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Fundamental and flotation techniques assessing the effect of water quality on bubble-particle attachment of chalcopyrite and galena

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Abstract

Bubble-particle attachment has been studied in the most fundamental way from as early as 1934 by bringing a bubble into contact with a flat mineral surface and since then, techniques measuring this interaction have advanced. Water quality within flotation will impact the bubble-particle attachment and as more operations recycle their water on site, an understanding of this process becomes vital. This study uses an Automated Contact Time Apparatus (ACTA) to assess the effect of water quality on bubble-particle attachment of selected sulfide minerals; galena and chalcopyrite, from a fundamental perspective. Classical microflotation tests are complemented with collector adsorption and mineral potential under degrading water quality to validate the ACTA and gain an understanding of the effect of water quality on bubble-particle attachment as well as subsequent flotation. This investigation showed that the results from the ACTA qualitatively showed similar trends as that of the classical microflotation technique for measuring floatability, however the quantitively these methods showed very different results. Due to the dynamic nature of the microflotation technique it may be assumed that plant recovery will resemble the results from this technique closer than that of the ACTA. Furthermore, this investigation showed an increase in zeta potential of both minerals as the concentration of inorganic electrolytes in the water increased. It can thus be speculated that the increase in bubble-particle attachment with increasing ionic strength of synthetic plant water may be attributed to electrical double layer compression and particle agglomeration.

1. Introduction

Sven-Nilsson (1934) first presented the concept of induction time and described it as the minimum time that a bubble and particle are in contact such that the thinning of the hydration film to critical thickness and film rupture occurs. This author first measured the induction time by bringing a captive bubble towards a flat mineral surface and since then, techniques measuring this interaction have advanced. Water quality within flotation will impact the bubble-particle attachment and as more operations recycle their water on site, an understanding of this process becomes vital. This study uses an Automated Contact Time Apparatus (ACTA) to assess the effect of water quality on bubble-particle attachment of selected sulfide minerals; galena and chalcopyrite, from a fundamental perspective. Classical microflotation tests are complemented with collector adsorption and mineral potential under degrading water quality to validate the ACTA and gain an understanding of the effect of water quality on bubble-particle attachment as well as subsequent flotation. This investigation showed that the results from the ACTA qualitatively showed similar trends as that of the classical microflotation technique for measuring floatability, however the quantitively these methods showed very different results. Due to the dynamic nature of the microflotation technique it may be assumed that plant recovery will resemble the results from this technique closer than that of the ACTA. Furthermore, this investigation showed an increase in zeta potential of both minerals as the concentration of inorganic electrolytes in the water increased. It can thus be speculated that the increase in bubble-particle attachment with increasing ionic strength of synthetic plant water may be attributed to electrical double layer compression and particle agglomeration.

Keywords: Bubble-particle attachment, Froth flotation, Galena, Ions, Water quality

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quartz and water quality effects on attachment probability of pyrrhotite respectively. Although in principle the ACTA is a more robust technique for measuring bubble-particle attachment, it is important to validate this instrument against a classical technique of measuring bubble-particle interactions before it can be used as a quick diagnostic testing tool on flotation plants (Aspila et al., 2018).

The efficiency of bubble-particle attachment is affected by various factors, chemical, hydrodynamic and operational. Water quality within flotation has been identified as an important parameter affecting the bubble-particle efficiency and has been gaining huge interest of late due to water scarcity in parts of the world (Biçak et al., 2012; Ikumapayi et al., 2012; Manono et al., 2012; Slatter et al., 2009). In studies with quartz it was found that increasing the ionic concentration, the induction time shortened; indicating that the particles’ susceptibility to float increased (Laskowski and Iskra, 1970; Yoon and Yordan, 1991). These authors ascribed this effect to the compression of the electrical double layer with increases in ionic concentration; which was said to ultimately lead to the accelerated rupturing of the film at the air-water and solid water interfaces. Less fundamental batch flotation studies have shown that total solids recovery increased as the ionic concentration of plant water increased (Corin and Wiese 2014; Manono et al., 2012). While studies by Ikumapayi et al. (2012) showed that as the water quality deteriorated, the mineral recovery decreased. Boujounoui et al. (2015) studied what happens at the mineral surface in terms of the change in particle potential and adsorption of collector with changes in water quality and how this may affect the way bubbles and particles interact.

