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COMBINED ROASTING AND LEACHING TREATMENT FOR REDUCING PHOSPHORUS, ALUMINUM AND SILICON IN OOLITIC IRON ORE

Purpose. To enhance the quality of oolitic iron ore extracted from the Gara Djebillet mine in southern Algeria by reducing the levels of silicon, aluminum, and phosphorus, thus making it more suitable for use in the steel industry.

Methodology. The study involves pre-treating the Gara Djebillet iron ore through roasting, followed by two stages of leaching with separate acid and base leaching steps. Additionally, the impact of introducing an additional roasting step at 800 °C between the two leaching stages is examined.

Findings. Chemical leaching of the raw ore without roasting pre-treatment results in a reduction in silicon, aluminum, and phosphorus contents from 4.45, 5.11 and 0.61 % to 2.68, 3.36 and 0.3 %, respectively. However, the iron content decreases from 52.42 to 45 %. Pre-treating the ore with roasting combined with the two leaching stages reduces the phosphorus content to 0.15 % and increases the iron content to 55.25 %. The silicon and aluminum contents decrease to 4.2 and 5 %, respectively. Introducing a second roasting step between the two leaching stages further decreases the phosphorus content to 0.15 %, but the iron content only increases to 54.25 % after the second acid leaching step. The aluminum and silicon contents increase to 4.5 and 5.3 %, respectively.

Originality. This study introduces a novel approach to improving the quality of oolitic iron ore by investigating the efficacy of pre-treatment with roasting followed by two stages of leaching. The research contributes valuable insights into the effectiveness of these methods for reducing undesirable elements in iron ore.

Practical value. The findings offer practical implications for the steel industry, suggesting potential methods for enhancing the quality of iron ore from the Gara Djebillet mine. Implementing these methods could lead to increased efficiency and cost-effectiveness in iron ore processing, ultimately benefiting steel production processes.

Keywords: *oolitic iron ore, leaching, roasting, phosphorus removal*

Introduction. The Gara Djebilet mine in southern Algeria has estimated ore reserves of 2.3 billion tons [1], and could serve as the main source of raw materials for the Algerian steel industry. The deposit, which is in Tindouf near the Algerian-Mauritanian border, is divided into three major areas: east, west, and central one. Each area is characterized by different amounts of iron and a diversity of mineral forms of various elements. Because of the variety of mineral forms, and the quantity of undesirable substances, a diverse range of enrichment technologies are required to meet industrial requirements. Processes need to be developed to remove (or reduce) the gangue content of the Gara Djebilet ore, and particularly the high content of phosphorus.

Gangue contaminants, especially phosphorus, can be difficult to remove from iron ore when their mode of occurrence is not known. Phosphorus is typically removed from iron ore using gravity concentration [2], chemical leaching [3], thermal treatment and bioleaching [4], froth flotation [5], and various combinations of these techniques, such as low-intensity magnetic separation-high-intensity magnetic separation-reverse

flotation [6]. Few studies have investigated improving the quality of low-grade iron ore from Gara Djebilet. Boucif, et al. investigated pelletizing Gara Djebilet iron ore to supply a blast furnace. They found that the disaggregation rates of Gara Djebilet pellets bound with bentonite and lime were 1.5 and 2.0, respectively, and these values met the requirements for a blast furnace. However, the disaggregation rates have not been measured for ores with different gangue compositions. Recently, Mansour, et al. investigated separation of iron and phosphorus in Gara Djebilet ore using sodium sulfate (Na₂SO₄) in coalbased direct reduction before adding iron ore-coal-CaO mixed pellets to an electric arc furnace. They obtained a final direct reduced powder (DRI) with 96 % Fe and 0.16 % P by mass using 5 % Na₂SO₄ in the mixed pellets. However, a high reduction temperature of 1,250 °C for 30 min was used [7]. Recently, Mochizuki et al. investigated improving the quality of low-grade iron ore from different countries by removing gangue using a combination of alkali roasting and hydrothermal treatment with NaOH. They achieved silicon, aluminum, and phosphorus removal rates of 10-92, 39-70 and 38-78 %, respectively [8]. Current studies have shown that low release, thermal, and mixed processes are the most appropriate meth-

