

Effect of Fenton-like oxidation reagent on hydrophobicity and floatability of chalcopyrite and molybdenite

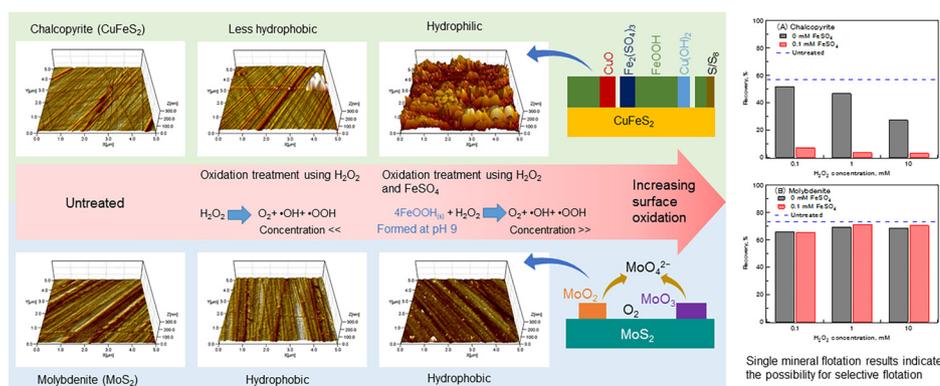


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GRAPHICAL ABSTRACT



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ABSTRACT

A fundamental study is provided in this work to understand the effect of Fenton-like reagent made by the addition of FeSO₄ in H₂O₂ aqueous solution on surface hydrophobicity and floatability of chalcopyrite (CuFeS₂) and molybdenite (MoS₂). Contact angle measurements were performed to assess the surface hydrophobicity. The contact angle results showed that Fenton-like reagent could alter the surface hydrophobicity of chalcopyrite at lower concentration of H₂O₂ aqueous solution compared to that of using H₂O₂ aqueous solution. On the other hand, molybdenite surface remained hydrophobic after the oxidation treatments using Fenton-like reagent and H₂O₂ aqueous solution. Surface characterizations using atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS) shows that the chalcopyrite surface covered with a thicker layer of oxidation products after the oxidation treatment using Fenton-like reagent, indicating a stronger surface oxidation. Flotation results were in agreement with contact angle results, showing that Fenton-like reagent could depress the floatability of chalcopyrite at lower concentration of H₂O₂ aqueous solution. On the other hand, molybdenite recovery remained high under various oxidation treatments owing to low surface oxidation.

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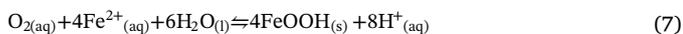
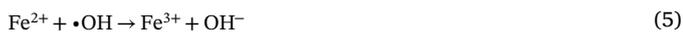
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1. Introduction

Separation of copper (Cu) and molybdenum (Mo) sulfide mineral is an important issue in flotation of sulfide minerals. Molybdenum minerals are often associated with copper sulfide minerals [1] with a typical molybdenum content ranges between 0.01 and 0.25%. Approximately about 50% of the world's molybdenum production comes from porphyry Cu-Mo ores [2,3]. Molybdenum is mainly produced by recovering molybdenite (MoS₂) from Cu-Mo ores as a by-product with copper at levels as low as 0.01% Mo [4]. In addition, molybdenum and copper are valuable materials in various fields and the recovery of molybdenum minerals, especially molybdenite, plays a very important role in making the Cu-Mo processing plants economically viable [5]. Moreover, contamination of molybdenum minerals in the copper concentrate reduces the concentrate value [6]. Therefore, it is important to separate molybdenum and copper minerals effectively and efficiently.

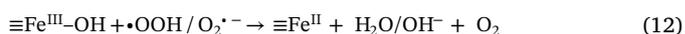
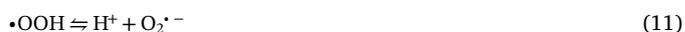
Conventionally, copper and molybdenum ores are separated in selective flotation circuits by using copper depressants (i.e., sodium hydrosulfide (NaHS), sodium sulfide (Na₂S), sodium thioglycolate (HSCH₂COONa), sodium thiopropionate (HSCH₂CH₂COONa), and Nokes reagent (P₂S₅+NaOH)) [2,7–12]. Recently, various methods have been developed to replace these toxic and hazardous depressants, for example, by synthesizing new non-toxic or less-toxic depressants such as 2,3-disulfanylbutanedioic acid, chitosan, sodium sulfite (Na₂SO₃), and lignosulphonate [6,7,13–15] or by using various oxidation methods such as sodium hypochlorite (NaOCl) in an alkaline medium, manganese dioxide (MnO), oxygen (O₂), ozone (O₃), and plasma pre-treatment [16–20]. Recent study by Chimonyo et al. [21] reported the effect of H₂O₂ on reducing the recoveries of sulfide minerals. Moreover, Hirajima et al. [19] demonstrated that H₂O₂ depressed the floatability of chalcopyrite (CuFeS₂) and could deliver a selective separation on copper and molybdenum (Cu-Mo) bulk concentrate in flotation process. However, they reported that this method required a higher concentration of H₂O₂ and a longer treatment time (4.5 h) to get a comparable result to those obtained by using NaHS as copper depressant.

