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**Improvement of flotation and suppression of pyrite oxidation using phosphate-enhanced galvanic microencapsulation (GME) in a ball mill with steel ball media**

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## **Abstract**

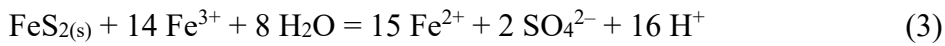
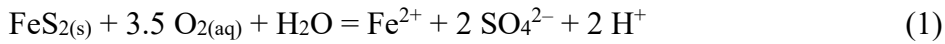
Acid mine drainage (AMD) is one of the biggest and most challenging environmental concerns of the mining and mineral processing industries. In our previous study, we reported a new and promising approach to passivate sulfide minerals and limit AMD formation called galvanic microencapsulation (GME), a technique that forms protective coatings via galvanic interactions between sulfide minerals (e.g., pyrite) and metal powders (e.g., zero-valent iron and aluminum). In this paper, the possible application of GME in a ball mill with steel ball media and phosphate to suppress pyrite floatability during coal cleaning and limit pyrite oxidation after disposal is proposed. The results showed that adding phosphate and maintaining sufficient oxygen supply during GME treatment in the ball mill passivated pyrite and suppressed its oxidation via the formation of protective iron phosphate coatings. Moreover, the iron phosphate coatings on pyrite made the mineral more hydrophilic, which improved the separation efficiency during coal flotation.

**Keywords:** Galvanic interactions, galvanic microencapsulation, ball mill, flotation, pyrite, passivation

## 1. Introduction

Large amounts of waste rocks and sediments containing sulfide minerals (e.g., pyrite) are generated not only from mining and mineral processing operations (Park et al., 2019; Tomiyama et al., 2019) but also in underground space development projects like tunnel construction for roads and railways, shopping malls, and parking spaces (Li et al., 2016; Tabelin et al., 2012a, 2014; Tamoto et al., 2015). When sulfide-rich waste rocks and sediments are exposed to the environment, acid-sulfate soils and acidic effluents generally referred to as acid mine drainage (AMD) are inevitably generated (Huyen et al., 2019a, b; Tabelin et al., 2009). AMDs are not only acidic ( $\text{pH} < 4$ ) but also typically contain high concentrations of toxic metalloids like arsenic (Suzuki and Katoh, 2019; Tabelin et al., 2012a, 2014; Tatsuhara et al., 2012) and hazardous heavy metals like copper, zinc and lead (Akcil et al., 2006; Barrie et al., 2005; Elsetinow et al., 2001; Sheoran, et al., 2006; Tabelin et al., 2012b; Yuting et al., 2015). AMD formation is initiated when pyrite reacts with water and dissolved oxygen (DO) as explained in Eq. (1) (Tabelin et al., 2017a, b). In addition to DO's direct role as an oxidant of pyrite, it also indirectly promotes pyrite oxidation and AMD formation by oxidizing  $\text{Fe}^{2+}$  to ferric ions ( $\text{Fe}^{3+}$ ) as illustrated in Eqs. 2 and 3 (Tabelin et al., 2018).  $\text{Fe}^{3+}$  is a far stronger oxidant of sulfide minerals than DO and its formation in the natural environment is promoted via the

mediation of microorganisms like Fe-oxidizing bacteria, a process that sustains AMD generation in many pyrite-rich waste rocks, sediments and tailings (Singer and Stumm, 1970).



Chemical neutralization is the most widely used technique to treat hydrometallurgical processing wastewaters including AMD (Park et al., 2019; Tabelin et al., 2019a). In this technique, basic materials such as limestone or lime are mixed with AMD to increase its pH and remove the bulk of dissolved heavy metals via precipitation as metal oxyhydroxides/oxides (Demers et al., 2015; Heviánková et al., 2013; Kleinmann et al., 1998). Although chemical neutralization is effective, AMD generation has been predicted and observed to persist for several centuries or even millenia, so this approach is unsustainable in the long-term because it requires continuous supply of chemicals, energy and manpower as well as the disposal of hazardous neutralization sludge (Davis et al., 2000; Igarashi and Sasaki, 2013; Tabelin et al., 2019b).

