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Recovery of Iron from Bauxite Red Mud by Reduction Roasting Method

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Abstract

Red mud is an important solid tailing with strong alkalinity that is obtained during the extraction of alumina in the Bayer process. The global reserve of red mud is more than 4 billion tons, and its disposal as tailing has always been a serious environmental problem. This tailing is considered as a potential source, due to its high content of valuable metal compounds including iron. In this research work, the extraction of iron in red mud is investigated by the method of reduction roasting. The main influencing factors are also investigated. These methods include reduction in muffle and tube furnace, and temperature, reduction agent, and additive type are as important factors. Reduction roasting of the samples in a tube furnace, with Argon gas and vacuum, a mixture of red mud, graphite, and sodium carbonate at 700–1000 °C results in the formation of Fe3O4. Magnetic measurements indicate that saturation magnetization increases from 0.239 to 38.205 emu/g due to the formation of Fe3O4. Applying the magnetic field intensity of about 1000 Gauss results in the iron recovery of 89.9%.

1. Introduction

Iron is the fourth most common element on the earth, the most used among metals, and it accounts for more than 90% of metal production worldwide. Red mud, which is the solid tailings from the Bayer process, contains 30-60% iron depending on the quality of bauxite, which is usually more than many low-grade iron ores, so red mud can be considered as an iron ore, and red mud with the small particle size does not need to be crushed or milled, so its production cost is lower than iron ore processing. The residue from the Bayer process is called red mud. This residue is obtained from the effect of sodium hydroxide on bauxite and generally includes aluminum oxides, silica, calcium compounds, iron oxides, titanium oxide, and rare earth elements. This substance is strongly alkaline, and its pH varies between 10 and 12 [1]. The amount of red mud produced in the Bayer process depends on various factors such as the amount of alumina in the composition of bauxite, the composition and characteristics of bauxite ore, the temperature of leaching, and the conditions of its ore processing but, in general, the average amount of red mud produced per one ton of alumina production is about 1-1.5 tons [2]. Red mud stored in the world is estimated to be more than 2.7 billion tons with an annual growth rate of 120 million tons [3]. It is very difficult to maintain and store red mud due to its strong alkaline properties and the large volume of its production. Various methods have been used in the world to store and maintain red mud but none of them are favorable from an environmental point of view. In this research work, the intention was to study, test, and investigate a method with high recovery to extract iron from the red mud of Jajarm alumina complex, Iran.

Liu *et al.* (2009) have investigated the recovery of iron from barren red mud by roasting process with direct reduction and then magnetic separation.

