# Galvanic Interaction of Pyrite with Cu Activated Sphalerite and its Effect on Xanthate Adsorption

Liuyin Xia,[1,3]* Brian Hart,[1] Faïçal Larachi [ORCID][2] and Olivier Gravel[2]

1. Surface Science, Western University, London, ON, N6G0J3, Canada

2. Department of Chemical Engineering, Laval University, QC, G1V 0A6, Canada

3. Mining and Minerals Division, Saskatchewan Research Council, SK, S7N 2X8, Canada

In the presence of Cu ions, a packed bed electrochemical reactor (PBER) was employed to deliberately avoid or induce galvanic coupling between pyrite and Cu-activated sphalerite. The effect of galvanic interaction on Cu ions uptake and xanthate adsorption were investigated. Solution chemistry and surface chemistry studies (ethylenediaminetetraacetic acid extraction and time of flight secondary ion mass spectrometry) have observed that when sphalerite and pyrite were galvanically coupled, Cu ions migrated from the pyrite surface to the surface of the sphalerite. Along with the marked decrease in the adsorption of Cu ions on pyrite, xanthate adsorption on the minerals also dramatically dropped. The pseudo-adsorption rate constant for the minerals in the mixed mode is only $0.0583 \, s^{-1}$, much less than that in the decoupled mode, which is $0.1368 \, s^{-1}$. This testing program shows that the galvanic coupling of minerals contributes to more copper transfer and Cu ions preferentially adsorbed by sphalerite rather than pyrite. This affects the pyrite surface and causes it to become xanthate unflavoured.

**Keywords:** sulphide ores, flotation, Cu activation, galvanic interaction

## INTRODUCTION

The adsorption of copper species from the solution to mineral surface to activate its surface for hydrophobic collector uptake is an important step in ore flotation. This activation process and the emergence of activation products on the minerals' surface have been studied for decades. For a detailed discussion regarding the copper activation of sulphide minerals, the authors refer the reader to some excellent review articles.[1–3] Among these sulphide ores, the most common example is using copper to activate sphalerite.[4,5] The surface of sphalerite makes xanthate attachment difficult because Zn-xanthate is easily removed from the mineral surface as soluble species in slurry. Cu ions as activator that either can be adsorbed on the surface of sphalerite or can replace the Zn that stays in the lattice of the sphalerite, significantly improving the reactivity of sphalerite for xanthate.[5–10] The activation flotation of sphalerite is normally carried out in a mixed mineral system (such as in a plant). This raises the possibility of unwanted co-activation of pyrite as a result of the introduction of Cu ions into the slurry, added as copper sulphate.[11–14] The major concern of this process was that the inadvertent activation would contribute to the recovery of pyrite, leading to the misplacement of pyrite to zinc concentrates.[15,16]

In contrast to copper accidentally activating pyrite during sphalerite activation, it has been shown that the pyrite flotation response was depressed when the system contained both Cu ions and sphalerite. Zhang et al.[17] performed activated pyrite flotation in the presence and absence of sphalerite and reported that the activated pyrite flotation was depressed significantly in the presence of sphalerite at all pH values. Xu[18] and Dichmann and Finch[19] both confirmed similar pyrite depression in mixed pyrite and sphalerite flotation in laboratory batch testing and plant mini-cell experiments. Dichmann and Finch[19] further investigated the role of copper ions in the selective separation of sphalerite and pyrite. There is a competition between sphalerite

and pyrite, first for copper ions and then for xanthate collector. Both Cu ions and xanthate favour sphalerite over pyrite.

Understanding the process can lead to an improvement in minerals separation and metals recovery. During Zn flotation, it is common to either add lime in an attempt to ensure that the pyrite is well depressed before the addition of the activating Cu ions, or to add the lime and copper at the same time for simplicity.[20] These traditional methods were optimized by changing the order of the reagents addition when the metallurgical process was understood.[21] It was suggested that Cu should be added first since the sphalerite would absorb it preferentially and since the copper favours sphalerite over pyrite. At this point, an increase in the pH due to the presence of lime may prevent the Cu from assembling on the sphalerite by forming Cu or Fe hydroxyl species.[22,23] Therefore, the addition of lime to elevate the pH should be done later. Altering the reagents order has been tested at two mining operations in Canada, Agnico-Eagle's Laronde and Noranda's Matagami-Bell Allard. The results showed the beneficial effect of the sequence $CuSO_4$-lime over the alternative. In the Matagami plant, the Fe assay in the final Zn concentrate was reduced by nearly half.[21]

