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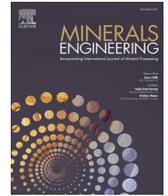
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Fundamental and flotation techniques assessing the effect of water quality on bubble-particle attachment of chalcopyrite and galena

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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 quantitatively 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 then moving it away from the mineral surface. Glombotsky (1953) used a similar technique to determine the induction time, however instead of a flat mineral surface, the single bubble was brought into contact with a bed of particles. The use of a bed of particles resembles an actual flotation system more than a flat mineral surface. Since then the concept of induction time and the equipment used to measure the induction time has advanced and commercialised (Albijanic et al., 2011; Gu et al., 2003; Ye and Miller, 1989; Yoon and Jordan, 1991). The equipment was developed such that software is used to control bubble motion parameters as this does affect the nature in which the bubble interacts with the particle bed. Furthermore, the induction time apparatus is equipped

with a camera to visualise the bubble-particle interaction and detect attachment.

Studies often use induction time and attachment time interchangeably, however Albijanic et al. (2010) clearly defined the difference between the two terms; the induction time being the time it takes for the thinning of the film at the air–water and solid–water interfaces to critical thickness. While the attachment time is the time it takes for the film to thin to critical thickness, rupture, and the attachment of particle to air bubble with contact angles in equilibrium.

The attachment timer described by Aspiala et al. (2018) and October et al. (2019a, 2019b) named the Automated Contact Time Apparatus (ACTA) generates six bubbles and tests the entire length of the particle bed. Furthermore, the particles that have successfully attached to the bubbles are captured and can be further analysed for recovery, shape, size and composition. Thus, the ACTA allows for a more robust bubble-particle attachment probability measurement. These studies Aspiala et al. (2018) and October et al. (2019a, 2019b), have looked at the attachment probability of a mixture of hydrophilic and hydrophobic

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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 (Aspiala 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 Jordan, 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) showed that the effect of ions on mineral recovery varies based on the mineral type.

Therefore, this investigation studies the effect of water quality on bubble-particle attachment in the most fundamental way by bringing bubbles into contact with a particle bed and uses classical microflotation as a validation technique for the ACTA. This study also aims to understand 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 both allowed for the collection of particles and clear visibility of particles on the images (October et al., 2020). The fraction below 38 μm was used for the zeta potential (i.e. electrokinetic potential) measurements as particles for this measurement must be fine enough such that some are still suspended in solution. The fraction between 38 and 75 μm was used for microflotation tests and adsorption studies (Castelyn, 2012; Mhlanga, 2011). The samples for each of the respective techniques, per mineral were split using a rotary splitter and placed into smaller plastic bags. Each bag was purged with nitrogen and stored below 30 °C. It is noted that the different particle size fractions used across the experimental techniques may be a limitation as particle properties may potentially be affected by particle size.

Table 1

Ionic composition of various water types.

Plant Water Type	Ca ²⁺ (mg/L)	Mg ²⁺ (mg/L)	Na ⁺ (mg/L)	Cl ⁻ (mg/L)	SO ₄ ²⁻ (mg/L)	NO ₃ ⁻ (mg/L)	CO ₃ ²⁻ (mg/L)	TDS (mg/L)	Ionic Strength (mol/L)
1 SPW	80	70	153	287	240	176	17	1023	0.0241
5 SPW	400	350	765	1435	1200	880	85	5115	0.1205
10 SPW	800	700	1530	2870	2400	1760	170	10,230	0.241

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 is 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 Aspiala 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.