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After analyzing the chemical composition and crystalline phase, the effects of different parameters on iron recovery have been done. The optimal reaction parameters are suggested as follows: the ratio of carbon powder to red clay is 18:100, the ratio of additives to red clay is 6:100, roasting at 1300 degrees for 110 minutes, with these optimal parameters, the total content of iron in the concentrated material is 88.77%., the ratio of metallization was 97.69%, and the recovery ratio was 81.40% [4]. Zhou *et al*. (2012), in a research work to recover iron from red mud, used the roasting-magnetic separation method with sodium carbonate reduction. The effects of sodium carbonate concentration, reduction temperature, and time on the quality of the final product and phase changes in the reduction process have been fully investigated. The results showed that the final product has iron with a grade of 90.87% and Al_2O_3 with a grade of 0.95% at this stage with a metallization degree of 94.28%. Reduction was done in the roasting stage at 1050 degrees Celsius for 80 minutes. Finally, magnetic separation was performed for particles smaller than 0.074 mm with a magnetic field intensity of 0.08 Tesla [5]. Rao *et al*. (2016) presented an effective method for the comprehensive utilization of red mud, which mainly focused on the first step of iron recovery from red mud with high iron content by reduction roasting-magnetic separation. During the reduction, iron oxides were reduced to metallic iron with the aid of sodium sulfate and sodium carbonate. Effects of the dosages of sodium sulfate and sodium carbonate, roasting temperature and roasting time on the metallization ratio of iron of roasted product, total iron grade, and iron recovery of magnetic concentrate were primarily investigated. In the presence of 6% Na_2S0_4, 6% Na_2C0_3, and optimal reduction roasting-magnetic separation conditions: roasting temperature of 1050 ˚C, roasting time of 60 min, magnetic separation feed fineness of 90% passing 74 ~m and magnetic field intensity of 0.1 T, a magnetic concentrate containing 90.12% iron with iron recovery of 94.95% was obtained from a red mud containing 48.23% total iron, 7.71 % AI_20_3 and 7.69% $Si0_2$. [6]. Chan *et al*. (2017) found that since red mud is a tailing produced during the production of alumina from bauxite; the reduction was carried out at high temperature of red mud in the absence and presence of sodium borate to facilitate the subsequent recovery of iron by further magnetic separation. Sodium borate increases the recovery of iron and also significantly increases the size of metallic iron particles. High-temperature reduction in the presence of 4% sodium borate increased the iron content of the metal powder to 90.05%, compared to 80.24% without sodium borate under the optimal annealing conditions at 1300 ˚C for 30 min. Experimental evidence showed that sodium borate reduces iron oxides and grows iron metal grains, leading to improved separation between iron and gangue during subsequent magnetic separation [7]. Panomar *et al*. (2018) conducted research on the synthesis and magnetic properties of magnetite prepared by chemical reduction of hematite with different particle sizes. magnetite was synthesized by hematite reduction at a temperature of 500 °C in an atmosphere of carbon monoxide. The source hematite was separated initially into ten particle size categories ranging from 0.05 to 2.5 mm. The reduction was carried out with carbon monoxide, which was synthesized by reaction of air and activated carbon at 750 ˚C. It was determined by X-ray diffraction analysis and magnetometry that, after reduction, samples of each particle size consisted mainly of magnetite. In addition, the magnetic properties of the resulting magnetite were noted to be somewhat different from those of natural magnetite. In particular, remanence and coercivity are higher for synthetic magnetite than for natural magnetite. The saturation magnetization of transformed samples increases to 60-80 Am^2/kg, while the source hematite does not have strong magnetic properties. Hematite with a particle size from 0.05 to 0.25 mm is completely converted into magnetite but if the size of hematite particles increases to from 0.25 to 2.5 mm, incomplete conversion of hematite to magnetite is observed. The obtained results are important for the development of technology for converting hematite to magnetite, which can, in turn, be used to beneficiate iron ores [8]. Agrawal *et al*. (2018) have conducted specific pre-treatment process with an incorporation of a primary magnetic beneficiation prior to reduction roasting followed by secondary magnetic beneficiation manifest optimal conditions for iron recovery and proved economical in terms of specific consumption of reductant. Carbonated red mud concentrated with magnetic separation followed by muffle furnace roasting using suitable reductant produced a 50.5% iron-rich concentrate with 70% iron recovery. This technique of iron recovery from red-mud consumes two harmful industrial wastes and shows promising results [9]. Sadangi *et al*. (2018) successfully used the regenerative cleaning method followed by magnetic separation in iron research. During the process, the hematitic and goethitic iron-phase minerals present in the red mud sample are converted into magnetite and metallic irons, which are subsequently recovered using low-intensity magnetic separator. The results showed that an iron recovery of 61.85% with an iron content of 65.93% of iron concentrate could be obtained at roasting temperature of 1150 °C, roasting time of 60 min, and magnetic field intensity of 0.18 Tesla. [10]. Li *et al.* (2019), considered the Bayer Red Mud as a low-grade iron ore with a grade of 5wt% to 20wt% iron. We adopted the reduction roasting-magnetic separation process to recover ferric from red mud by electromagnetic induction furnace. The effects of different parameters on the recovery rate of iron were studied in-depth. The optimum reduction reaction conditions were obtained that is 1wt% of carbon in red mud at 1450 °C roasting for 60 min and the magnetic field intensity is about 0.19T. The experimental results indicated that the grade of total iron and the iron recovery were 66.50% and 65.4%, respectively. The prove the electromagnetic induction furnace is more beneficial to iron recovery [11]. Mumbley *et al.* (2019) used a mixture of pure graphite and blast furnace slag as a reducing agent with different carbon concentrations, and For all the conditions tested, the metallization degree was higher than 70%, and the best condition to reduce red mud through blast furnace sludge was identified at 1:1 red mud/blast furnace sludges equal to 0.85 C/Fe_2O_3 [12]. Jain *et al.* (2021) had a creative use of red mud through simultaneous reduction with coal tailings for the separation and recovery of iron and aluminum minerals. Under the optimal conditions of simultaneous reduction, an iron concentrate containing 57.25% of total iron content with a recovery of 65.22% and an aluminum-rich product containing 27.26% of Al_2O_3 with a recovery of 71.37% had been obtained after magnetic separation [13]. In 2021, Wan *et al.* investigated the effects of manganese-rich iron ore in the direct reduction process of iron-rich red mud through various tests such as reduction temperature, time, C/Fe mass ratio and the amount of manganese-rich iron ore added. The results showed that the rate and amount of metallization caused by the direct reduction of red mud rich in iron at 1150 degrees of heat, 150 minutes, and a mass ratio of C/Fe equal to 2 reached 84%. Due to simultaneous reduction, with 60% iron ore rich in manganese, the level of iron enrichment was increased to 93.52% [14]. In this research work, reduction roasting method in muffle and tube furnaces under various conditions was investigated to achieve the most iron recovery from red mud.