Multilevel testing has confirmed the occurrence of pyrite depression in the presence of sphalerite after copper activation. Presently, further work is needed in order to develop an understanding of the process because the galvanic effects on copper activation/depression in mixed sphalerite and pyrite flotation remain unclear.[24,25] While previous researchers specu-
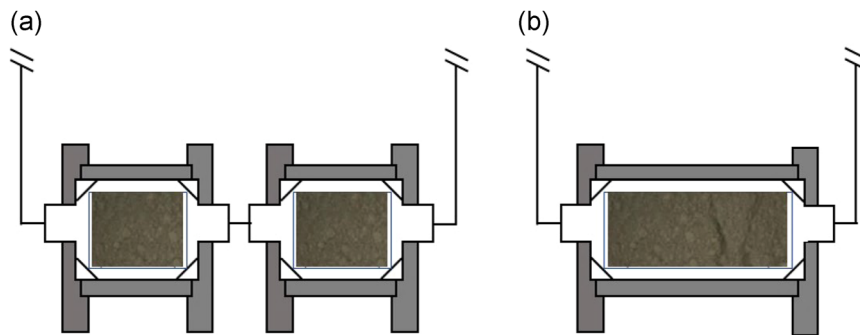
**Figure 1.** Sketch of packed-bed electrochemical reactor (PBER) strategies used to study the effect of galvanic interaction on sphalerite and pyrite flotation: (a) pyrite and sphalerite individually decoupled; and (b) pyrite and sphalerite physical contact.

lated that the galvanic coupling between the activated sphalerite and pyrite would not be strong yet did not sufficiently study this concept.

A packed-bed electrochemical reactor (PBER) was adopted and tested. It was filled with homogeneous pyrite and sphalerite, either directly through physical contact or separately in two chambers. Xanthate adsorption was monitored after the activation of copper ions. The surface chemistry of sphalerite and pyrite in all of the tests was measured by ethylenediaminetetraacetic acid (EDTA) extraction and time of flight secondary ion mass spectrometry (ToF-SIMS) analysis. With these tests, this work aims to investigate the effect of galvanic coupling between pyrite and Cu-activated sphalerite on the adsorption of xanthate on pyrite.

## EXPERIMENTAL

### Materials and Reagents

Pure pyrite and sphalerite were received from Ward's Natural Science. The ore was crushed, dry ground, and sieved to obtain a size finer than 75 μm and coarser than 38 μm for experiments. The prepared pyrite and sphalerite samples were analyzed by in-house SEM/EDS. The EDS data indicated that the pyrite was pure (>98 wt%) and the sphalerite sample possessed a dominant proportion of sphalerite (~90 wt%). The reagents used in this study were NaOH, HCl, $CuSO_4.5H_2O$, and EDTA and were all from certified reagents retailers such as Sigma-Aldrich Canada or Fisher Scientific Canada. Xanthate (potassium ethyl xanthate abbreviated as PEX) from the metallurgical operation was commercial products grade. De-ionized water was used for the preparation of all of the solutions and adsorption experiments.

### Equipment and Procedures

To investigate the galvanic effect on pyrite and sphalerite flotation, a packed-bed electrochemical reactor (PBER) was adopted. Prior to loading the minerals into the PBER, the dry prepared samples were rinsed using acid solution (1 wt% HCl) and cleaned using sonication in a DI water bath. The clean pyrite and sphalerite particulates were added to the PBER using either the individually decoupled mode (the two minerals were not directly in contact and only shared the circulating solution), shown in Figure 1a, or physically in contact (the two minerals were fully mixed), as shown in Figure 1b. 80 mL of the xanthate solution (200 mg/L) was continuously circulated in a closed loop through the fixed bed using a peristaltic pump. The flow rate was controlled at 0.05 mL/s for all of the tests. The sketch of the

adsorption setup is shown in Figure 2. The concentration of the xanthate was in-situ analyzed with a UV spectrophotoscopy. The amount of xanthate adsorbed on minerals at a time (t), Q, was calculated by the difference between the initial xanthate concentration ($C_0$) and the instantaneous xanthate concentration (C), as in Equation (1), where D is the adsorbent dosage (g/L):

$$Q \text{ (mg/g)} = \frac{C_0 - C}{D} \tag{1}$$

After adsorption testing, the pyrite and sphalerite particulates were removed from the PBER and were immediately introduced into the fore-vacuum chamber of the ToF-SIMS for the surface chemistry study.

Prior to xanthate adsorption, 25 mg/L $CuSO_4$ was circulated in the closed loop for 15 min for the activation of the minerals. After the activation of the Cu, the concentration of the metal ions in the circulated solution was analyzed using inductively coupled plasma mass spectrometry (ICP-MS). The PBER solids discharge was extracted by the EDTA by placing the 2 g pyrite into the EDTA solution (1 g/L) and conditioning the solution for 10 min. The EDTA extraction solution was preserved using 2 % $HNO_3$ for elemental analysis. The data was reported by the amount of metal ions extracted per unit mass of dry solids, $E_m$, (Table 1).
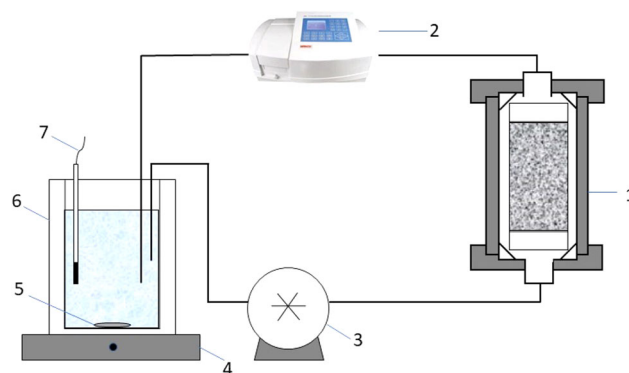