2. Materials and methods

2.1. Sample preparation

Galena and Chalcopyrite were obtained from Ward’s Science in 1 kg bulk packs. The respective samples were hammered into chunks passing 1000 μm. The respective chunks were placed into a pulveriser for about 10 s and subsequently dry screened using 125 μm, 106 μm, 75 μm and 38 μm screens for 30 min. The fraction above 125 μm was pulverised again and dry screened similarly. The fraction between 106 and 125 μm was used for the attachment time tests, as particles in this size fraction were then adjusted to 6.5 using the NaOH and HCl solutions. The particle bed was made from approximately 18 g of mineral sample in the fraction 106–125 μm. A slurry was made with the mineral particles and the water quality of interest, an equivalent of 50 g/t of SIBX was added (volume of 90 μL) and conditioned for 2 min. The pH of the slurry with 1 SPW prior to the addition of SIBX was 6.5, the subsequent mineral and water mixtures were then adjusted to 6.5 using the NaOH and HCl solutions. The particles in the slurry were allowed to settle for about 3 min and the liquid at the top was pipetted out, filtered and placed in the glass pool. The settled particles were used to build the particle bed in the pool. Each of the water qualities were tested in this way and each condition was performed in duplicate to minimise experimental error.

2.2. Water quality

Synthetic Plant Water (1 SPW) used by Wiese et al. (2005) was prepared using salts of analytical grade and was used as the standard synthetic plant water in this investigation. In order to mimic recycled plant water, the baseline plant water (1 SPW) was increased by five and ten times the total dissolved solids. These waters were termed 5 SPW and 10 SPW respectively. The composition of each of the synthetic plant waters are shown in Table 1.

2.3. Reagents

Sodium isobutyl xanthate (SIBX) of 97% purity, sourced from Senmin was used as a collector in this investigation. Collector solutions of 1% w/v were made fresh for each day of the experimental work, which was 0.06 mol/L SIBX xanthate solution. NaOH and HCl supplied by Merck were used for pH adjustment; 1% w/v solutions were made each day for both NaOH and HCl.

2.4. Automated contact time apparatus

The automated contact time apparatus used in this work has previously been described in Aspila et al. (2018) and October et al. (2019a, 2019b) and is illustrated in Fig. 1 (B). Details of the functioning of the machine and operational details have comprehensively been documented in October et al. (2019a, 2019b). The particle bed was made with approximately 18 g of mineral sample in the fraction 106–125 μm. A slurry was made with the mineral particles and the water quality of interest, an equivalent of 50 g/t of SIBX was added (volume of 90 μL) and conditioned for 2 min. The pH of the slurry with 1 SPW prior to the addition of SIBX was 6.5, the subsequent mineral and water mixtures were then adjusted to 6.5 using the NaOH and HCl solutions. The particles in the slurry were allowed to settle for about 3 min and the liquid at the top was pipetted out, filtered and placed in the glass pool. The settled particles were used to build the particle bed in the pool. Each of the water qualities were tested in this way and each condition was performed in duplicate to minimise experimental error.

2.5. Microflotation

In a beaker, 3 g of mineral was mixed with 50 mL of the water type under study. The pH of the slurry was then adjusted to 6.5 using the NaOH and HCl solutions. This was transferred into the microflotation cell (Fig. 1 (A)) developed by Bradshaw and O’Connor (1996) with a funnel; the funnel was washed with the water type until the slurry reached just above the recycle point of the microflotation cell. The pump was switched on to 90 rpm and the pulp was circulated for 5 min. The volume equivalent to 50 g/t of SIBX was added to the slurry (volume of 15 μL). The water type under study was topped up to the 250 mL point of the cell and the cone was put in place. The microsyringe, providing an air supply of 7 mL/min was inserted at the base of the microflotation cell. Concentrates were collected after 2, 6, 12 and 20 min of flotation. The concentrates were oven dried and weighed. This procedure was followed for each of the water types and each condition was performed in duplicate to minimise error.

<table>
<thead>
<tr>
<th>Water Type</th>
<th>Plant</th>
<th>Ca²⁺ (mg/L)</th>
<th>Mg²⁺ (mg/L)</th>
<th>Na⁺ (mg/L)</th>
<th>Cl⁻ (mg/L)</th>
<th>SO₄²⁻ (mg/L)</th>
<th>NO₃⁻ (mg/L)</th>
<th>CO₃²⁻ (mg/L)</th>
<th>TDS (mg/L)</th>
<th>Ionic Strength (mol/L)</th>
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<tr>
<td>1 SPW</td>
<td>80</td>
<td>70</td>
<td>153</td>
<td>287</td>
<td>249</td>
<td>176</td>
<td>17</td>
<td>1023</td>
<td>0.0241</td>
<td></td>
</tr>
<tr>
<td>5 SPW</td>
<td>400</td>
<td>350</td>
<td>765</td>
<td>1435</td>
<td>1200</td>
<td>880</td>
<td>85</td>
<td>5115</td>
<td>0.1205</td>
<td></td>
</tr>
<tr>
<td>10 SPW</td>
<td>800</td>
<td>700</td>
<td>1530</td>
<td>2870</td>
<td>2400</td>
<td>1760</td>
<td>170</td>
<td>10230</td>
<td>0.241</td>
<td></td>
</tr>
</tbody>
</table>
3. Results

