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### Impact of mechanical activation and mechanochemical activation on natural pyrite dissolution



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#### A R T I C L E I N F O

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#### ABSTRACT

In this paper, a new pretreatment method was employed for the enhancement of pyrite mineral dissolution by using mechanochemical activation. The mechanochemical activation was carried out by co-milling of pyrite and iron powder for 20 and 50 min. The XRD analysis indicated that a fraction of pyrite and iron phase remained and troilite (FeS) was formed as a new phase after 50 min milling. In addition, the microstructural changes of mechanochemically and mechanically activated pyrite were investigated by Rietveld method. The results revealed that amorphization degree increased from 53% in mechanically activated pyrite for 50 min to 83% in mechanochemically activated pyrite for 50 min. The mechanochemical activation intensified crystallite size reduction of pyrite in comparison with mechanical activation. In spite of mechanochemical reaction. Although, both the mechanochemical and mechanical activations of pyrite can enhance the dissolution of pyrite, total iron extractions in the 1 M sulfuric acid leaching tests at ambient temperature increase from 1.12% for the initial pyrite to 21.22% for 50 min mechanically activated pyrite leaching.

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#### 1. Introduction

The leaching of sulfide minerals and the hydrometallurgical extraction of valuable metals from sulfide ores are technologically difficult processes for extreme reaction conditions required (Tkacova and Balaz, 1996). The operational difficulties of hydrometallurgical processing of sulfide minerals such as extreme leaching conditions, formation of elemental sulfur, high acid consumption, low recovery and high cost have been hindering the commercialization of these processes (Zarate-Gutierrez et al., 2012). Among these minerals, hydrometallurgical treatment of pyrite has been studied for several aims such as leaching of pyrite as a pretreatment for extraction of gold from goldbearing minerals, pyrite removal from coal concentrate and enhancement of sulfide minerals leaching (Hu et al., 2002; Koleini et al., 2011). Although, pyrite is described as a refractory mineral due to difficulty of its treatment (Lehmann et al., 2000), the sulfate system for hydrometallurgical processing of pyrite has been preferable regarding the low cost and sufficient supply of sulfuric acid. A number of investigations were carried out on pyrite for its hydrometallurgical processing with sulfuric acid under various leaching conditions. The reaction between pyrite and sulfuric acid was complete at a temperature of 160 °C, while when less than 160 °C, formation of elemental sulfur terminated the reaction before completion by covering the surface of pyrite particles (Papangelakis and Demopoulos, 1991). Additionally, the complete decomposition of pyrite mechanically activated under nitrogen atmosphere for 120 min was reported by using a lixiviant of 0.5 M nitric acid and 1.5 M sulfuric acid at a temperature of 40 °C (Hu et al., 2004). Also, the high measures of pyrite conversion were attained under severe leaching conditions during pyrite oxidation by using sulfuric acid under oxygen pressure (Long and Dixon, 2004). Regarding the previous investigations, a new method has to be proposed to balance between these advantages and disadvantages promising high recovery of process, low reaction temperature and cheaper leaching agent. Absolutely, these methods cannot avoid all disadvantages, but it can reduce their impact on sulfide mineral of pyrite hydrometallurgical processing.

In mechanochemistry science, mechanical energy is employed for the performance of chemical reactions and physicochemical changes through high-energy ball milling process. The mechanochemical reactions occur between reactants involved providing thermodynamic rules permit when the free energy changes of reactions are negative. Keeping chemical composition of involved powders unchanged during high-energy ball milling, mechanical activation phenomena happened. Mechanical activation, a branch of mechanochemistry science, can promote mineral reactivity by affecting a wide range of changes in their structure. Structural changes such as formation of defects, polymorphic transformation, grain and crystallite refinements and lattice strain induced during mechanical activation lead to a large number of positive

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influences on the processes in which the minerals are involved (Pourghahramani and Akhgar, in press, 2008; Akhgar et al., 2012).