**Figure 2.** Adsorption testing setup with PBER, peristaltic pump, in situ UV spectrometer, and pulp potential measuring probe. Legend as follows: 1 PBER working section (Teflon); 2 UV-visible spectrophotoscopy; 3 peristaltic pump; 4 magnetic stirrer; 5 magnetic stirrer bar; 6 solution container; and 7 oxidation and reduction potential probe.

**Table 1.** Metal ions on pyrite surface after Cu activation in the presence and absence of sphalerite

| | The amount of extract metal ions (mg/g pyrite) | | |
| --- | --- | --- | --- |
| | Fe | Cu | Zn |
| 2 g Pyrite + 2 g Pyrite | 0.0554 | 0.0181 | 0.0000 |
| 2 g Pyrite + 2 g Sphalerite | 0.0520 | 0.0014 | 0.0066 |

The pulp potential was monitored by an oxidation reduction potential (ORP) sensor during adsorption as a function of adsorption time.

The surface chemistry measurements were performed using an ION-TOF, TOF SIMS IV™ secondary ion mass spectrometer to analyze the outer-most layer of surface of minerals. A pulsed liquid metal $Bi^{3+}$ primary ion beam operating at 25 kV was used to ionize species from a sample surface and at least six representative regions on each sample were examined. The intensities of the surface components for regions of interest as positive and negative secondary ions are normalized by the total ion intensity (counts of the recorded total mass spectrum), and further normalized data was expressed by the mean and median values with box plots, as shown in Figure 3.

## RESULTS

### Cu Ions Adsorbed on Pyrite

The PBER chamber was in individually decoupled mode, as shown in Figure 1a. It was added either with 4 g pyrite equally divided into two cones or with 2 g of pyrite and 2 g of sphalerite for each cone yet sharing the solution. After activation, the pyrite surface metal ions were extracted by the EDTA and further analyzed using the ICP-MS. The EDTA extraction data provides a general comparison between Cu ions on the surface of the pyrite and the presence of sphalerite, as shown in Table 1. A marked decrease in Cu ions on the pyrite surface was shown when sphalerite was present. Previously, Zhang et al.[17] and Dichmann and Finch[19] made essentially the same observation.

Copper activation of pyrite does not involve the ion exchange of Fe with Cu. Therefore, Cu is simply physically adsorbed and is readily solubilized by the EDTA. In the meantime, there is only a slight chance of Cu ions converting to copper hydroxides, which prevents EDTA extraction, because the x-ray photoelectron spectroscopy (XPS) study showed little evidence of copper hydroxides on the mineral surface in comparison to other metal ions such as Fe, Zn, and Pb.[26] The lower copper level in the presence of sphalerite is evidence of a shortage of copper on pyrite surface.

### Effect of Galvanic Interaction

The PBER chamber was filled in two ways, shown in Figure 1a and b, either with pyrite and sphalerite decoupled or physically in contact. After the Cu activation, the concentration of the metal ions in the circulated solution was analyzed by ICP-MS. The results are revealed in Figure 4.

When minerals were in direct contact and tightly packed in the PBER (mixed mode), the galvanic interaction, if any, would occur. The data in Figure 4 shows a significant increase in the concentration of Cu ions in the circulating solution when galvanic coupling occurred. This indicates that as a result of galvanic interaction, the adsorption of Cu ions onto pyrite is dramatically depressed, and thus the ions that should have been attached to the surface of the minerals could not attach and were detected in the solution. On minerals surface, there should have a significant reduction of the copper adsorption. The transfer of Cu ions from the mineral surface back to the solution was mainly occurred on the pyrite surface. This was demonstrated by the fac that the Zn ions concentration remained unchanged, shown in Figure 4.

According to the accepted theory of the Cu activation of sphalerite, Cu ions can be incorporated into the sphalerite matrix. Meanwhile, Zn ions displaced by Cu would be released into the solution. Theoretically, the amount of Zn ions is almost equal to the amount of Cu that is displaced. In this circumstance, any changes by Cu ions on the sphalerite surface would cause corresponding changes in the Zn ions in the solution. As shown in Figure 4, however, the concentration of Zn ions in the two modes testing is almost identical (the difference is smaller than 5 %). This indicates that the Cu ions adsorbed on the sphalerite has maintained the same level prior to and after the galvanic coupling. Thus, we conclude that the galvanic interaction between Cu-activated sphalerite and pyrite significantly affect the Cu adsorption on the surface of pyrite rather than sphalerite.