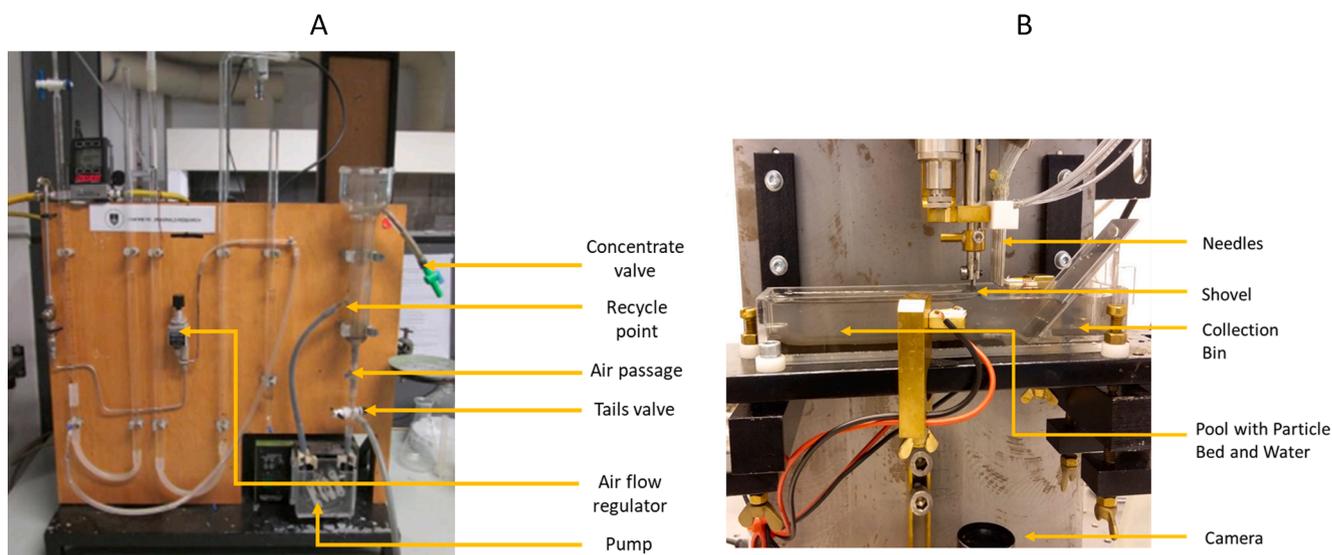


Fig. 1. Bubble-particle attachment equipment (A) Microflotation Cell and (B) ACTA.

2.6. Adsorption tests

Five standard solutions ranging from 0.5 mg/L to 5 mg/L of SIBX were made up. The absorbance of each solution was measured in a quartz cuvette using the Ultrospec 2100 UV-vis spectrophotometer (Biochrom, USA) across the 200 to 500 nm range; step size was 1 nm and spectral bandwidth < 3 nm. The peak in absorbance was observed to be at 301 nm; the 5 absorbances at this wavelength were plotted against the concentration of SIBX to obtain a calibration curve.

In a conical flask, 3 g of mineral was mixed with 30 mL of the water quality under study. The pH of the slurry was then adjusted to 6.5 using the NaOH and HCl solutions. The equivalent of 50 g/t of SIBX (15 μ L) was added to the mineral mixture; thus, the maximum xanthate concentration in the solution was 5 mg/L. The conical flasks containing the mineral mixture was covered with foil and inserted in the Ecobath shaker at 20 °C with shaking intensity of 141 rpm. After 15 min in the water bath a plastic syringe was used to draw out 10 mL of slurry. A 0.45 μ m filter was attached to the end of the syringe and the solution was filtered. The filtrate was placed into a quartz cuvette, which was inserted into the UV-Vis spectrophotometer where the absorbance at 301 nm was measured. Measurements were corrected for the blanks for each of the solutions.

The equation of the calibration curve obtained and the absorbance at 301 nm was used to calculate the concentration of SIBX left in the solution and by mass balance the concentration adsorbed on the mineral surface. This technique was applied to each of the water types with each condition tested in duplicate to minimise experimental error.

2.7. Zeta potential measurements

0.075 g of mineral sample was mixed in 100 mL of the water quality under study and placed on a magnetic stirrer for 5 min. The 100 mL suspension was evenly divided into 6 containers. The pH was adjusted to 6.5 using HCl and NaOH in order to match the pH at which the Microflotation and ACTA experiments were conducted. Each container was placed on the magnetic stirrer and the pH was measured again and readjusted if needed. The particles in each container were allowed to settle for 3 min and the particles suspended in the liquid were removed with a plastic pipette and inserted in the Malvern Dip Cell (Malvern Instruments Ltd., Malvern, UK). The dip cell was placed in the Zeta sizer Nano ZS90 (Malvern Instruments Ltd., Malvern, UK) and the zeta potential was measured. This procedure was followed for each water quality with each condition tested in triplicate in order to minimise

experimental error.