The new term entitled "mechanochemical activation" can arise from mechanochemistry science wherein a mechanochemical reaction was designed for activation of minerals, not for mechanochemical synthesis of a product. The mechanochemical activation of a mineral can be made by co-milling of a constituent element of the mineral during short period of intensive grinding without completion of the mechanochemical reaction, for example; Fe addition during intensive milling of chalcopyrite, ilmenite or pyrite. The short period of intensive milling in mechanochemical activation can increase capacity of related installations and reduce energy consumption and costs. Additionally, there is the hope that the chemical bonds in the mineral composition are weakened during mechanochemical activation more than mechanical activation, promoting reactivity of mechanochemically activated minerals more.

Due to the valuable metals present in pyrite, its refractory character and simple crystallographic arrangement (face-centered cubic, FCC), the introduction of new treatment for hydrometallurgical processing of pyrite may be a matter of interest and the used technique can give some insight into hydrometallurgical processing of other sulfide minerals. Also, pyrite (FeS<sub>2</sub>) as the most common gangue sulfide mineral is frequently associated with valuable minerals in ore deposits. By innovation of a proper process to benefit the inexpensive source of iron, pyrite would not be considered as gangue mineral. As part of our work for the production of advanced and nano-structure material from natural pyrite, intensive milling was employed for increasing the reactivity of natural pyrite. The aim of this paper is to investigate mechanical and mechanochemical activation influences on reactivity of pyrite during its hydrometallurgical processing from microstructural view point. The structural changes of pyrite during mechanical and mechanochemical activation were characterized using XRD analysis to provide precious information about the mechanochemical activation effectuality.

#### 2. Experimental

#### 2.1. Materials

A high-purity pyrite concentrate was obtained using conventional beneficiation methods. XRD analysis of pyrite concentrate disclosed that pyrite reflections solely corresponded with JCPDS No. 1-1295 (Fig. 1).

The iron powder (Merck 3800.1000) was used for mechanochemical activation treatment as will be described in the following section.

#### 2.2. Mechanical and mechanochemical activations

Mechanical activation of pyrite was conducted by a planetary ball mill (Pulverisette 6, FRITSCH, Germany) in a 250 cm<sup>3</sup> cup for 20 and 50 min with a rotation speed of 400 rpm under argon atmosphere to



Fig. 1. XRD pattern of the initial pyrite.

prevent unwanted chemical reactions. 15 stainless steel balls having diameters of 20 mm were used for milling by the ball to powder weight ratio of 20:1. The milling conditions during mechanochemical activation were similar to the mechanical activation in milling speed, ball to powder ratio, milling atmosphere, milling time and ball size and number. For mechanochemical activation of pyrite, the natural pyrite and iron powder were co-milled by the stoichiometry ratio presented in the following reaction.

$$FeS_2 + Fe \rightarrow 2FeS$$
 (1)

#### 2.3. Leaching tests

The leaching experiments were carried out in a 500 ml baker reactor by 1 M  $H_2SO_4$  solution. According to experimental details (Table 2), the initial feed of leaching experiments was the natural pyrite, mechanically and mechanochemically activated pyrite. The experiments started with the addition of the initial feeds and an agitator with 475 rpm stirring speed was used to trigger mechanical agitation. The leaching experiments were performed for 5 h and then the leach liquors were separated from the leach residues. Fig. 2 shows a schematic route of the used approach for comparison of mechanical activation and mechanochemical activation impact on the leaching behavior of natural pyrite.

#### 2.4. Characterization

The X-ray diffraction patterns were collected with BrukerAxs D8 advanced instrument (Germany), applying Cu K $\alpha$  radiation ( $\lambda = 1.5406$  Å) at 50 KV and 250 mA in the two theta ranges of 25–70°. The patterns were recorded using a step size of 0.02 and a counting time of 3 s per step. The final concentrations of extracted iron in the obtained leach liquors were measured by UV spectrophotometer (Specord 200 plus, Analytikjena, Germany).



Fig. 2. A schematic flow sheet of approach used.

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