Following copper activation, xanthate was introduced into the circulation using two PBER testing modes, decoupled and mixed.
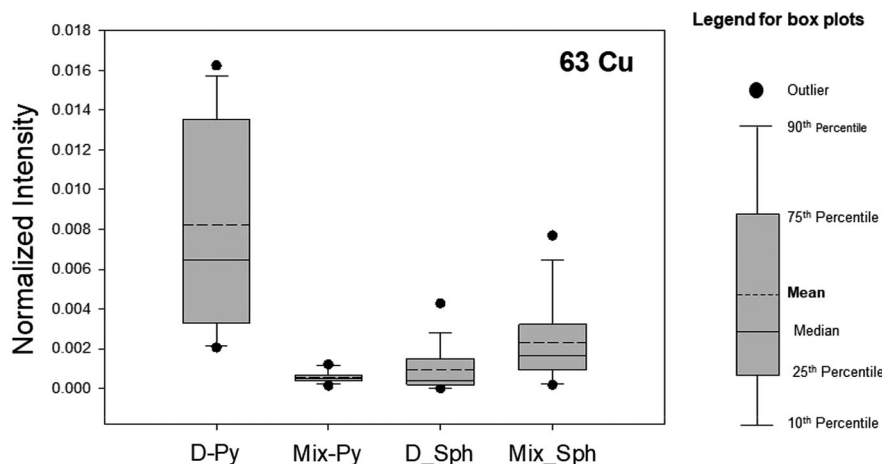


**Figure 3.** An illustration of a box plot showing the various components that are discussed throughout the test.
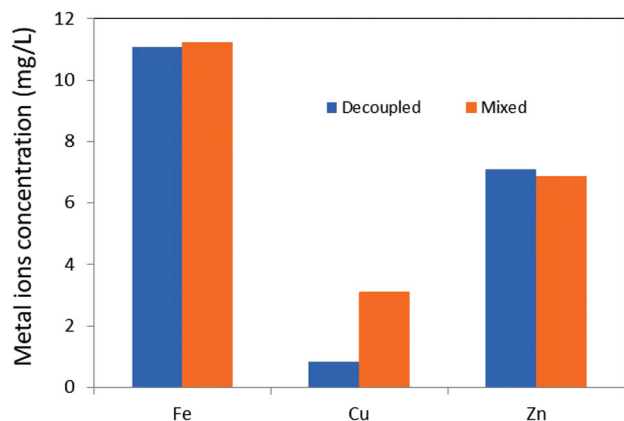
**Figure 4.** The concentration of metal ions in testing circulating solution.

A sketch of the circulation is shown in Figure 2. The amount of xanthate that was adsorbed on the minerals was calculated based on the initial xanthate concentration and the in-situ measured value. The kinetic sorption of the xanthate on the surface of the minerals is shown in Figure 5. There is a logarithmic increase in the adsorption capacity as a function of adsorption time. To express the adsorption of xanthate onto the surfaces of the ores, the pseudo-first order adsorption kinetic model was verified. The kinetic sorption equation is given in Figure 5. According to the curve fitting results, the decoupled testing showed an $R^2$ of 0.9989 and a pseudo-adsorption rate constant of $0.1368 \, s^{-1}$, while the mixed testing showed an $R^2$ of 0.9717 and its pseudo-adsorption rate constant is $0.0583 \, s^{-1}$, much smaller than in decoupled mode. When pyrite and sphalerite was in the direct contact mode, the xanthate adsorption was significantly slower.

Results shown in Figure 6 reveal the measurement of an oxidation reduction potential (ORP) sensor during adsorption as a function of adsorption time. The ORP data decreased in the first 15−20 min for the decoupled testing, indicating that dominated surface oxidation products formed within this time, whereas the formation time for the oxidation products for the mixed testing was ∼30 min. The fact that the oxidation products took longer to form indirectly demonstrates the lower xanthate adsorption rate
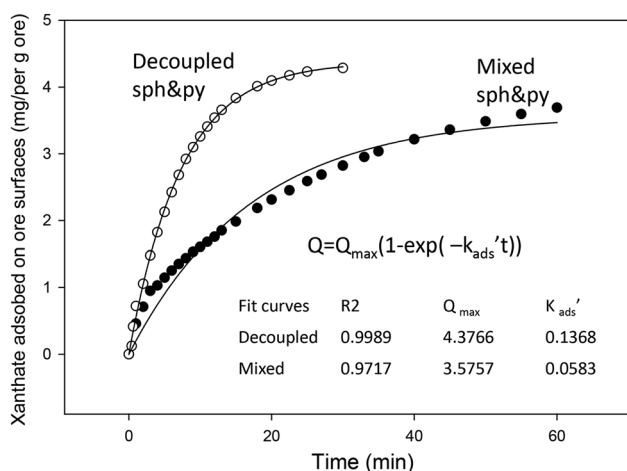