2. Materials and methods

The red mud used in this research work was obtained from Jajarm Alumina Complex, Iran. According to the XRD analysis in Figure 1, the main minerals of red mud include katoite, hematite, calcite, chamosite, anatase and quartz. A series of other minerals can also be found in some places. The result of XRF analysis is also presented in Table 1.

Table 1. Main chemical composition of red mud/wt% (XRF).

Element	Al_2O_3	SiO_2	CaO	Fe_2O_3	Na_2O	TiO_2	Others
Content	16.56	13.09	17.87	25.5	4.58	5.48	14.05

2.1. Experimet method

First, three groups of experiments were designed to investigate the effect of different parameters on iron recovery from red mud including reduction temperature, reduction agents and additive type. The representative red mud sample was mixed with carbon as a reducing agent with a reaction stoichiometric ratio of 1-3 (Eq. 1) and an additive with a weight ratio of 0.06 to red mud [15].

$$Fe_2O_3 + 3C \rightarrow 2Fe + 3CO \quad (1)$$

The prepared powder was poured into a porcelain crucible and placed in a muffle furnace for reduction. After 120 minutes, the reduced samples remained in the furnace until they reach room temperature. Then the samples were removed from the furnace and analyzed by wet chemical method. In the next step, according to the results of reduction tests in the muffle furnace, two different test groups were designed in the tube furnace under vacuum and under Argon at different temperatures (Table 2). The representative red mud sample was completely mixed with graphite as a reducing agent with a stoichiometric ratio and sodium carbonate with a weight ratio of 0.06 to red mud. The prepared powder was poured into a porcelain crucible and placed in a tube furnace for reduction. After 60 minutes, the reducted samples remain in the oven until they reach room temperature. Then the samples were removed from the furnace, and vibrating sample magnetometer (VSM) analysis was performed to determine the degree of magnetization.

Table 2. Test conditions in the tube furnace.

Test	T (°C)	Furnace conditions
1	700	Vacuum
2		Argon
3	800	Vacuum
4		Argon
5	900	Vacuum
6		Argon
7	950	Vacuum
8		Argon
9	1000	Vacuum
10		Argon

2.2. Test equipment

Reduction roasting in this research work was done in the muffle and tube furnaces.

3. Results and Discussion

3.1. Effect of temperature on iron recovery in muffle furnace

Temperature is one of the main factors affecting iron reduction and recovery. A mixture of red mud, carbon with considering stoichiometric ratios and additives equal to 0.06 in a period of 120 minutes at temperatures of 500, 600, 700, 800, 900, and 1000 °C was reducted. Figure 3 shows the effect of temperature on iron recovery.