3.1. Effect of water quality on the bubble-particle interactions

The automated contact time apparatus (ACTA) produced two outputs, the first is an attachment probability which is obtained from the images taken by the machine for each cycle (that is 66 cycles, with 396 possible bubble-particle attachment and thus the bubble-particle attachment probability for each run is the amount of bubbles that had particles attached to it over 396) and the mass of particles recovered in the collection bin. Fig. 2 shows the attachment probability and the mass of galena and chalcopyrite particles recovered. The results presented in Fig. 2 show that the attachment probability of galena was generally the same across the three water qualities, while the mass of galena particles recovered shows an increase with increasing ionic strength of the synthetic plant water. The attachment probability and mass recovered of chalcopyrite are seen to increase as the ionic strength of the synthetic plant water increases. The attachment probability of chalcopyrite in 1 SPW is observed to be 69.57% and increases to 90.40% in 10 SPW; the attachment probability of galena is generally the same across the various water qualities, however after 5 min of flotation it becomes clear that higher galena recoveries are achieved with synthetic plant water of increasing ionic strength. This trend of an increase in particles recovered with increasing ionic strength of plant water in the micro flotation tests qualitatively match the trend observed in the ACTA in terms of mass recovered. However, it must be noted that quantitively the results from these two methods are vastly different when studying the percentage increase in recovery between the water types. The collector-chalcopyrite system in Fig. 4 shows similarly high recoveries of chalcopyrite across the water types; ranging from 84.42% in 1 SPW to 88.80% in 10 SPW. A trend of increases in chalcopyrite recovery with increasing ionic strength is however observed even for a system with no collector. These results complement the
attachment probability and mass recovered data provided by the ACTA.

The microflotation results in Figs. 3 and 4 also allow for a comparison between collector and collectorless test results across the varying water qualities. In the system without collector the difference in recovery of both galena and chalcopyrite becomes clearly evident in that the recovery increases as the ionic strength of the synthetic plant water increases. Further, the expected result of collector flotation resulting in higher recoveries compared to collectorless flotation is observed; however, in both mineral systems recovery with 10 SPW in the absence of a collector is very close to the mineral recovery with 1 SPW and 50 g/t collector.

3.2. Effect of water quality on collector adsorption

The effect of water quality on bubble-particle interactions was examined using a newer technique (the ACTA) as well as an established microflotation technique. The trends from these two techniques were generally similar. However, it is still not understood what happens at the mineral surface and why the differences in recoveries are observed under varying water quality. It therefore became important to understand the effect of ionic strength on collector (xanthate) adsorption. Fig. 5 depicts the concentration of xanthate left in the solution and by mass balance xanthate adsorbed on the galena surface under the varying water qualities. It is evident that more xanthate is left in solution and less adsorbed on the mineral surface when the ionic strength of the synthetic plant water is increased. Under 1 SPW only 12% of xanthate was left in the solution while 67.8% of the xanthate stayed in solution at 10 SPW.

The residual and adsorbed xanthate concentration in the chalcopyrite system is depicted in Fig. 6. It can be seen that the amount of xanthate adsorbed does not seem to differ greatly with increases in the ionic strength of the synthetic plant water.

3.3. Effect of water quality on zeta potential

To further understand what happens at the mineral surface under the varying water qualities, it was deemed necessary to study the effect of ionic strength on the zeta potential of the minerals. Fig. 7 gives an...
account of the zeta potential of galena with synthetic plant water of increasing ionic strength at pH 6.5. It is clearly evident that ultra-purified water yields the most negative potential of galena particles and that the potential of galena increases with increases in the ionic strength of the synthetic plant water. It should be noted that other studies by October et al. (2019a, 2019b) showed a negative zeta potential for both galena and chalcopyrite in ultra-purified water over the pH range 2 to 12. This study also showed that generally the zeta potential of these minerals increased with increasing ionic strength across the pH range.

The zeta potential results observed with galena are largely similar those of chalcopyrite shown in Fig. 8, where the potential of the chalcopyrite increases with an increase in the ionic strength of synthetic plant water.

