispersion of charged colloidal microbubbles. Recently, Fuda and Jauregi[8] carried out a more detailed study on the mechanism of separation of proteins by ionic charged colloidal microbubbles. They concluded that electrostatic interactions were indeed the driving force for the separation. The flotation of fine particles has become particularly important in recent years as advances in grinding are allowing low-grade mineral deposits to be economically exploited. The poor recovery of fines by flotation can be attributed to the low probability of bubble–particle collision, which decreases with decreasing particle size. However, bubble–particle interactions (such as electrostatic and hydrophobic forces) are important in determining the selectivity of a separation. One of the major problems in the flotation of fine particles is the decreased probability of collision between the particles and the bubbles, which can be improved by a reduction in bubble size. Additionally, smaller particles having lower momentum may not be able to break through the liquid barrier surrounding a bubble. In order to overcome this limitation, flotation exploiting electrostatic interactions has a good potential. Also, one of the major problems in the flotation of fine particles is the decreased probability of collision between the particles and the bubbles, which can be enhanced by a reduction in bubble size. Therefore, the study of the flotation of fine solids by micro bubbles is likely to intensify of the flotation process.

MATERIALS

Zinc (II) oxide (ZnO), silicon dioxide (SiO_2) andcationic surfactant cetyl trimethyl ammonium bromide (CTAB)were purchased from MERK Chemicals. Hydrochloric acid (HCl) (1 M) and 1 M sodium hydroxide (NaOH), used to alter the pH of the mineral suspensions and were purchased from Sigma"Aldrich (India). All the chemicals used in the present work had a purity of > 96%. Throughout the entire experiment distilled water was used. Particle–surfactant interactions have been studied by measuring the zeta potential changes at different surfactant concentrations and pH.

EXPERIMENTAL PROCEDURE

In this study the potential of ionic microbubbles (IMB) for the separation of a binary mixtureof fine mineral particles (i.e. zinc (II)oxide and silica) is demonstrated. The results are compared to conventional batch scale flotation on the same system. The surfactant was Cetyl Trimethyl Ammonium Bromide, a commonly used cationic surfactant. Pressurized dissolution method was used to produce the microbubbles in the surfactant solutions. Air was allowed to dissolved in water by applying a pressure of about 3-4 atm. The microbubble dispersions were prepared in a $17\times10^{-3}m^3$ vessel. The dispersion of microbubbles in water were continuously recycled through the vessel. The concentration of CTAB was varied from 5×10^{-3}–$20\times10^{-3}kg/m^3$CTAB. The dispersion had an air hold-up of 15% at the CTABconcentration of $5\times10^{-3}kg/m^3$. Preliminary tests carried out on the stability of ionic microbubble showed that $5\times10^{-3}kg/m^3$CTABwas sufficient to ensure the dispersion did not break down when being pumped. The batch mode of flotation was carried out in a vessel of 0.25 m diameter and 0.32 m height. The vessel was filled with a suspension of $16 \times10^{-3} m^3$ water, 10 g zinc(II) oxide and 10 g silica. The IMB dispersion was pumped into the base of the vessel, rising through the slurry to form froth on the surface. The ionic microbubbles were pumped into the flotation vessel at a rate of 16.67×10^{-5}–$33.33\times10^{-5} m^3/s$. The initial height of the feed suspension was 0.32 m, where 0.27 m was considered to be the lowest point. The froth reached the top of the vessel after an average of 3 min, after which the concentrate was recovered for 30 min. Concentrate samples were taken at 3 min intervals. The mass of solids recovered was measured, and the grade was determined by sulfuric acid digestion of the zinc oxide.