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 chalcopyrite is thus considerably higher compared to galena whose highest attachment probability was below 50%.

Microflotation of galena and chalcopyrite results are displayed in Figs. 3 and 4 respectively. The solid curve represents a system with xanthate collector and the dotted curve in the absence of a collector. The tests conducted with the ACTA were in the presence of the collector and thus their trend can be compared to the microflotation testwork with collector. Fig. 3 shows that in the first 4 min of flotation, the recovery 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 microflotation tests qualitatively match the trend observed in the ACTA in terms of mass recovered. However, it must be noted that quantitatively 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

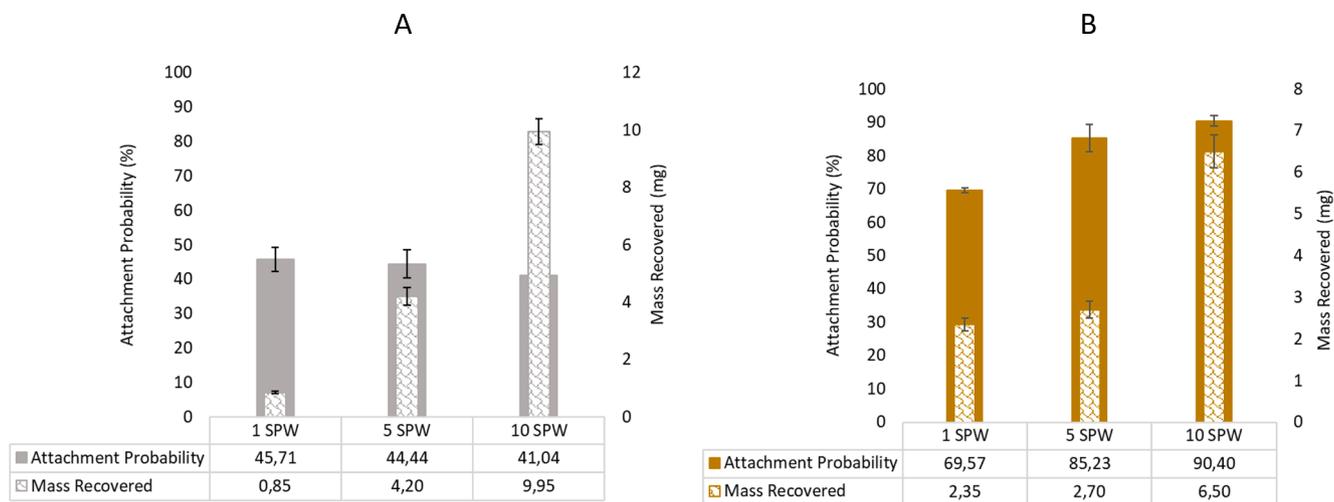


Fig. 2. Attachment Probability and Mass Recovered under varying water quality for (A) galena and (B) chalcopyrite with ACTA (Error bars represent standard error of the mean).

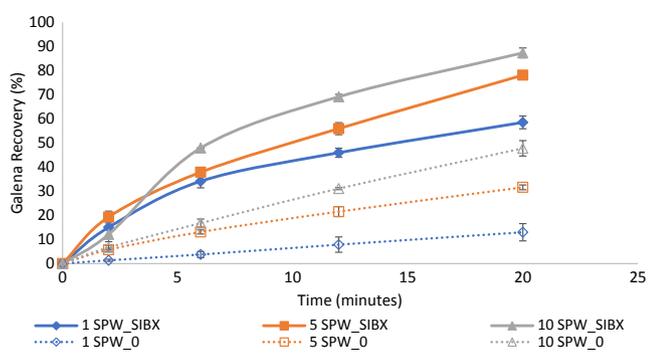


Fig. 3. Recovery of galena under varying water quality with and without collector by microflotation.

