



Formation of hydrogen peroxide by pyrite and its influence on flotation



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ABSTRACT

Formation of hydrogen peroxide (H₂O₂), an oxidising agent stronger than oxygen, by pyrite (FeS₂), the most abundant metal sulphide on Earth, during grinding was investigated. It was found that pyrite generated H₂O₂ in pulp liquid during wet grinding and also the solids when placed in water immediately after dry grinding. Type of grinding medium on formation of hydrogen peroxide revealed that the mild steel produced more H₂O₂ than stainless steel grinding medium, where Fe²⁺ and/or Fe³⁺ ions played a key role in producing higher amounts of H₂O₂. The effect of grinding atmosphere of air and N₂ gas showed that nitrogen environment free from oxygen generated more H₂O₂ than air atmosphere suggesting that the oxygen in hydrogen peroxide is derived from water molecules. In addition, the solids after dry grinding producing more H₂O₂ than wet grinding indicate the role of pyrite surface or its catalytic activity in producing H₂O₂ from water. This study highlights the necessity of relooking into the electrochemical and/or galvanic interaction mechanisms between the grinding medium and pyrite in terms of its flotation behaviour.

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1. Introduction

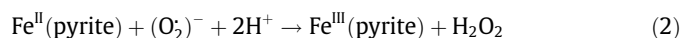
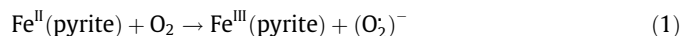
Oxidation of sulphide minerals takes place when they are exposed to atmosphere and in the grinding process for reducing the particle size for flotation. Numerous studies (Rey and Formanek, 1960; Heyes and Trahar, 1977; Gardner and Woods, 1979; Trahar, 1984; Adam et al., 1984; Adam and Iwasaki, 1984a,b,c; Natarajan and Iwasaki, 1984; Yelloji Rao and Natarajan, 1989a,b,1990; Ahn and Gebhardt, 1990 and Peng et al., 2003a) have been done on the influence of type of mill and grinding media on the flotation of sulphide ores. An iron mill reduced the natural floatability of sphalerite significantly (Rey and Formanek (1960). Adam and Iwasaki (1984a,b, c) reported that the more active the metal (mild steel > austenitic > stainless steel), the larger the decrease in floatability of pyrrhotite. The floatability of chalcopyrite is sensitive to the type of grinding media, grinding atmosphere and even the material of the mill (Heyes and Trahar, 1977; Gardner and Woods, 1979; Trahar, 1984; Yelloji Rao and Natarajan, 1989a,b,1990; Ahn and Gebhardt, 1990 and Peng et al., 2003a). A glass mill may improve the recovery of chalcopyrite (Heyes and Trahar, 1977), while noble grinding media such as stainless steel (Ahn and Gebhardt, 1990) and 30 wt.% chromium (Peng et al., 2003a) produced a larger recovery of chalcopyrite than active grinding media, such as mild

steel or high carbon steel under the same atmosphere. The high chromium media had a significantly weaker galvanic interaction with arsenopyrite, and produced a very much lower amount of oxidised iron species in the mill discharge than mild steel medium (Huang and Grano, 2006). The use of iron grinding materials slightly depressed the floatability of galena and pyrite due to iron materials mainly provoking the iron oxy-hydroxide precipitation on the surface of galena and pyrite (Cases et al., 1990).

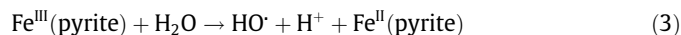
Various mechanisms have been proposed to explain the influence of grinding media on flotation. many authors has been reported (Adam and Iwasaki, 1984b; Natarajan and Iwasaki, 1984; Yelloji Rao and Natarajan, 1989a,b; Iwasaki et al., 1983; Forsberg et al., 1993; Cheng et al.,1993; Yuan et al., 1996; Greet and Steinier, 2004; Greet et al., 2005; Wei and Sandenbergh, 2007) that galvanic interactions occur in every grinding media-sulphide mineral system, which affects the subsequent flotation properties of the sulphide minerals through unselective surface coatings by iron oxidation products. Recently it was revealed that H₂O₂ formation take place during wet grinding of complex sulphide ore (Ikumapayi et al., 2012) and that pyrite (FeS₂), the most common metal sulphide mineral, generates hydrogen peroxide (H₂O₂) (Borda et al., 2001) and hydroxyl radicals, HO· (· denotes an unpaired electron) (Borda et al., 2003) when placed in water. In the presence of dissolved molecular oxygen, ferrous iron associated with pyrite can form superoxide anion (O₂)⁻ (Eq. (1)), which reacts with ferrous iron to form H₂O₂ (Eq. (2)) (Cohn et al., 2006).

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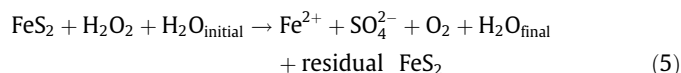
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Borda et al. (2003) showed that pyrite can also generate H_2O_2 in the absence of molecular oxygen. He reported that an electron is extracted from water and a hydroxyl radical is formed (Eq. (3)). Combining two hydroxyl radicals leads to the formation of H_2O_2 (Eq. (4)):



Oxidation of pyrite by H_2O_2 can be described by the following generalised reaction (Lefticariu et al., 2007):



Oxidation of pyrite by H_2O_2 increases the level of complexity of the overall system due to the formation of reactive oxygen species during decomposition of H_2O_2 , such as hydroxyl radicals $\cdot\text{OH}$, superoxide ion radicals $\text{O}_2^{\cdot-}$, and hydroperoxy radicals HO_2^{\cdot} . (McKibben and Barnes, 1986; Lefticariu et al., 2006).