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ods for ores with a high content of phosphorus in the iron matrix [3, 4]. It has been shown that heat treatment disrupts the structure of goethite and liberates the phosphorus associated with this mineral, which makes it accessible to extraction by a leaching solution. Chemical leaching is an effective method for phosphorus removal and does not result in dissolution or loss of iron if the mineralogical phases of the ore are resistant to treatment. Acid leaching is an efficient method for removal of phosphorus from iron ore. Experimentally, leaching is a simple method to treat the fine particles in sinter feed without the restrictions that are necessary in flotation. Alkaline leaching is effective for removal of the phosphorus present in apatite and silicates [9]. Research by Mansour et al. indicated that the phosphorus in Gara Djebilet iron ore probably is presented as apatite and silicates [7].

Taking into account all of the above, we propose a new process for phosphorus removal consisting of two leaching steps with acid (HCl) and base $(Ca(OH)_2)$ combined with roasting at 800 °C. We assessed the effect of the roasting step on the removal efficiencies of phosphorus, silicon and aluminum. Samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) coupled with energy dispersive Xray analysis (EDS) and X-ray fluorescence (XRF).

Material and methods. Sample preparation and characterization. The samples were first crushed to coarse particles, and then ground to 100 μ m in size. The samples were then homogenized. Before analysis and processing, representative raw ore samples were dried under vacuum for 24 h at 105 °C and then cooled at room temperature.

XRD. The degrees of crystallinity of raw and roasted iron ore samples were analyzed by XRD with a 2θ scan range of 2 to 80° . The instrument was operated at 40 kV and used a Cu-K α ($\lambda = 1.540056$ A°) radiation source. The scan step size was 0.03° and the scan step time was 0.25 s. The equipment was installed at the Centre de Recherche Scientifique et Technique en Analyses Physico Chimiques (Algiers, Algeria).

XRF. The chemical compositions of the samples were determined by XRF using a portable spectrometer (ZSX Primus II, Rigaku) at the Centre de Recherche Scientifique et Technique en Analyses Physico Chimiques. The spectrometer was equipped with miniaturized silver anode X-ray tubes (50 kV, 200 μ A) and a high-performance geometrically optimized large-area drift detector.

SEM. The surface morphologies of the Gara Djebilet iron ores were observed by SEM (FEI Quanta 400 MK2). The SEM instrument was coupled with an EDS instrument at the Centre de Recherche Scientifique et Technique en Analyses Physico Chimiques. The samples for analysis were prepared using an aluminum support covered with a self-adhesive carbon pad, which was used to ensure sample conductivity and avoid accumulation of electrons.

Sample roasting. Sample pre-treatment and calcination were carried out in a muffle furnace at 600, 700, 800 and 900 °C. After roasting at the optimum temperature, the samples were removed from the furnace, cooled to room temperature, and then analyzed by XRD.

Leaching of samples. For the acid leaching step (L1), the leaching experiments were performed in 250 mL Pyrex flasks at ambient temperature (20-25 °C). The solutions were homogenized using magnetic stirrers at a controlled speed. In each leaching experiment, the sample (10 g) was added to the flask with a solid/liquid ratio of 1:4 (10 g ore/40 mL) and the mixture was stirred for 45 min. After leaching, the solution was filtered, and the residue was washed and dried at 100 °C for 24 h. For the base leaching step (L2), the ore samples were treated with a lime solution under the same conditions as for the acid leaching. At the end of each leaching step, the contents of silicon, phosphorus, and aluminum were analyzed by XRF.

To increase the rate of impurity removal obtained after the first stage of acid and base leaching steps, we repeated the same treatment steps a second time (L3 and L4). Calcination

was used as for pre-treatment before the leaching process and also after the first acid and base leaching steps (L1 and L2) as shown in Fig. 1.

Results and discussion. *Ore composition.* The elemental composition of the Gara Djebilet ore was determined by XRF. The results are shown in Table 1.