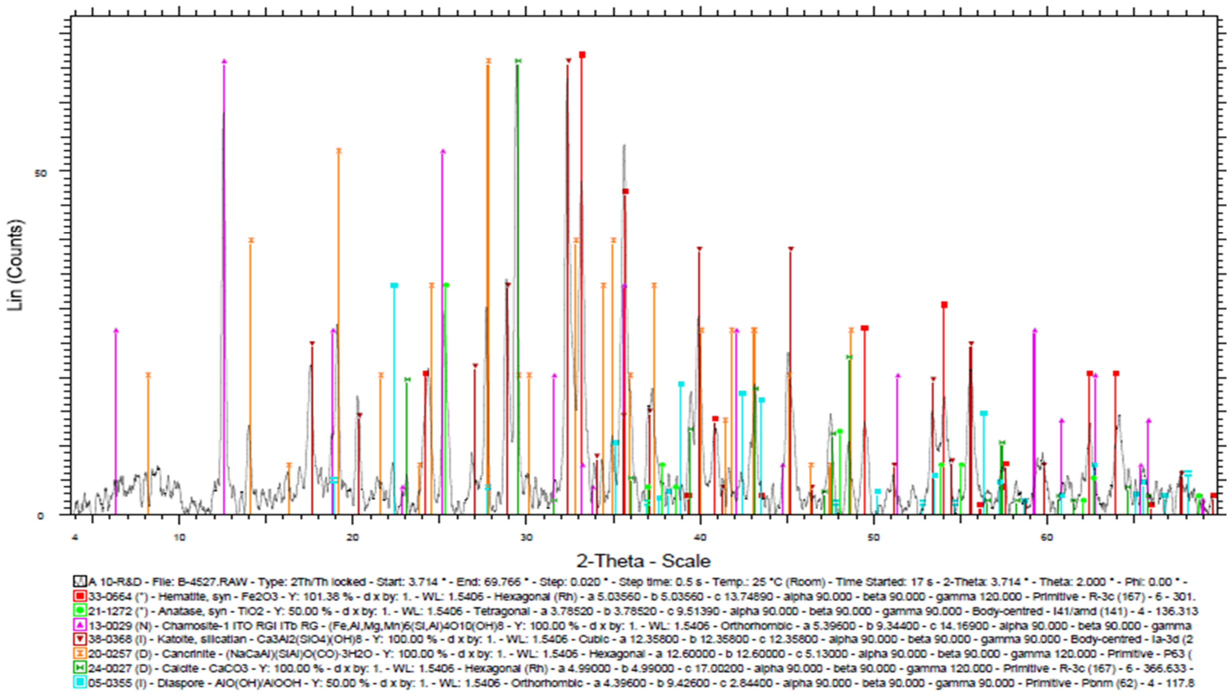

Figure 1. XRD pattern of the red mud.

As shown in Figure 3, temperature had a positive effect on the recovery of iron from red mud, and recovery value increased as the temperature increased. Since reduction roasting is an endothermic reaction, increasing the temperature is an effective way to improve the reduction of iron oxides. The higher the reduction temperature, the higher the iron grade and iron recovery to the concentrate. The reduction rate usually increases with increasing temperature, but a very high temperature will cause overreduction, thus resulting in the formation of weakly magnetic wustite (FeO) or fayalite (Fe_2SiO_4). These minerals are unresponsive to LIMS (low intensity magnetic separator), and lead to a decrease in iron recovery.

3.2. Effect of type of additive on iron recovery

To investigate the effect of the type of additive on the amount of iron recovery, three experiments were conducted with calcium carbonate, sodium carbonate as additives, and one experiment without additives. The results of the tests are presented in Figure 4.