However, participation of H_2O_2 and HO^{\cdot} , if any, in non-selective oxidation of the sulphide ore pulp components and hence in deteriorating of the concentrate grade and recovery of metal-sulphides has not yet been explored. In an attempt to fill the gap, we have estimated the concentration of H_2O_2 in pulp liquid during different time of grinding and in different grinding environments. The effect of two types of grinding media (mild steel and stainless steel) on formation of hydrogen peroxide and pyrite flotation was investigated.

2. Experimental

2.1. Materials and reagents

Crystalline pure pyrite mineral sample was procured from Gregory, Bottley & Lloyd Ltd., United Kingdom. The XRD analysis of the sample showed that this pyrite sample was very pure (Fig. 1) and contains 44.4% Fe, 50.9% S and 0.2% Cu. All the pyrite used in this study was simultaneously crushed through a jaw crusher and then screened to collect the -3.35 mm particle size fraction. The homogenised sample was then sealed in polyethylene bags. Potassium amylose xanthate (KAX) was used as collector and MIBC was used as frother. Solutions of sodium hydroxide (AR grade) and HCl (1 M) were used to maintain the pH at the targeted

value during flotation. Deionised water was used in the processes of both grinding and flotation. Solutions of 2, 9-dimethyl-1, 10-phenanthroline (DMP), copper (II) (0.01 M), and phosphate buffer (pH 7.0) were used in the estimation of H_2O_2 .

2.2. Wet grinding and flotation tests

One hundred grams of -3.35 mm size fraction of pyrite for each grinding test was combined with 400 cm³ of deionised water and ground in a new laboratory stainless steel ball mill (Model 2VS, CAPCO Test Equipment, Suffolk, UK) with either of two types of grinding media (mild steel or stainless steel) at natural pH (i.e. pH 3–4). The slurry samples were collected at pre-determined time intervals of grinding and they were filtered (Millipore 0.22 μm) and liquid (filtrate) was analysed for hydrogen peroxide.

After grinding for 60 min, the mill was emptied and the pulp was screened to free from grinding media and it was split into five samples for flotation at different pH values. In each flotation almost 7.5 g of sample that was <106 μm was transferred to a cell of 150 ml capacity (Clausthal flotation equipment), conditioned with pH modifier, collector and frother. Flotation concentrate was collected after 2.0 min at air or N_2 flow rate of 0.5 dm³ min⁻¹. The flotation froth was scraped every 10 s. Dosages of collector in flotation was 10^{-4} M KAX. The conditioning times for adjusting pH and collector were 5 min and 2 min respectively. The frother dosage was one drop MIBC in all cases. Pyrite flotation was investigated at different pH (pH 10.5, 11.5 and 4.5 with 10^{-4} M KAX), then it was investigated with collector (10^{-4} M KAX) and without collector at pH 4.5, and finally it was investigated in different gas bubbling (air or N_2 bubbling at pH 4.5 and 10^{-4} M KAX). The pH was regulated to 10.5 and 11.5 with NaOH solution and 4.5 with HCl solution. Experiments were performed at room temperature of approximately 22.5 °C.

2.3. Dry grinding

One hundred grams of -3.35 mm size fraction pyrite was ground in a laboratory stainless steel ball mill with two types of grinding media (mild steel and stainless steel) for 60 min. After grinding, the mill was emptied and the pyrite was screened from grinding media. A 5 g of sample that was <106 μm was mixed with 50 cm³ of water in a magnetic stirrer for 0.5 and 5 min. The slurry sample was then collected and analysed for hydrogen peroxide. The pH was regulated with HCl or NaOH solution.

2.4. Gas purging

To study the effect of the atmosphere, pyrite was wet-ground in a laboratory ball mill with stainless steel medium in either air or N_2 atmosphere. For N_2 atmosphere, first the laboratory ball mill was filled with 400 ml deionized water and purged with N_2 gas for a minimum of 30 min. After 30 min, pyrite was added and the laboratory ball mill was again purged with N_2 gas for a minimum of 30 min and finally pyrite was wet-ground for 1 h. Though we did not measure dissolved O_2 concentrations in our experiments, it has been reported that for 1 L solutions of ultrapure water purged for 1 h with N_2 gas, O_2 concentrations did not exceed 0.19 ± 0.05 ppm (Butler et al., 1994). The concentration of H_2O_2 was measured after 60 min of grinding.

2.5. Hydrogen peroxide analysis

So far, various methods have been used for the measurement of H_2O_2 in oxidation processes. Such methods use metallic compounds such as titanium oxalate, titanium tetrachloride (Volk et al., 1993; Roche and Prados, 1995; Sunder and Hempel, 1997;

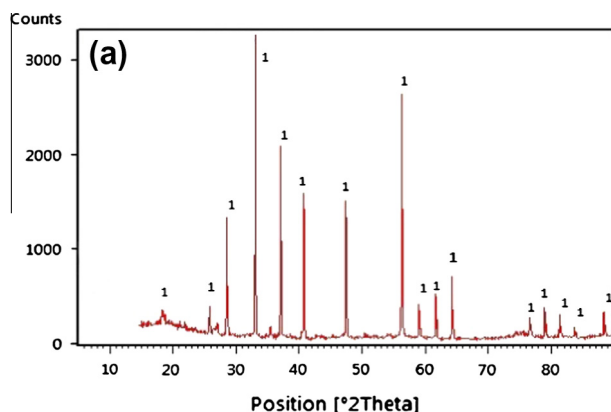


Fig. 1. XRD analysis of the pyrite sample. (1) pyrite.

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