



## Improvement of phosphorus removal from iron ore using combined microwave pretreatment and ultrasonic treatment



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### ABSTRACT

Most of the past studies examined the effects of ultrasonic treatment on the removal of phosphorus, silica and alumina minerals from iron ores. In the present work, the effect of combined microwave pretreatment and ultrasonic treatment on the efficiency of disintegration and removal of phosphorus and other gangue minerals associated with iron ores has been studied. Three different iron ore samples have varying total iron concentration (TFe) and P<sub>2</sub>O<sub>5</sub> content and mineralogical textures were studied.

Microwave pretreatment generated intergranular fractures between the gangues (fluorapatite and chamosite) and oolitic hematite. These intergranular fractures improved liberation of iron ore, and accelerated ultrasonic disintegration and removal of phosphorus and gangue minerals from oolitic hematite. The results indicated that microwave pretreatment increases the efficiency of ultrasonic disintegration and removal of particles by about 20% compared to untreated sample. The results of ultrasonic treatment are quite promising. Significant increase in iron grade and reduction in phosphorus and alumina content of enriched product can be obtained. Depending on the sample texture and phosphorus distribution, about 59% phosphorus removal can be obtained.

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### 1. Introduction

There is a rapidly increasing demand for iron resources with the rapid development of iron and steel industry, therefore iron and steel industry facing the risk of raw material shortage. Deposits of high phosphorus oolitic iron ores are widely spread worldwide [1–7]. The main obstacle associated with exploiting these deposits is the fine dissemination of silica, aluminum and in particular phosphorus minerals, which affects the economy of iron making process and the quality of the produced steel. Song et al. [8] observed that fine grinding (commonly 1–5 μm) is required to liberate iron minerals from associated gangue minerals. Such fine particles are very difficult to beneficiate via conventional mineral processing operations. For upgrading high phosphorus oolitic iron ores, many hydrometallurgical and pyrometallurgical processes were proposed [9–15]. Although some of these methods achieved the purpose of phosphorus removal, they suffer from some disadvantages. For instance, low efficiency of dephosphorization, environmental pollution, relatively high cost, and low iron

recovery represent drawbacks that make these techniques impractical. The development of a successful and economic process to remove phosphorus from the high phosphorus iron ores would significantly extend the reserves of high grade low phosphorus iron ores, and develop iron and steel industries [16].

A growing interest in microwave heating in mineral treatment has emerged in recent years, and a number of potential applications regarding microwave processing have been investigated [17–21]. Microwave treatment improves the liberation of high phosphorus oolitic iron ores by generating intergranular fractures in oolitic iron ores [8]. The difference in the absorption of microwave energy, thermal expansion and the dielectric properties of iron and gangue minerals leads to the generation of intergranular fractures between iron and gangue minerals [17,22–25]. High phosphorus oolitic iron ores are usually composed of hematite, dolomite, clinocllore, quartz and apatite (fluorapatite or hydroxyl fluorapatite), these minerals differ in how they absorb microwave energy [15]. These minerals have different reactions regarding thermal expansion, and thus thermal stresses are generated on the boundaries between them. When these thermal stresses reach a critical level, cracks and fissures are formed at the boundaries [26]. Omran et al. [27,28] studied the influence of microwave pre-treatment on the liberation of high phosphorus oolitic iron

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ore. They concluded that intergranular fractures formed between the gangues (fluorapatite and chamosite) and oolitic hematite after microwave treatment, leading to improved liberation of iron ore [27,28]. Omran et al. [27–30] concluded that compared to conventional thermal treatment, microwave treatment consumes considerably less energy, improves liberation and reduces processing time. Many researchers investigated the influence of microwave pre-treatment on magnetic separation and surface characteristics of ores [31–33]. They concluded that microwave treatment of ores is potentially high efficient pre-treatment technique with low energy consumption [31–33].

In the present study, ultrasonic treatment was applied to the microwave treated samples with the aim of investigating the effect of combined microwave pre-treatment and ultrasonic treatment on the separation of phosphorus and other gangue minerals from iron ore.

When ultrasonic waves are propagating through liquid, vibrations in the medium create a series of rarefactions and compressions that initiates nucleation of micro-bubbles “cavitation”. These cavitations take place primarily on the phase boundary since solid/liquid interactions are weaker than liquid cohesion forces [34–39]. When high temperature and pressure are attained inside the cavitation bubbles, their diameter exceeds a critical value, they collapse asymmetrically in the vicinity of solids present within the liquid [34–41]. Asymmetric collapse leads to the formation of high speed micro-jets that have the potential to remove the gangue constituents adhered on the surface of iron mineral particles [40,41].