The iron content of Gara Djebilet ore sample was 52.42 % and close to the literature values for other ores (53.2-57 %) [7]. The phosphorus content (0.6 %), which was probably in the form of apatite species (e.g., fluorapatite and hydroxyapatite) as suggested by several authors [7, 8], was high in terms of the requirements of the steel industry. The phosphorus content found in this work in Gara Djebilet iron ore from the southwest of the Tindouf basin (130 km south-east of the town of Tindouf) was lower than that found by Mansour, et al. This result could indicate that the gangue content depends on the site of the deposit.

The Gara Djebilet ore had low contents of alkaline earth metals (CaO and MgO), which indicated that the clays were poor in carbonates. The XRD patterns of the samples are shown in Fig. 2.

The XRD results showed that the total iron (TFe) ore was composed mainly of hematite (50.2 % TFe), goethite (4.8 % TFe), maghemite (24.5 % TFe), and magnetite (20.5 % TFe). The XRD pattern showed that the gangue contained kaolinite and quartz.

SEM and EDS analysis. Fig. 3, *a* shows the SEM results for polished pieces of ore. The surface of the ore sample appeared uniform (clear area) with the presence of cavities (dark areas) attributed to defects or impurities [10, 11]. Magnification (2000×) of the image in Fig. 3, *a* showed that the deposit was granular, porous, and contained nanoneedles (Fig. 3, *b*).

The porosity of the sample could facilitate hydrometallurgical treatment for impurity removal [12]. The presence of



Fig. 1. Sample processing steps

Table 1

Chemical composition of the ore sample

Element	Fe	Р	Ca	Mn	Al	Si	Mg
Mass fraction (%)	52.42	0.61	1.45	0.29	4.95	5.11	1.07



Fig. 2. XRD pattern of the Gara Dejbilet iron ore with labeling of major peaks corresponding to specific minerals



Fig. 3. SEM and EDS analysis: a – SEM observation of polished raw ore; b – the deposit was

a – SEM observation of polisnea raw ore; b – the aeposit was granular, porous

nanoneedles in the ore sample was interpreted by several authors as an indication of the presence of goethite. These authors showed that thermal dehydration between 200 and 400 °C did not change the geometry of the needles in goethite samples [2, 13]. EDS analysis performed on different parts of the sample surface indicated that the clear zone was mainly composed of iron (Fig. 4, *a*). The dark zone contained phosphorus impurities, aluminum, and silicon (Fig. 4, *b*). Additionally, calcium, potassium, and oxygen were present (Figs. 4, *a* and *b*). The low content of alkaline oxide (K₂O) could indicate a low percentage of flux [14]. It is well known that Si and Al in iron ore are present as quartz, alumina kaolinite, aluminosilicate, and other silicates [8]. The correlation between Al and Si suggested that these elements in their oxide forms were derived from one mineral phase (clay) [15].

Mineralogical phases of the roasted ore. The influence of the calcination temperature (600-900 °C) on the phase trans-



Fig. 4. EDS of polished raw ore for (a) dark and (b) clear areas shown in Fig. 3

formation of Gara Djebilet ore samples was investigated (Fig. 5). The XRD patterns indicated that no obvious changes were observed despite the increase in the heating temperature. Additionally, as the temperature increased, the intensity of the peak for hematite increased considerably while that for goe-thite decreased (Fig. 5).

This transformation occurred at all roasting temperatures, but the optimum reduction occurred at 900 °C when the ratio of Fe_{Hematite}/TFe peaked at 98.9 % (Fig. 6). However, according to several authors, roasting at higher temperatures induces crystallization of the spinel phase of silicon, which is highly resistant to acid attack [16, 17]. Thus, 800 °C was selected as the optimum roasting temperature, which was consistent with the results from several other studies [16]. This increase in XRD hematite peaks occurred because of the transformation of goethite (FeO(OH)) to hematite (α -Fe₂O₃) with water removal, which was followed by dehydroxylation of iron oxyhydroxides [13].