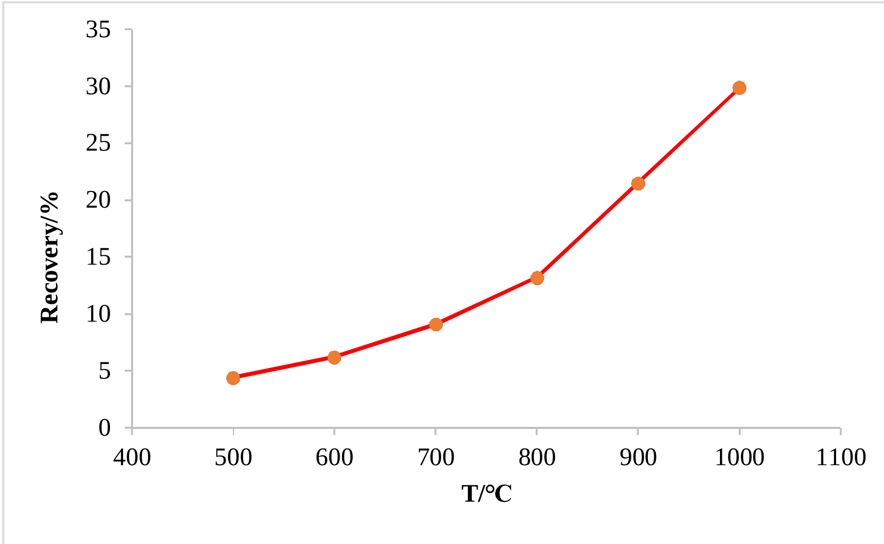

Figure 2. Effect of the roasting temperature on iron recovery.

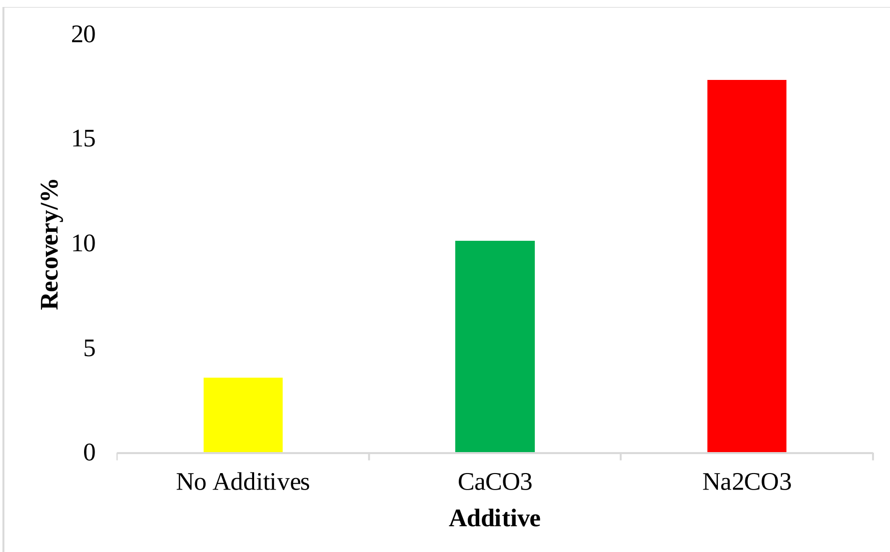

Figure 3. Effect of additive on iron recovery.

As shown in Figure 4, the presence of the additive had a significant effect on the recovery and reduction process, and sodium carbonate had a greater effect on iron recovery than calcium carbonate. Both $CaCO_3$ and Na_2CO_3 additions produce CO_2 gas, which is based on Equation 2 for CO reduction gas production.

$$CO_2 + C \rightarrow 2CO(g) \quad (2)$$

As seen in the XRF analysis of the red mud sample, we have Fe_2O_3 and SiO_2 minerals at high temperatures. The reactions between FeO and silica and aluminum would be improved. Fayalite or hercynite would be formed, which prevent the reduction of red mud. On the other hand, the sodium present in sodium carbonate reacts with Al_2O_3 and SiO_2 and forms sodium silicate and sodium aluminosilicate, which prevents the formation of fayalite, so sodium carbonate is the most effective additive in the red mud reduction process.

3.3. Effect of reducing agent on iron recovery

To investigate the effect of the reduction agent on the measure of magnetization, four tests were conducted with coking, semi-thermal, thermal coal, and graphite at a temperature of 1000 °C and a time of 120 minutes. The results of the tests are given in the Figure 5.