RESULTS AND DISCUSSION

Flotation using IMBs initially results in a high concentrate grade (i.e. 64.16%)of zinc oxide in the first minute. However, at the end of the experiment, the cumulative grade decreased to 38.81%. The recovery was 15.72% higher than the recovery achieved in conventional flotation beyond the cumulative grade of 45%. This is due to the greater depth of the froth in the IMB flotation system. The deep froth generated in the IMB system allowed the drainage of the unattached particles from the froth into the pulp. The collapsed bubbles releases the non-selectively entrained particles which resulted in a higher concentrate grade but a lower recovery. The decrease in concentrate grade with time for the IMB system can be ascribed to both the froth stability and the rising pulp–froth interface. The stability of the IMB-generated froth was such that when the froth reached the top of the vessel, it did not immediately overflow but continued rising before it overflowed. While the froth continued to rise, the entrained liquid and particles overflowed to the concentrate. As the pulp–froth interface rose, the froth depth decreased which led to a decrease in the particle froth residence time, and consequently, less drainage of the unattached particles. The fraction of zinc oxide particles that were recovered attached to bubbles can be estimated by assuming that all the silica recovered in both systems wasentrainment. This, however did not take into account particle size or density difference. The results of the same study were compared with that in conventional batch flotation(i.e Denver cell)as shown in Figure 1, which shows the cumulative grade and recovery of zinc oxide over the course of the experiments. The study wascarried out in a 1 $\times 10^{-3}$ m^3 Denver cell, with 0.6 $\times 10^{-3}$ m^3water, 10 g zinc oxide and 10 g silica. The dispersion was conditioned for 2 min with CTAB at a concentration of 1.5 kg/ m^3 at an impeller speed of 1500 rpm. Air was then introduced at a rate of $3.33\times 10^{-5} m^3/s$. The bubble of size in the conventional flotation was of the order of 6×10^{-4} m. The concentrate samples were collected. The mass and grade of the samples was determined by following a procedure similar to the IMB flotation. All flotation experiments were carried out at the natural pH 7. In case of the Denver cell, over around 7 minutesof flotation time, from an initial feed grade of 50%, the cumulative grade was just 46.05%, with a recovery of 58.4%. This suggests that there was high recovery of non-selectively entrained particles. Over the course of the experiments, the average attached was 31.95% for the Denver system and 31.78% for the IMB system, suggesting that the IMB flotation system was more selective. In order to take into ac-

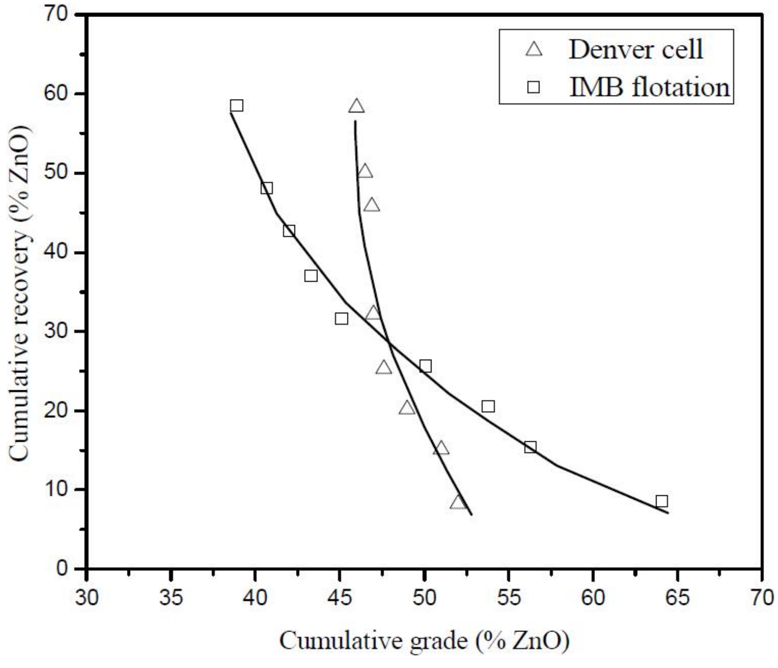

Figure 1 : Comparison of grade-recovery results between Denver cell flotation and IMB flotation