Donskoi et al. [41–48] extensively studied the effect of ultrasonic treatment on upgrading of iron ore. They concluded that it is possible to increase iron grade and recovery if hematitic/goethitic iron ore treated with ultrasound [41]. They also found that the application of ultrasound with stirring have a significant effect on the product grade [43]. Ozkan [49] reported that recovery values of magnesite slimes increased with ultrasonic treatment. Franko and Klima [38] have concluded that ultrasonic treatment helped to separate ultra-fines attached to larger particles in iron ore beneficiation processes. Pandey et al. [40] also reported that ultrasonic treatment followed by de-sliming significantly reduced alumina, silica and phosphorus for two Indian iron ores.

The findings of the previous studies [8,27–33] indicated that microwave heating can improve the liberation, grindability, and magnetic separation of valuable minerals from ores and has a potential to provide a new method to treat ores successfully. The aim of this investigation is to study the effect of microwave pre-treatment on the effectiveness of ultrasonic disintegration and separation of phosphorus and other gangues minerals from iron ores. The hypothesis of using combined microwave pretreatment and ultrasonic treatment is that intergranular fractures formed

between the gangues and oolitic hematite grains after microwave treatment, when ultrasonic treatment applied to the microwave treated sample, these intergranular fractures facilitate ultrasonic waves to remove the gangue constituents adhered on the surface of iron mineral particles.

## 2. Experimental and materials

### 2.1. Iron ore samples

The iron ore samples used in this study were collected from Aswan region, Egypt. Eastern Aswan area represents the main occurrence of the Cretaceous ironstone bands of South Egypt [6,7]. Three different samples were obtained from different locations from Aswan region. Tables 1 and 2 show the chemical and mineralogical analysis of the iron ore samples investigated in this study. The main mineral phase found in all samples is hematite as the main iron bearing mineral. The main associated gangue minerals are fluorapatite, quartz and chamosite.

### 2.2. Microwave treatment

Samples were treated using a 2.45 GHz microwave oven with 900 W maximum output power. Three representative samples with grain size in the range (–1000  $\mu\text{m}$  +500  $\mu\text{m}$ ), (–500  $\mu\text{m}$  +250  $\mu\text{m}$ ) and (–250  $\mu\text{m}$  +125  $\mu\text{m}$ ) were used in each test. Iron ore samples were treated under normal atmosphere condition and directly used without any pretreatment. Samples were placed in the microwave oven in crucibles made of pure alumina. The temperature of each sample was measured by quickly inserting a thermocouple into the sample after the power was turned off, and the temperature was monitored by a digital display temperature controller. The measured temperatures are the bulk temperature of the samples (Fig. 1). The samples were then allowed to cool in the microwave oven to room temperature.

### 2.3. Ultrasonic treatment

An ultrasonic cleaning vessel (Elmasonic P30H) with ultrasonic frequency 37 kHz and ultrasonic power 350 W was used. The internal dimensions of ultrasonic tank are (240  $\times$  137  $\times$  100 mm). The experimental setup is shown in Fig. 2. The pulp sample was placed in the experimental vessel together with an impeller (for stirring) in the ultrasonic bath. A schematic diagram of the process flow sheet is shown in Fig. 3.

The impeller rotation speed was varied from 350 rpm to 700 rpm and it was observed that with increasing rotation speed the power

**Table 1**  
Chemical analyses (wt.%) for the three iron ore samples and selected size fractions from each sample.

Sample code		TFe (%)	P <sub>2</sub> O <sub>5</sub> (%)	CaO (%)	SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	MgO (%)	F (%)
G1	Bulk sample	48.33	5.64	7.59	15.03	3.37	1.23	0.37
	+125 –250 $\mu\text{m}$	48.57	3.48	5.36	17.70	3.70	1.25	0.21
	+250 –500 $\mu\text{m}$	47.97	3.17	4.83	20.07	3.40	1.14	0.22
	+500 –1000 $\mu\text{m}$	51.11	3.09	4.70	17.25	3.10	1.03	0.19
G2	Bulk sample	46.08	2.25	2.67	29.54	3.27	0.27	0.32
	+125 –250 $\mu\text{m}$	46.96	1.89	2.44	30.32	2.58	0.36	0.28
	+250 –500 $\mu\text{m}$	45.35	1.45	1.75	33.26	2.41	0.45	0.29
	+500 –1000 $\mu\text{m}$	44.23	1.33	1.59	34.61	2.24	0.26	0.3
G3	Bulk sample	58.27	3.24	5.44	7.48	4.47	1.26	0.19
	+125 –250 $\mu\text{m}$	59.61	2.50	4.67	8.16	3.99	0.92	0.07
	+250 –500 $\mu\text{m}$	58.50	2.79	4.97	8.66	4.12	0.99	0.09
	+500 –1000 $\mu\text{m}$	62.30	2.28	4.10	6.84	3.40	0.74	0.05

<sup>(G1)</sup>High phosphorus sample. <sup>(G2)</sup>Low phosphorus sample. <sup>(G3)</sup>Intermediate phosphorus sample.

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