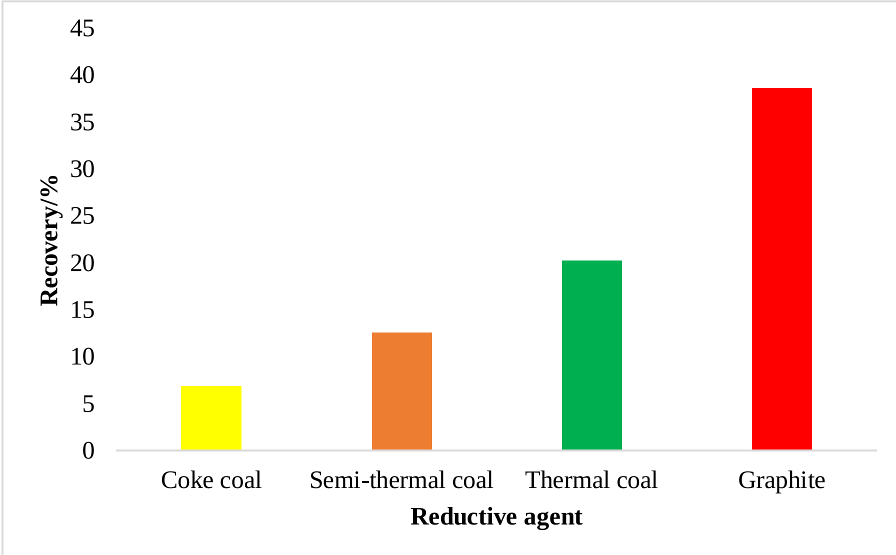

Figure 4. Effect Reductive agent on iron recovery.

As shown in Figure 5, when graphite is used as a reducing agent in the reduction process, iron recovery has increased significantly compared to coking, semi-thermal, and thermal coal. CO reduction gas is needed for the reduction process but because it was not possible to use a CO capsule due to laboratory limitations, so carbon was used as the reduction agent. Graphite was more suitable than thermal and semi-thermal coals; on the other hand, although the percentage of carbon in coking coal is higher than thermal and semi-thermal coals, coking coal at high temperatures reduces gas production and hinders the reduction process due to its high plastometer. Also due to its high lubrication properties, graphite reduces adhesion and improves the reduction process.

3.4. Effect of temperature on measure of magnetization in a tube furnace under vacuum

VSM is a scientific instrument that measures the magnetic properties based on Faraday's Law of Induction. A sample is first placed in a constant magnetic field and if the sample is magnetic it will align its magnetization with the external field. The magnetic dipole moment of the sample creates a magnetic field that changes as a function of time as the sample is moved up and down. This is typically done through the use of a piezoelectric material. The alternating magnetic field induces an electric field in the pickup coils of the VSM. The current is proportional to the magnetization of the sample - the greater the induced current, the greater the magnetization. As a result, typically, a hysteresis curve will be recorded and from there the magnetic properties of the sample can be deduced. Figure 6 shows the sample heat treated at various temperatures. It can be seen that the reduction process proceeds with increasing the heat treatment temperature from 700 to 900 °C and decreased from then on.

According to Boudward's diagram, the reaction of converting Fe_2O_3 to Fe_3O_4 and then converting Fe_3O_4 to metallic iron depends on the ratio of %CO to %CO_2, so the suitable temperature for different reduction modes in different furnace conditions and gas type is different. The appropriate temperature should be found by designing the experiment, at a temperature lower than this value, there is incomplete regeneration and at a temperature higher than this temperature, the product (Fe_3O_4) may be over-reduced to generate wustite which is weakly magnetic and difficult to recycle.

3.5. Effect of temperature on measure of magnetization in a tube furnace under Argon

The mixture of red mud, graphite, and sodium carbonate was reduced with Argon under the same conditions as the previous step. Figure 7 shows the sample heat treated at various temperatures. It can be seen that the reduction process proceeds with increasing the heat treatment temperature from 700 to 1000 °C.