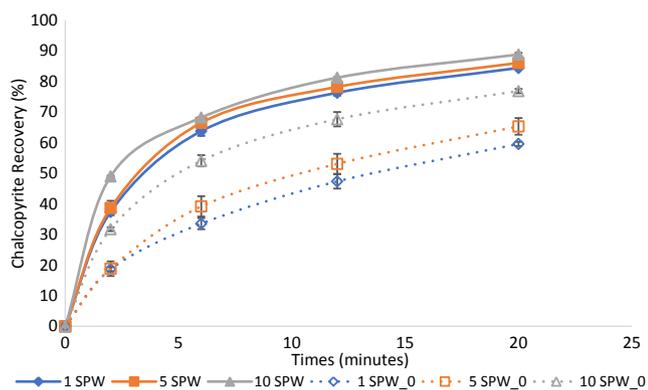


Fig. 4. Recovery of chalcopyrite under varying water quality with and without collector by microflotation.

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

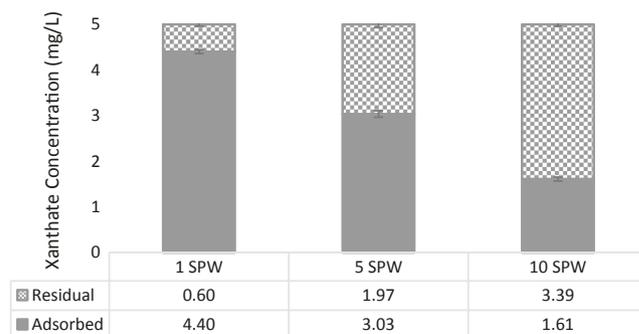


Fig. 5. Collector concentration adsorbed on galena surface and remaining in solution.



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

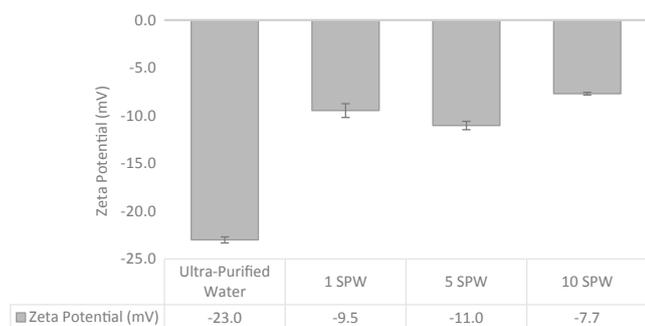


Fig. 7. Zeta potential of galena under varying water quality at pH 6.5.

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.

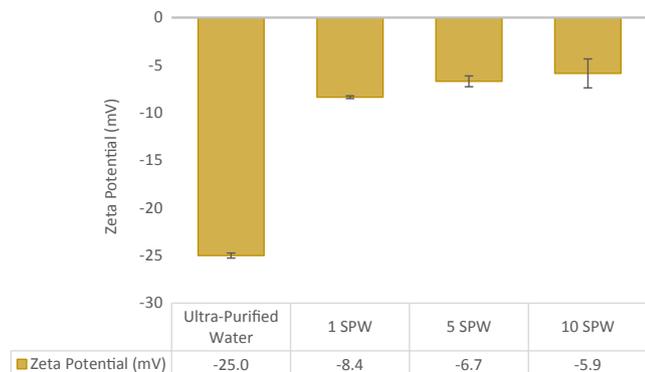


Fig. 8. Zeta potential of chalcopyrite under varying water quality at pH 6.5.