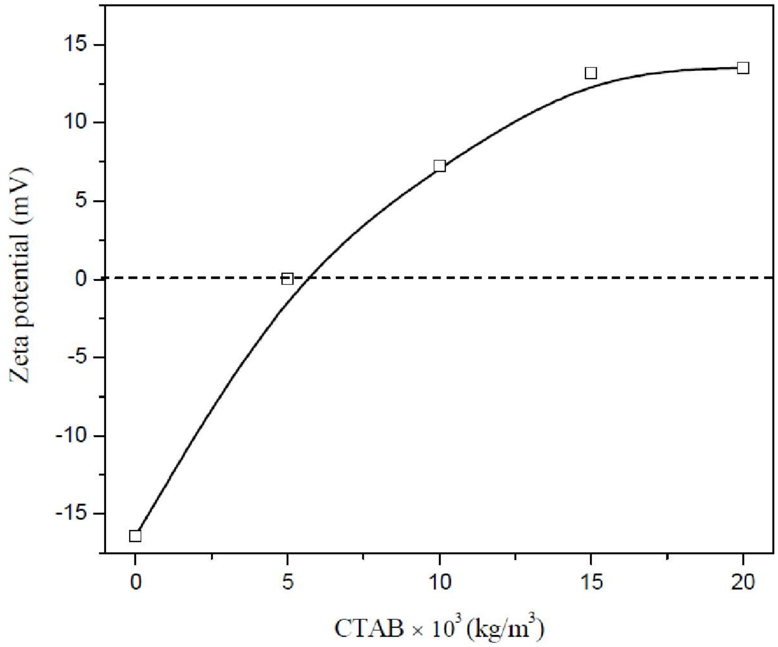

Figure 2 : The effect of CTAB addition on the zeta potential of ZnO

count the issues surrounding the stability of the IMBs and the rising pulp–froth interface, however, the fraction of attached zinc oxide recovered in the first minute of flotation can be considered.

In the first minute, the fraction of attached zinc oxide recovered to the concentrate was 28.1% and 58.4% for Denver and IMB flotation, respectively. The decrease in the fraction of zinc oxide that is recovered attached to bubbles in the IMB system over the course of an experiment demonstrates further the effect of the rising pulp–froth interface, showing that the separation becomes less selective over time. Moreover, in the first minute of flotation, the results clearly show the increased selectivity of the IMB flotation compared with the conventional flotation. In conventional flotation, surfactant selectively adsorbs to particles during the conditioning phase. The selectivity was electrostatic in nature, with oppositely charged particles and surfactant interacting. The effect of CTAB addition on the zeta potential ofZnO is shown in Figure 2.

The adsorbed surfactant hydrophobises the particles such that they adsorb to the air bubbles introduced into the system. Conversely, in ionic microbubble flotation, both the bubbles and the particles carry a charge. As the ionicmicrobubbles pass through the feed solution, oppositely charged particles are attracted due to electrostatic forces. The driving force for the initial contact between the particle and the microbubble occurs via electrostatic interaction. This also means that the IMBs are being used as a means to bring the flotation reagents into direct contact with the mineral particles. One of the key properties of IMBs is their stability. This was noted during the IMB flotation experiments. After stopping the operation, the froth phase in the vessel remained stable without collapsing. Furthermore, the mass and grade of particles remaining in this froth was measured after a period of 30 min after flotation stopped. This sample accounted for 12.3% of the initial zinc oxide feed, at a grade of 76.12%. Including this froth sample in the results gives a cumulative grade of 62.56% and a cumulative recovery of 36.2%. The interaction between particles of different charge could pose an additional problem, such as multilayering and agglomeration. However, the results obtained in these experiments suggest that this has not occurred. However there are clearly issues relating to scale-up. The potential use of IMBs for the selective separation of minerals has been demonstrated. Further work is recommended in order to optimise the IBM flotation system to compare the separation efficiency between IMB flotation and other small bubble flotation systems.

CONCLUSION

The following conclusions can be drawn from the study

(1) Over the course of the experiments, the average attached was 31.95% for the Denver system and 31.78% for the IMB system, suggesting that the IMB flotation system was more selective.

(2) Over around 7 min flotation time, the cumulative grade of the concentrate varied in case of the conventional flotation cell. From an initial feed grade of 50%, the cumulative grade over 7 min was just 46.05%, with a recovery of 58.4%.

(3) Flotation using IMBs initially resulted in a higher concentrate grade, 64.16% zinc oxide in the first minute. However, by the end of the experiment, the cumulative grade decreased to 38.81%.

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