The principal steps during the production of goethite and hematite are well known by the scientific community. However, the mechanism of the solid-state transformation of goethite to hematite is not fully understood yet. Nevertheless, based on thermogravimetric and differential thermal analysis, Gialanella, et al. concluded that the dehydration reaction de-



Fig. 5. XRD patterns of roasted ore samples



Fig. 6. Ratio of hematite in total iron as a function of the roasting temperature

picted in reaction 1 proceeds via structurally uncharacterized intermediates with the general formula $Fe_{2-x}O_{3-3x}(OH)_{3x}$ (where x = 0, α -Fe₂O₃, where x = 1, Fe(OH)₃ [2].

$$2\alpha - \text{FeOOH} \xrightarrow{T^{\circ}} \alpha - \text{Fe}_2\text{O}_3 + \text{H}_2\text{O}$$
 (1)

In the literature, this intermediate is referred to as hydrohematite or protohematite [2].

Two XRD peaks for maghemite-magnetite at approximately 63 and 43° disappeared completely at temperatures above 700 °C. The intensity of peak at 25°, also attributed to maghemite-magnetite, increased. These results could indicate that crystallographic transformation of maghemite-magnetite occurred with annealing, and/or that some of the maghemite-magnetite was transformed into hematite [4].

The intensity of the kaolinite peak at approximately $2\theta = 32^{\circ}$ decreased with increases in the temperature, and the XRD peak located at 39° disappeared completely at temperatures above 600 °C. According to the literature, kaolinite starts to decompose between 456 and 576 °C, and then hydroxyl groups are removed from the crystal network of the ore sample and the kaolinite (Al₂Si₂O₅(OH)₄) is transformed to metakaolinite with H₂O loss according to the following reaction [18]

$$SiAl_2O_5(OH) \xrightarrow{T^\circ} Al_2O_3 \cdot 2SiO_2 + 2H_2O$$
 (2)

Evaluation of multiple chemical leaching treatment steps. To remove or reduce the contents of phosphorus, aluminum, and silicon in the raw sample, the use of multiple chemical leaching and roasting steps was evaluated (L1, L2, L3 and L4; Fig. 1). The contents of gangue and iron were determined by XRF after each treatment step. The first experiment was carried out on the raw sample without roasting. The contents decreased to 3.36 % for silicon, 2.68 % for aluminum and 0.3 % for phosphorus after the L4 step (Fig. 7).

The results for the gangue contents were promising; however, after the second acid leaching step (L3), the iron content decreased to approximately 45 %. This low iron content would not be suitable for iron mining.

Next, we evaluated the process with pre-roasting of the sample at 800 °C before the chemical leaching steps (L1–L4) (Fig. 8). The phosphorus content was reduced to approximately 0.15 % and the aluminum and silicon contents were reduced to 4.2 and 5 %, respectively.

Additionally, the iron content increased from 52.4% to a stable value of 55.25% after leaching step L4. These results indicate that this process is promising for treatment of iron ore.

The changes in the silicon, aluminum, phosphorus, and iron contents after each chemical leaching and roasting step (roasting-L1-L2-roasting-L3-L4) are shown in Fig. 9. With this process, the phosphorus content decreased to 0.15% but the contents of aluminum and silicon increased. After leaching step L3, the iron content increased to 55.25%, but it then decreased to 54.25% after L4. The introduction of the calcination step after L2 appeared to be responsible for these results.

Although there has not been a thorough study of the processing of the oolitic iron ore deposit at the Gara Djebilet mine because of its complex mineralogical chemical characteristics, our results are very promising compared with those in the literature. Mansour, et al. reported a similar phosphorus content (0.16 %) and the Fe content of 96 % was higher than that obtained in this work; however, they used a high proportion of coal and a higher temperature (1,200 °C) [7]. In a recent study, Zajnullin, et al. evaluated the influence of the temperature and duration of magnetizing roasting and leaching with an aqueous solution of sulfuric acid on the iron content. This increased the iron content from 52.1 to 65.3 % and reduced the phosphorus content to 0.16 %, which were similar to the results obtained in other studies [7]. Additionally, Badjoudj, et al. studied the granulometry of the iron ore particles