Among the numerous alternatives proposed for AMD management and control, microencapsulation strategies are promising and potentially sustainable because they limit AMD formation by suppressing sulfide oxidation directly by encapsulating acid-generating sulfide minerals like pyrite and arsenopyrite with an unreactive or passivating coating (Evangelou, 1995; Li et al., 2019a, b; Park et al., 2018a). In a recent study of Seng et al. (2019), a promising microencapsulation approach called galvanic microencapsulation (GME) was introduced that could be directly integrated into existing grinding circuits employed in mineral processing. In GME, sulfide minerals are coated with a protective layer of iron phosphate via galvanic interactions between sulfide minerals like pyrite and zero-valent iron (ZVI).

In this study, the possible use of a ball mill with steel ball media as dissolved Fe source for GME was investigated. Fig. 1 shows a schematic diagram of how GME in a ball mill containing steel ball media and phosphate would work. Because pyrite is the most common sulfide gangue mineral in the mineral processing of non-ferrous sulfide ores and is the main cause of AMD in tailings dams (Li et al., 2019b; Park et al., 2019b; Tabelin et al., 2013), passivation of this mineral during mineral processing would greatly limit AMD formation in the tailings dam after disposal. Moreover, iron phosphate phases are inherently hydrophilic, so their formation on pyrite could make this mineral less

oleophilic (i.e., strong affinity to oil) and could improve the separation of coal and pyrite during flotation.



Fig. 1. A schematic diagram of galvanic microencapsulation (GME) in a ball mill containing steel ball media.

## 2. Materials and methods

### 2.1 Materials

The pyrite sample used in this study was obtained from Huanzala Mine, Huanuco, Peru, and its chemical and mineralogical compositions were reported in the previous study of the authors (Seng et al., 2019). The sample is mainly composed of pyrite and contains 44% of Fe and 54.3% of S with only trace impurities like calcium (0.36%) and silicon (0.32%). For GME treatment in a ball mill, the pyrite sample was crushed (using

a jaw crusher and an agate mortar and pestle) and sieved to obtain a size fraction of 500–710  $\mu\text{m}$ .

## **2.2. GME treatment in a steel ball mill jar and leaching experiments**

Before GME treatment, the crushed pyrite sample was washed using the technique developed by McKibben and Barnes (1986) to remove any oxidized layer formed on pyrite during sample preparation and storage. The washing procedure is as follows: (1) ultrasonic cleaning in ethanol, (2) acid washing using 1 M  $\text{HNO}_3$ , (3) triple rinsing with deionized (DI) water (18  $\text{M}\Omega\cdot\text{cm}$ , Milli-Q<sup>®</sup> Integral Water Purification System, Merck Millipore, USA), (4) dewatering with acetone, and (5) drying in a vacuum desiccator. After this, GME treatment of washed pyrite (100 g) was conducted in a steel ball mill jar (inner diameter of 8 cm) with steel balls (average diameter of 0.6 cm) and 130 mL of either DI water or 50 mM of phosphate solution for 3 hours. The solution containing 50 mM of phosphate was prepared using reagent-grade  $\text{NaH}_2\text{PO}_4$  (Wako Pure Chemical Industries, Ltd. Japan), and its pH was adjusted by dilute HCl solution. After GME treatment, the steel balls were removed by hand and pyrite was washed six times with DI water.