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 oxyhydroxyl, 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 adsorbs 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

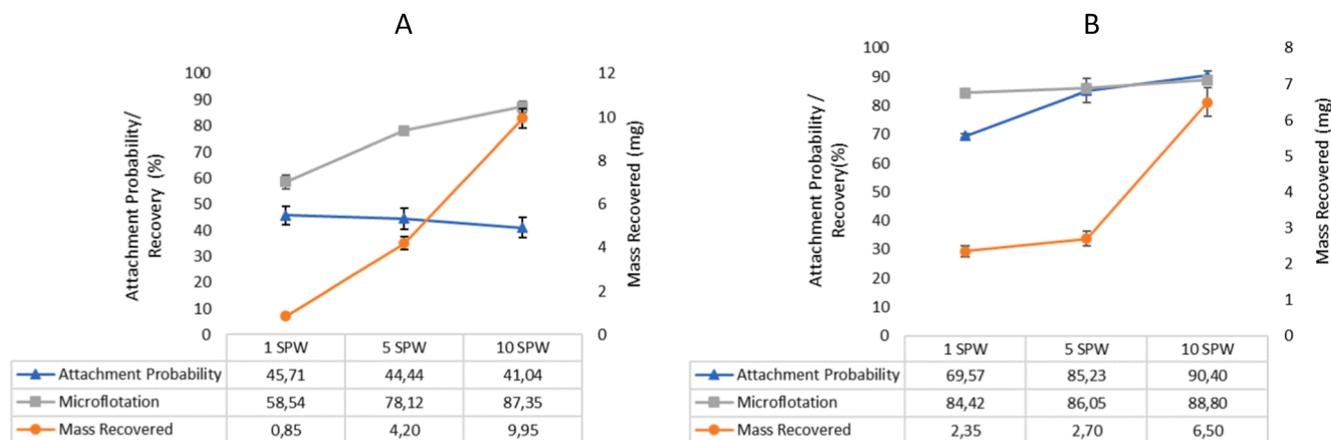


Fig. 9. Trend comparison of the microflotation and ACTA output (A) galena and (B) chalcopyrite.

chalcopyrite. This result is further reinforced by the high xanthate adsorption on the chalcopyrite surface regardless of the water quality. Kinetics studies by Mustafa et al. (2004) showed that equilibrium of xanthate adsorption on chalcopyrite is established within ten minutes. It may thus be for this reason the effect of xanthate adsorption is more evident on the galena surface than with chalcopyrite.

A difference in the chalcopyrite recovery is however clearly evident in the absence of a collector. Thus, also suggesting that there is another mechanism having a significant impact on the 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 the ionic 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.

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 more compact and less labour intense due to its automation.

In terms of the effect of water quality on bubble-particle attachment,

both microflotation and ACTA showed that the bubble-particle attachment increased with increases in the concentration of inorganic electrolytes in plant water. Both chalcopyrite and galena showed an increase in zeta potential as the concentration of inorganic electrolytes in the synthetic plant water increased. In terms of xanthate adsorption, an obvious decrease in xanthate adsorption on the galena surface was noted as the ionic strength of the plant water increased, while the xanthate adsorption on chalcopyrite seemed unaffected by the water quality.

The increase in zeta potential of the mineral particles as the ionic strength of water increases as seen in the experimental results may potentially be as a result of electrical double layer compression as shown in previous studies or charge neutralisation. EDL compression is known to ultimately lead to an increase in bubble-particle attachment; meaning that although less xanthate was adsorbed on the galena surface with deteriorating water quality, the effect of the increase in zeta potential of the mineral surface appeared to override this adsorption.

Furthermore, the galena-10 SPW system showed a large concentration of unabsorbed xanthate. Thus, a lower xanthate concentration should be considered in future studies to ascertain whether the same effect of increasing ionic strength of water results in lower collector adsorption on the mineral surface.

CRediT authorship contribution statement

L.L. October: Formal analysis, Writing - original draft. **M.S. Manono:** Conceptualization, Formal analysis, Supervision, Writing - review & editing. **J.G. Wiese:** Conceptualization, Supervision. **N. Schreithofer:** Formal analysis, Funding Acquisition, Resources, Supervision, Writing - review & editing. **K.C. Corin:** Conceptualization, Formal analysis, Funding acquisition, Resources, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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