4. Discussion

4.1. Comparison between the outputs of the ACTA and microflotation

Although the exact numbers and magnitude of the outputs of these instruments cannot be compared, the trend may be directly compared. Fig. 9 provides a graphical representation with regard to the output trends of the two techniques under degrading water quality. The galena test work showed a general increase in the mass of particles recovered in the ACTA, while attachment probability did not change with increase in the ionic strength of the plant water. The microflotation tests however corroborated the mass recovered in the ACTA as seen in Fig. 9(A). The discrepancy between these two measures may be due to either multiple particles sitting on one bubble or heavy and agglomerated particles being collected when attachment does occur on few bubbles; which would lead to low attachment probability, but high mass recovered. Fig. 9(B) represents the chalcopyrite trends in bubble-particle interactions; the two ACTA outputs complement the microflotation trend. A clear trend of an increase in bubble-particle interactions with an increase in ionic strength of water is observed. Generally, the ACTA yielded both reproducible results and brought about results qualitatively comparable to the classical microflotation method. Quantitatively these two methods yield very different results, the ACTA recovery shows an increase of more than tenfold, whilst the increase in microflotation recovery is less than 50%. Due to the dynamic nature of the microflotation technique it may be assumed that plant recovery will resemble the results from this technique closer than that of the ACTA.

4.2. Effect of water quality on bubble-particle interactions

The ACTA and microflotation techniques respectively showed an increase in the bubble-particle attachment and recovery as the ionic strength of the plant water was increased. This result is in agreement with fundamental bubble-particle attachment tests where authors have shown that increases in ionic concentration lead to shorter induction times and ascribed this to the additional ions compressing the electrical double layer, resulting in the faster rupturing of the liquid film which makes bubble particle attachment occur faster (Laskowski and Iskra, 1970; Wang et al., 2015; Yoon and Yordan, 1991). These authors have also referred to certain ions in water having a “bridging” effect between negatively charged particles and negatively charged collector molecules, thus resulting in an increase in particle hydrophobicity. Other studies have however shown that in the presence of a collector, mineral hydrophobicity is reduced in saline waters due to the presence of oxy-hydroxyl, carbonate and sulfoxy species (Hodgson and Agar, 1989; Ikumapayi et al., 2012; Kirjavainen et al., 2002).

4.3. Effect of water quality on collector adsorption

The adsorption of xanthate on the galena surface decreased as the ionic concentration of the plant water increased, this is in agreement with studies that have proposed that inorganic electrolytes compete with xanthate collector for adsorption on the mineral surface or that the amount of xanthate available for adsorption on the surface is reduced due to the formation of insoluble complexes between the collector and ions (Fuerstenau and Somasundaran, 2003; Hodgson and Agar, 1989; Ikumapayi et al., 2012; Laskowski, 2013). Considering that the galena surface adsors less xanthate as the ionic strength of the water increases, it is expected that the galena particles will be less floatable; this was however not observed in this investigation as microflotation recovery, showed increases with deteriorating water quality. This suggests another mechanism having a greater effect on the galena bubble-particle attachment than xanthate adsorption; which may be related to the charge of the particles. Microflotation tests showed that in a chalcopyrite-SIBX system, high recoveries are achieved, regardless of the water quality, and may be due to the floatable nature of

Fig. 6. Collector concentration adsorbed on chalcopyrite surface and remaining in solution.

Fig. 7. Zeta potential of galena under varying water quality at pH 6.5.

Fig. 8. Zeta potential of chalcopyrite under varying water quality at pH 6.5.
5. Conclusions

This investigation showed that the results from the ACTA qualitatively showed similar trends as that of the classical microflotation technique for measuring floatability. Although the magnitude of the results from these two techniques are very different, the ACTA may be useful in showing the effect of changing a parameter on bubble-particle attachment when studying trends become important. From a practical perspective although microflotation tests are faster, the ACTA set-up is useful in showing the effect of changing a parameter on bubble-particle attachment in solutions of high ionic strength.

4.4. Effect of water quality on mineral zeta potential

The sulfide minerals in this study showed a very negative potential in ultra-purified water and became less negative as theionic strength of the water was increased. This is indicative of the adsorption of metal cations on the mineral surface and the compression of the electrical double layer (Moignard et al., 1977). As previously described, this may lead to a faster thinning of the water between the solid particle and the air bubble improving bubble-particle interactions. Due to increases in inorganic electrolytes in the bulk solution, counter-ions enter the Stern layer which in turn thicken the diffuse layer due to an increase in attraction force and decrease in repulsion force (Wang and Peng, 2014). Thus, the compression of the electrical double layer in high ionic strength solutions may promote particle aggregation; particle aggregation is common when the potential is close to 0 mV (Salopek et al., 1992; Wang and Peng, 2014). This work was conducted at pH of 6.5, the potential of the minerals, particularly at 10 SPW is closer to 0 mV. In addition to faster rupturing of the water film between the bubble and particle, particle agglomeration may also be responsible for the increase in bubble-particle attachment with increasing ionic strength of the plant water.

It should also be noted that the zeta potential of the bubble is affected by the solution pH, concentration of electrolytes and type of metal ions present in the solution (Yang et al., 2001) and thus this may be important to investigate in future studies.

Fig. 9. Trend comparison of the microflotation and ACTA output (A) galena and (B) chalcopyrite.
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