The leaching experiments were conducted by mixing 1 g of pyrite GME-treated in DI water or phosphate solution with 10 mL of DI water in an Erlenmeyer flask and shaking the suspension up to 7 days in a constant temperature water bath shaker (25°C) at a shaking amplitude and speed of 40 mm and 120 strokes/min, respectively. After the pre-determined leaching time, the pH and redox potential (Eh) of suspensions were measured, and the leachates were collected by filtration through 0.2 µm syringe-driven membrane filters (Sartorius AG, Germany). Some of the leaching experiments were done in triplicates to ascertain that the observed trends were statistically significant. The concentrations of total S as  $\text{SO}_4^{2-}$  (dissolved S) and dissolved Fe were determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES, ICPE-9820, Shimadzu Corporation, Japan) (margin of error  $\pm 2\%$ ). Meanwhile, the residues were washed thoroughly with DI water and dried in a vacuum drying oven at 40°C for 24 hours and then analysed by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) (InTouchScope™ JSM-IT200, JEOL Ltd., Japan).

### **2.3 Flotation experiments**

Mixtures of 16 g of Kushiro coal (100–150 µm), 3 g of pyrite (with and without GME treatment, 100–150 µm) and 1 g of quartz (100–150 µm) were used in the coal

flotation experiments based on the modified release analysis method developed by Tsunekawa et al. (1998). Flotation tests were carried out in an Agitair-type batch flotation cell (FT-1000, Heiko, Japan) with 400 mL of suspension containing 20 g of coal-pyrite-quartz mixture, an impeller speed of 1000 rpm, and aeration rate of 0.75 L/min. The suspension was first conditioned for 3 minutes followed by the addition of 605 g/T of methyl isobutyl carbinol (MIBC) as the frother. Agitation was continued for 3 more minutes prior to the introduction of air for flotation. Froth products were recovered for 3 minutes without adding a collector, after which, 80 g/T of kerosene (collector) and 605 g/T of MIBC were added. The pulp was again conditioned for 3 minutes and froth products were collected after the introduction of air for 3 minutes. This procedure was repeated with 120, 200 and 200 g/T of additional kerosene for cumulative kerosene dosages of 80, 200, 400 and 600 g/T. The froth products were collected separately from each flotation stage and the ash value of the froth and tailings products were determined according to the Japanese Industrial Standards (JIS) (M8812).

### **3. Results and discussion**

#### **3.1 GME treatment in a ball mill and coating formation**

Preliminary GME treatment using a ball mill with steel ball media were carried out with and without phosphate and the results showed that dissolved Fe concentrations remained high (110 mg/L) even though the pH was around 5, suggesting that the supply of oxygen was not enough to oxidize  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  because the ball mill jar was completely sealed during treatment. To improve the procedure, another set of experiments were conducted with intermittent air introduction using an air pump to replenish the supply of DO needed for galvanic interactions,  $\text{Fe}^{2+}$  oxidation and iron phosphate precipitation. The results showed that dissolved Fe concentrations decreased below 10 mg/L with air introduction, suggesting that the supply of DO is crucial for iron precipitation during GME treatment in a ball mill. To evaluate whether this improved iron precipitation after air introduction translated to enhanced coating formation, leaching experiments of treated products were conducted.

GME-treated ball mill products were leached in DI water for up to 7 days to evaluate the suppression of pyrite oxidation, and the leaching results showed substantially lower dissolved S and Fe concentrations with phosphate and air introduction (Fig. 2). SEM-EDX observations of the leaching residues after 1 day also detected strong signals of O and P in the same areas on pyrite with weaker signals of S and Fe (Fig. 3). These results indicate that pyrite was coated with an Fe–P–O containing phases and GME could

be applied in a ball mill with steel ball media and phosphate provided that there is enough oxygen supply into the system (e.g., intermittent air introduction).