**Figure 5.** Amount of xanthate adsorbed on minerals surface as a function of adsorption time in two testing ways, pyrite and sphalerite decoupled or pyrite and sphalerite mixed (initial xanthate concentration was 200 mg/L).
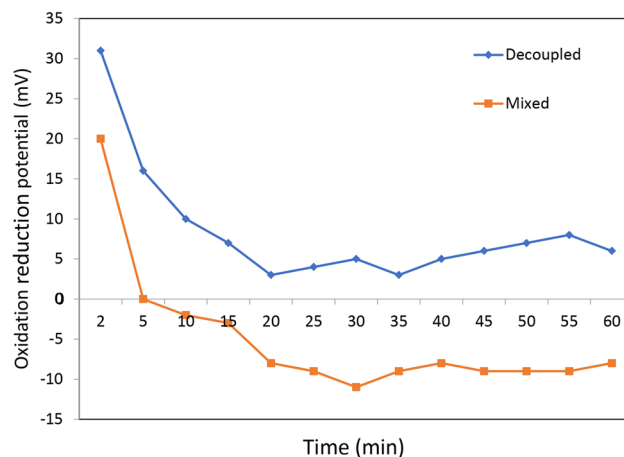


**Figure 6.** Measurement of an oxidation reduction potential (ORP) sensor during adsorption as a function of adsorption time.

after the galvanic coupling of minerals because the dominating oxidation products during xanthate adsorption would be xanthate oxidase species such as xanthogen or dixanthogen. In other words, The ORP measurement indicates that the mixed minerals adsorbed the xanthate slower than they did in the decoupled mode. The ORP value remains ∼15 mV higher during the decoupled testing than during the direct contact testing. This indicates ore mixture resulting in less oxidizing environments. The galvanic interaction between the minerals consumes more oxygen in the system.

### Surface Chemistry of Pyrite and Sphalerite

The limitation of EDTA extraction when studying surface metal ions lies in the displacement of Zn by Cu on the surface of the sphalerite, where Cu ions are incorporated into the matrix of the sphalerite. They are not extractable by the EDTA. In this paper, mineral surface chemistry was analyzed by time of flight secondary ion mass spectrometry (ToF-SIMS), which has a surface sensitivity superior to that of XPS. This technique allows for the analysis of the outermost 1−3 atomic layers of a surface by mass spectrometry, including the chemisorbed monolayer of xanthate, and the multilayer Cu species that forms during activation.

Figure 7 shows the normalized intensities for four of the diagnostic peaks that indicate the collector xanthate (PEX) adsorption on the surfaces of the sphalerite and pyrite. It reveals that with the direct contact of sphalerite and pyrite, the galvanic coupling results in a marked decrease in xanthate on the surface of the pyrite, as shown in Figure 7.