It is well known that the oxidizing performance of H₂O₂ can be improved by adding the iron ions. The reaction between H₂O₂ and iron ions in the solution can produce hydroxyl and hydroperoxyl radicals (\cdot OH and \cdot OOH, respectively) by following the Fenton reaction as shown from Eqs. (1)–(6) [22–24]. The hydroxyl radical (\cdot OH) is a stronger oxidizing agent than that of the oxygen [23,25], thus a much more effective oxidant than oxygen. Fenton reagent is usually used for treating organic wastes (i.e., humid acid, pesticides, phenol, dimethyl phthalate, and dyes) in water [26–32]. In addition, Fenton reagent has been used in flotation process to separate polyvinyl chloride (PVC) from plastic waste for recycling [33] and to recover LiCoO₂ and graphite from spent lithium-ion batteries [34]. However, the Fenton reaction is limited to acidic pH ranges and the optimum pH is 2.5–4 for treating organic contaminants [35,36]. The formation of amorphous iron oxide precipitates as shown in Eq. (7) [37] reduced the effectiveness of the Fenton reagent at alkaline conditions.



Recently, many researchers utilized the heterogeneous catalyst to overcome the problems generated from the Fenton reaction, i.e., the

generation of secondary pollutants, the ferric ions, the low pH conditions, and the difficulties to recover and recycle the dissolved iron catalyst, without losing its oxidation performance [38,39]. The catalysts are basically iron metals such as hematite (Fe₂O₃), magnetite (Fe₃O₄), ferrihydrite, goethite (α -FeOOH), lepidocrocite (γ -FeOOH), mixed element oxides, and steel slag [40–49]. Xueyan Li et al. [46], showed that the presence of FeOOH species, such as α -FeOOH, in the solution could accelerate the degradation of H₂O₂, thus producing a higher concentration of oxygen and hydroxyl radicals. Lin and Gurol [50] proposed a mechanism for the reaction between Fe³⁺ from FeOOH precipitate with H₂O₂ producing O₂ (8)–(12).



In this study, ferrous sulfate (FeSO₄) was used to improve the oxidation performance of H₂O₂ through the formation of Fenton-like reagent. FeSO₄ can produce various oxidation products (i.e., lepidocrocite, ferrihydrite, and goethite) under various alkaline conditions [51]. Therefore, it may act as a catalyst to enhance the oxidation treatment using H₂O₂ at alkaline conditions. Indeed, FeSO₄ has been used to improve the oxidation process using H₂O₂ for treating sand-packed columns containing pentachlorophenol (PCP) and trichloroethylene (TCE) [52].

As mentioned previously, molybdenite can be recovered from Cu-Mo ores as a by-product in a flotation process. This is because of large differences between surface properties, typically surface hydrophobicity, of molybdenite and other sulfides minerals. However, flotation is a dynamic process and in this process hydrophobicity is a necessary, but not sufficient condition for floatability [53]. Therefore, the objective of this study is to investigate the effect of oxidation treatment using Fenton-like reagent made by the addition of FeSO₄ in H₂O₂ aqueous solution on surface hydrophobicity and floatability of chalcopyrite and molybdenite. Moreover, the effects of treatment time and various concentrations of H₂O₂ aqueous solution on hydrophobicity and floatability of both minerals are presented in this work. For this purpose, contact angle measurements and flotation tests were performed.

2. Materials and method

2.1. Materials

Chalcopyrite and molybdenite from Miyatamata Mine, Akita Prefecture, Japan, and Wolfram Camp, Queensland, Australia, respectively, were used for contact angle measurements and surface characterizations by atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS) analysis. Mineral powders were used for flotation tests. Molybdenite powder (< 30 μ m) was supplied by Sumitomo Metal Mining Co., Ltd., and cleaned using a 1 M HNO₃ aqueous solution by following the procedures described by Suyantara et al. [54]. The powder was then rinsed with ultra-pure water to give a filtrate pH of ca. 5. Afterwards, the powder was freeze-dried (FDU 1200, Eyela, Japan) over 24 h to give the dried powder. Chalcopyrite (Miyatamata Mine, Akita Prefecture, Japan) was carefully hand-picked, was ground using an agate mortar and pestle, and was dry screened (< 38 μ m).

XRF analysis showed that the purity of chalcopyrite powder was 99.96% with a small amount of Si (0.03%) as contaminant. Meanwhile, the purity of molybdenite was 99.42% with small amounts of Al (0.09%), Si (0.18%), P (0.19%), and Fe (0.11%) as contaminants. For surface characterization using AFM and contact angle measurements, the mineral samples were cut as a flat surface and were casted in an

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