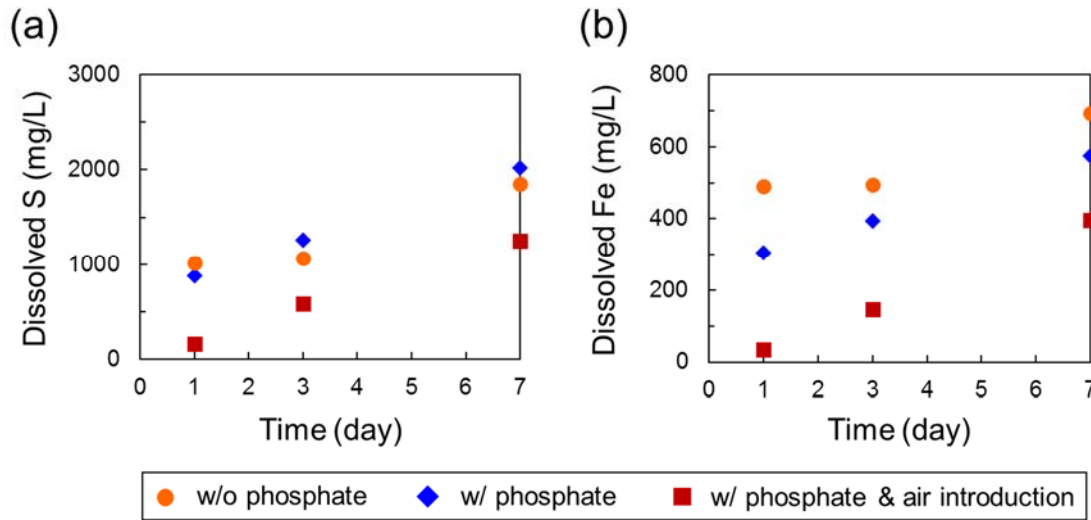


Fig. 2. Changes with time of (a) dissolved S concentration, and (b) final dissolved Fe concentration in the leaching experiments after GME treatment in a ball mill under various conditions.

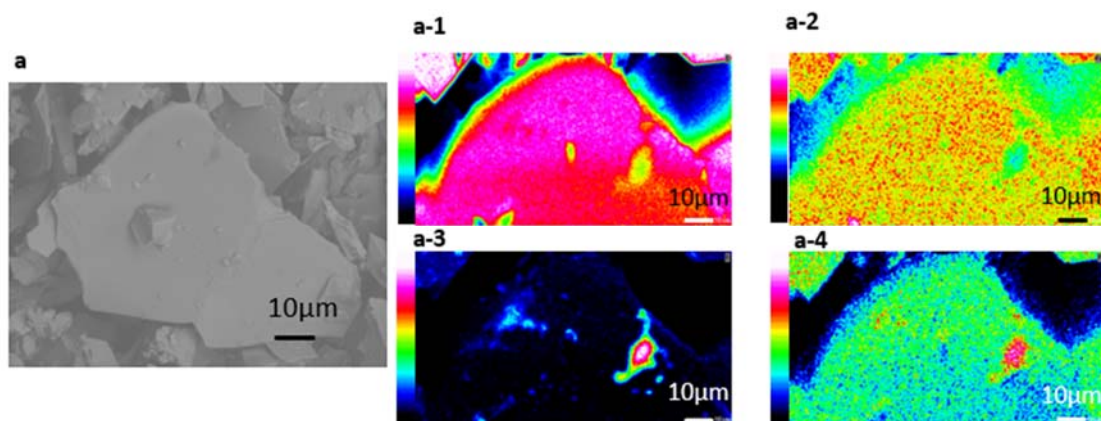


Fig. 3. (a) SEM-EDX photomicrograph of pyrite after leaching for 1 day with DI water and the corresponding elemental maps of (a-1) S, (a-2) Fe, (a-3) O and (a-4) P.