Fig. 7. Changes in the iron, aluminum, silicon, and phosphorus contents after chemical leaching (L1-L4) of the raw ore



Fig. 8. Changes in the iron, aluminum, silicon, and phosphorus contents after chemical leaching (L1-L4) and pre-treatment of the raw ore by roasting

as a function of the $CaCl_2$ dose. For particle sizes between 0.045–0.063 mm, the phosphorus content was 0.06 % and



Fig. 9. Changes in the iron, aluminum, silicon, and phosphorus contents after chemical leaching and additional roasting of the sample (roasting -L1-L2-roasting -L3-L4)

iron content was 61.67 %. However, the content of the other gangue elements (Si and Al) were not measured [19].

Conclusion. Gangue (Si, Al and P) was removed from Gara Djebilet iron ore using acid and base leaching combined with roasting. The XRD results showed that roasting of the ore samples at a temperature between 600 and 900 °C increased the intensities of the XRD peaks for hematite and decreased those for goethite. These changes could be explained by the transformation of goethite to hematite. With roasting at a temperature above 700 °C, the peaks for maghemite/magnetite at 63 and 43° disappeared, and the intensity of the peak at 25° increased. These changes suggested that some of the maghemite/magnetite underwent a crystallographic transformation and some was transformed into hematite. Additionally, the XRD pattern suggested that kaolinite was transformed into metakaolinite at a temperature of approximately 600 °C. SEM observation of the surface of a sample from the ore deposit showed nanoneedles on the surface, which confirmed the presence of goethite.

After evaluating different orders for the treatment steps (Fig. 1), pre-roasting at 800 °C with two stages of chemical leaching steps (acid and base) was selected as the most effective. This process gave a low phosphorus content of 0.15 % and a high iron content of 55.25 %.

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Комбінована обробка випалюванням і вилуговуванням для зниження вмісту фосфору, алюмінію та кремнію в оолітовій залізній руді

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Мета. Підвищення якості оолітової залізної руди, яка видобувається на шахті Гара Джебіле на півдні Алжиру, шляхом зменшення вмісту кремнію, алюмінію та фосфору, що дозволить зробити її більш придатною для використання в металургійній промисловості.

Методика. Дослідження включає попередню переробку залізної руди шахти Гара Джебіле шляхом випалювання, за яким слідують два етапи вилуговування з окремими стадіями вилуговування в кислотному й лужному середовищі. Крім того, досліджено вплив уведення додаткового етапу випалювання при 800 °С між двома стадіями вилуговування.

Результати. Хімічне вилуговування сирої руди без попереднього випалювання призводить до зниження вмісту кремнію, алюмінію й фосфору з 4,45; 5,11 та 0,61 % до 2,68; 3,36 та 0,3 % відповідно. Однак уміст заліза зменшується з 52,42 до 45 %. Попередня переробка руди випалюванням у поєднанні із двома стадіями вилуговування зменшує вміст фосфору до 0,15 % і збільшує вміст заліза до 55,25 %. Вміст кремнію та алюмінію зменшується до 4,2 і 5 % відповідно. Уведення другого етапу випалювання між двома етапами вилуговування ще більше знижує вміст фосфору до 0,15 %, але вміст заліза зростає лише до 54,25 % після другого етапу вилуговування в кислому середовищі. Вміст алюмінію та кремнію збільшується до 4,5 і 5,3 % відповідно.

Наукова новизна. Це дослідження впроваджує новий підхід до підвищення якості оолітової залізної руди шляхом дослідження ефективності попередньої переробки шляхом випалювання з подальшим двоетапним вилуговуванням. Дослідження робить цінний внесок у розуміння ефективності цих методів щодо зменшення вмісту небажаних елементів у залізній руді.

Практична значимість. Отримані результати мають практичне значення для металургійної промисловості, пропонуючи потенційні методи підвищення якості залізної руди шахти Гара Джебіле. Упровадження цих методів може сприяти підвищенню ефективності й рентабельності переробки залізної руди, що в кінцевому рахунку принесе користь процесам виробництва сталі.

Ключові слова: оолітові залізні руди, вилуговування, випалювання, видалення фосфору

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