In addition to the collected xanthate adsorption information, the adsorption of Cu ions on the surfaces of the minerals was also examined (Figure 8). The ToF-SIMS provides valuable information in addition to that obtained from the solution chemistry, as mentioned in Figure 4. Massive positive and negative secondary ions spectra were found, yet only a few are shown in Figure 8. The authors encourage readers interested in seeing more secondary ions on the surface of sphalerite and pyrite to contact the authors. Evidence from Figure 8 supports the copper migration from pyrite to sphalerite due to the galvanic interaction. The copper species, including positive Cu and $Cu_2H$ and negative copper sulphate and oxides, all decreased in adsorption in the mixed mode relative to the decoupled mode. However, not only is there more copper uptake on the surface of the sphalerite when in direct contact but there is also a larger amount of Zn ions. This suggests either an
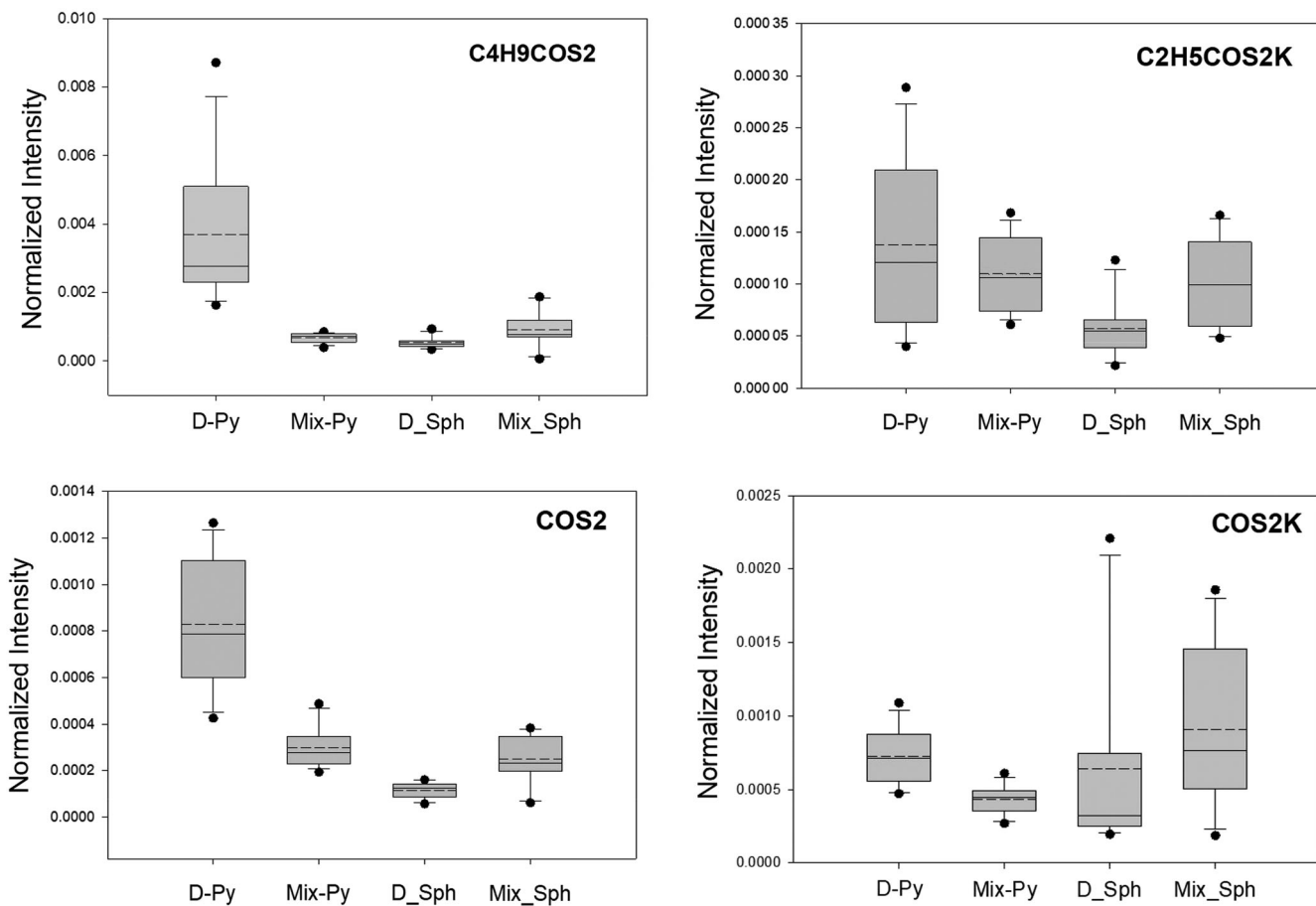
**Figure 7.** Normalized intensities for diagnostic peaks indicative of xanthate adsorption on sphalerite and pyrite surfaces (pyrite in decoupled mode was abbreviated as D-Py; pyrite in mixed mode was abbreviated as Mix-Py; sphalerite in decoupled mode was abbreviated as D-Sph; sphalerite in mixed mode was abbreviated as Mix-Sph).

increase in the Cu attachment on the surface of the sphalerite or the galvanic interaction emerging between the two contact minerals. In a reference article, Cu ions that were deliberately added to Kidd Creek ore slurry samples were not detected by EDTA extraction, yet an amount of Zn approximately equal to the amount of Cu added was found.[18] This indicated that the more copper that attaches on the surface of the sphalerite, the larger the amount of Zn found in the slurry. However, the unchanged concentration of of Zn ions in the slurry, as shown in Figure 4, does not support the ion exchange of Cu and Zn. It is shown that the galvanic interaction is only occured in the mixed mode.

Figure 9 shows the zinc and iron hydroxyl species on the surface of the pyrite and sphalerite. The copper hydroxyl species could be the copper sulphate oxidation products but are not listed here since the XPS study had shown very little amount of copper hydroxides on minerals surface relative to iron and zinc.[27,28] The surface of the pyrite grains have a higher proportion of ZnOH and FeOOH in the test with minerals in the mixed mode. The generation of hydroxyl species appears to be linked to the large amount of oxygen consumption in the ORP sensor measurement (Figure 6).

## DISCUSSION

In the presence of copper ions, we decided to either avoid or to induce galvanic coupling between sphalerite and pyrite, and we

studied the effect of the galvanic interaction on Cu ions uptake and xanthate adsorption. It is observed that when sphalerite and pyrite were galvanically coupled, Cu ions migrated from the pyrite surface to the sphalerite surface. Evidence of this can be seen from the solution chemistry analysis by ICP-MS (Figure 4) and the minerals surface analysis by ToF-SIMS (Figure 8). In addition to the marked decrease in the adsorption of copper on the pyrite surface, the xanthate adsorption on the minerals surface decreased significantly (Figure 7). Data shown in Figure 5 supports this observation since the adsorption rate constant for the two minerals mixed mode of testing is only $0.0583 \, \text{s}^{-1}$ and is much less than for testing in the decoupled mode, which is $0.1368 \, \text{s}^{-1}$.