### **3.2 Flotation of coal-pyrite-quartz mixtures with and without GME treatment**

The results of coal flotation (combustible or ash recovery vs yield) are plotted in Fig. 4. The Kushiro coal sample, which contains organic matter and gangue minerals, was mixed with pyrite and quartz, so after ashing, the ash is composed of substances from not only the gangue minerals of Kushiro coal but also those of pyrite and quartz, which were added to the coal sample. For a simple interpretation of the results of release analysis, a ‘hatch-lined’ area is drawn between the combustible and ash recovery curves and when this area becomes smaller, it means that more gangue minerals (e.g., pyrite) are recovered in froth products (Tsunekawa et al., 1998). The results showed that the ‘hatch-lined’ area was smaller without GME treatment (Fig. 4a) than that with GME treatment (Fig. 4b), suggesting that the recovery of pyrite to the froth decreased. This improvement in coal flotation recovery could be attributed to the hydrophilic coating on pyrite after GME that limited the attachment of GME-treated pyrite to bubbles (Fig. 3). Based on these results, GME treatment of pyrite using a ball mill not only passivated pyrite and limited its oxidation but also improved the separation efficiency of coal and pyrite during flotation.

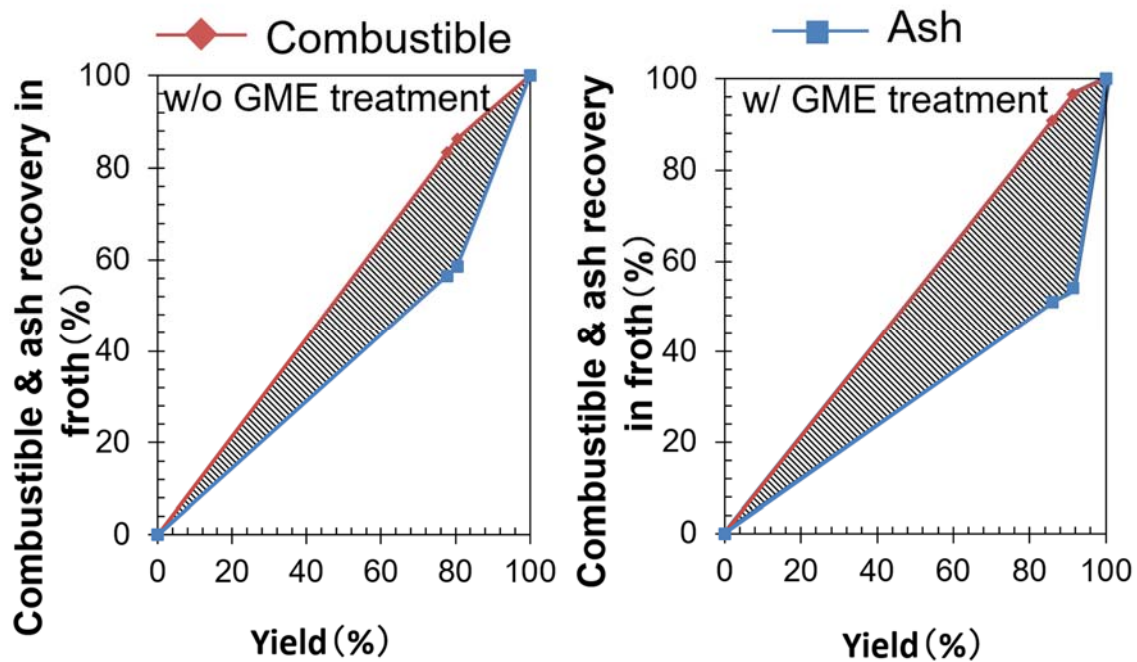


Fig.4. Combustible and ash recovery of coal (a) without GME treatment, and (b) with GME treatment.

#### 4. Conclusions

In this study, GME treatment of pyrite in a ball mill with steel ball media and phosphate was investigated. Iron phosphate coatings were formed on pyrite that passivated the mineral and limited its oxidation. For the coating to form, sufficient supply of oxygen was crucial during GME treatment. Furthermore, the hydrophilic iron phosphate coating on pyrite improved the separation efficiency by flotation of a coal-pyrite-quartz mixture.

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