The hypothesis about the competition for copper adsorption between sphalerite and pyrite, leading to pyrite depression in the presence of Cu ions, was studied many years ago. Dichmann and Finch[19] even designed an EDTA extraction testing program to verify the migration of Cu ions. They identified that the amount of EDTA-extractable Cu ions on the pyrite surface decreased with an increase in the contact time of the sphalerite. Copper uptake on the surface of the pyrite decreased by nearly 50 % after 30 min of contact. It was even hypothesized that, given time, all of the Cu ions could migrate to the sphalerite surface. In this testing program, the competition adsorption of copper was further developed. It appears that galvanic coupling between pyrite and the Cu-activated sphalerite enables more
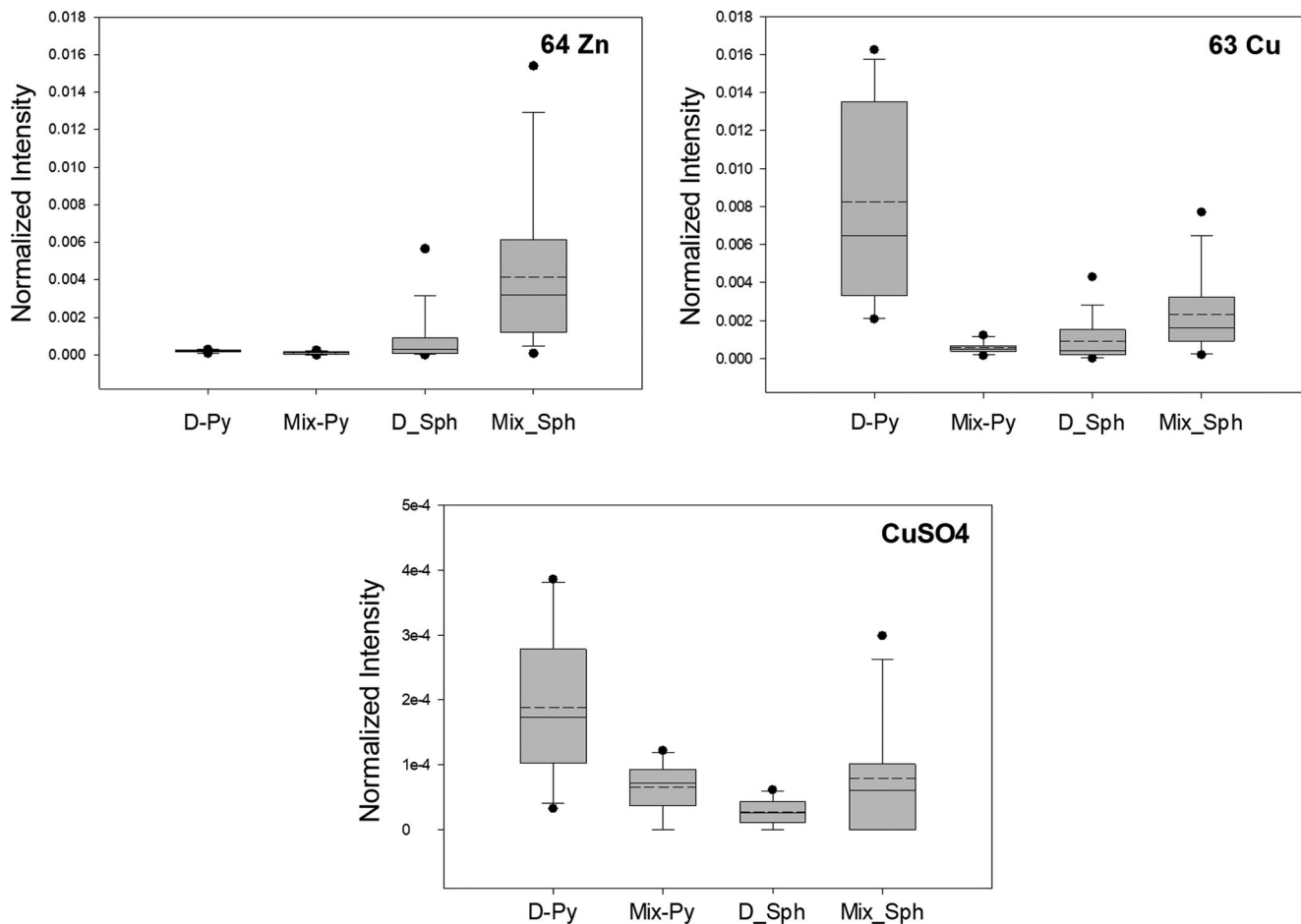
**Figure 8.** Normalized intensities for positive and negative peaks indicative of metal ions adsorption on sphalerite and pyrite surfaces.

copper migration and the Cu ions preferentially adsorb on the surface of the sphalerite. This causes the pyrite surface to become unflavoured xanthate.

In contrast to a shortage of copper ions, Dichmann and Finch[19] tested pyrite micro-flotation using an overdosage of copper ions and a large amount of collector. Under these extreme conditions, the recovery of pyrite remained high in the presence of sphalerite, suggesting that a competition between the sphalerite and pyrite for Cu ions is not evident when the copper was excessive. Returning to the test work in this paper, the concentration of Cu ions in the circulating solutions in two testing modes are shown in Figure 4. The minerals that were mixed resulted in more Cu ions present in the filtrate solution relative to the testing performed in the decoupled mode. In the mixed mode,
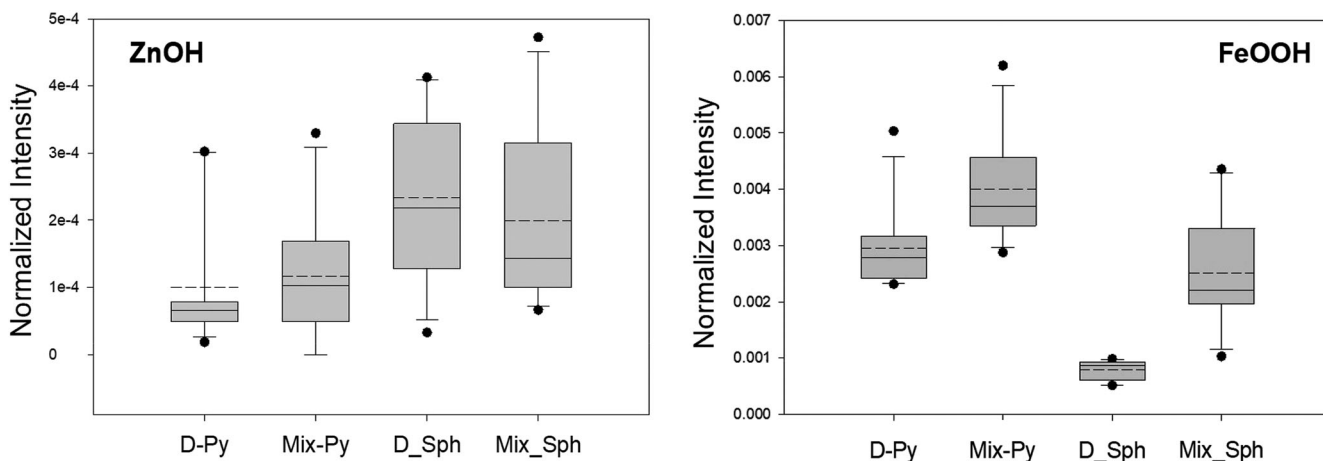


**Figure 9.** Normalized intensities for negative peaks indicative of Zn and Fe hydroxyl species adsorption on sphalerite and pyrite surfaces.

there was a certain amount of Cu ions (> 3 mg/L) in the solution. At this point, the sphalerite's needs for Cu ions had been satisfied. Cu ions in the solution could be adsorbed by the pyrite. Thus, it is unlike the shortage of copper for the pyrite. The galvanic interaction not only provides a reduction in the xanthate adsorption that is caused by competition for Cu ions but also provides metal hydroxyl species on the surface of the pyrite as a result of the electrochemical reaction products of oxygen. Therefore, as a result of the galvanic interaction, even if there is a certain amount of copper in the solution, it would not be adsorbed by the pyrite. Instead, the pyrite has a higher rest potential than the sphalerite and after coupling its surface becomes coated with OH products, resulting in the pronounced hydrophilic thus being depressed. This is demonstrated in the adsorption testing (Figure 5). An increase in the production of hydroxyl ions also leads to a high consumption of oxygen and then a decrease in the ORP value (Figure 6). The galvanic coupling of minerals may affect the adsorption of xanthate on pyrite in two ways. On one hand, the migration of Cu ions on the surface of the minerals decreases the attachment of the xanthate on the pyrite. On the other hand, as a result of galvanic coupling, oxygen is reduced to for the hydroxide ions on the surface of the pyrite because the pyrite has a higher rest potential than the copper activated sphalerite.[25,29,30]

## CONCLUSION

Galvanic coupling between Cu-activated sphalerite and pyrite affects the uptake of Cu ions on the surface of pyrite. It contributes to the preferential adsorption of copper by sphalerite and then controls the pyrite that is difficult to attract xanthate. In the presence of galvanic interactions, copper is not absorbed by pyrite even though a certain amount of Cu is present in the solution. The surface of pyrite becomes hydrophilic due to the generation of hydroxyl ions, resulting in the xanthate attachment becoming less responsive.

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