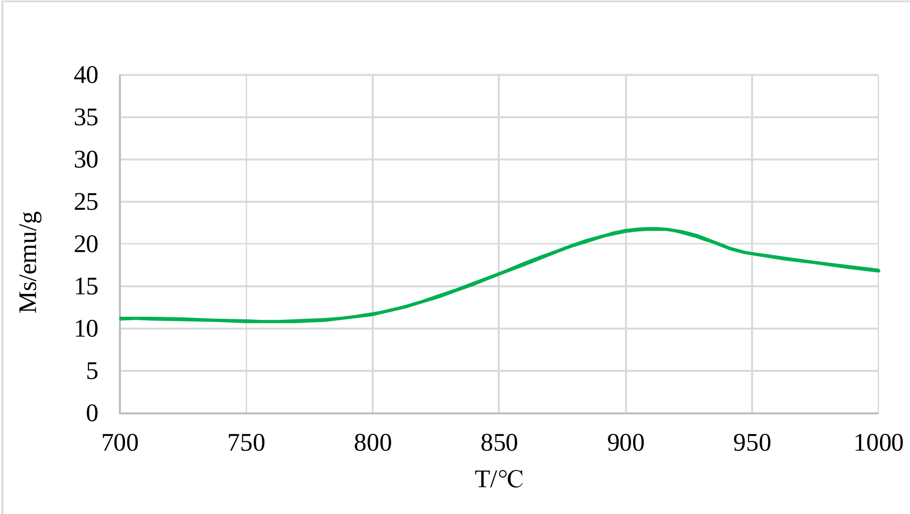

Figure 5. Effect of temperature on saturation magnetization in vacuum.

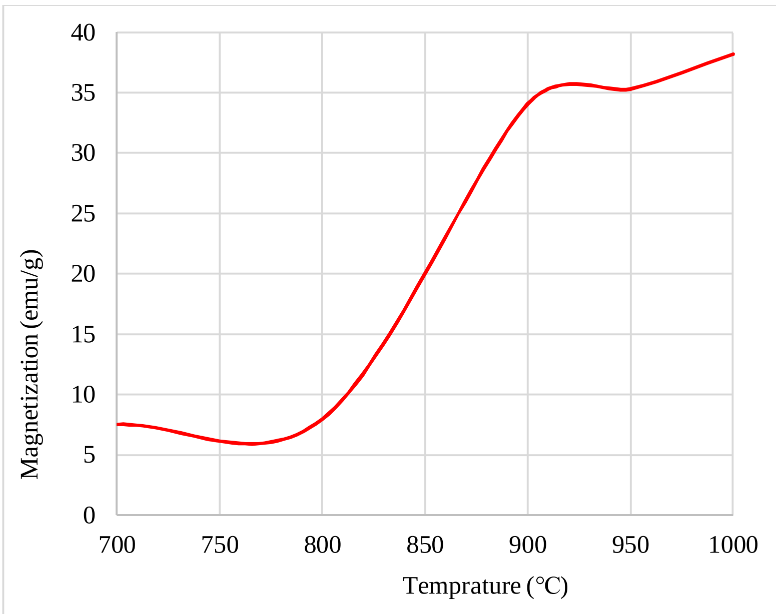

Figure 6. Effect of temperature on saturation magnetization under Argon.

Figure 8 shows the magnetic hysteresis loops of the samples, after heat treatment at 700 to 1000 ˚C in vacuum. Heat treatment sample resulted an increase in saturation magnetization to 900 ˚C from 0.239 to 21.538 and decreaded to 16.834 emu/g at 1000 ˚C.

Figures 9 shows the magnetic hysteresis loops of the samples, after heat treatment at 700 to 1000 ˚C under Argon gas. Heat treatment sample resulted an increase in saturation magnetization to 1000 ˚C. It can be seen that raising the heat treatment temperature from 700 to 1000 ˚C increases the saturation magnetization from 0.239 emu/g of powder to 7.534, 7.976, 34.08, 35.316 and 38.205 emu/g for heat treated samples at 700, 800, 900, 950, and 1000 ˚C, respectively.

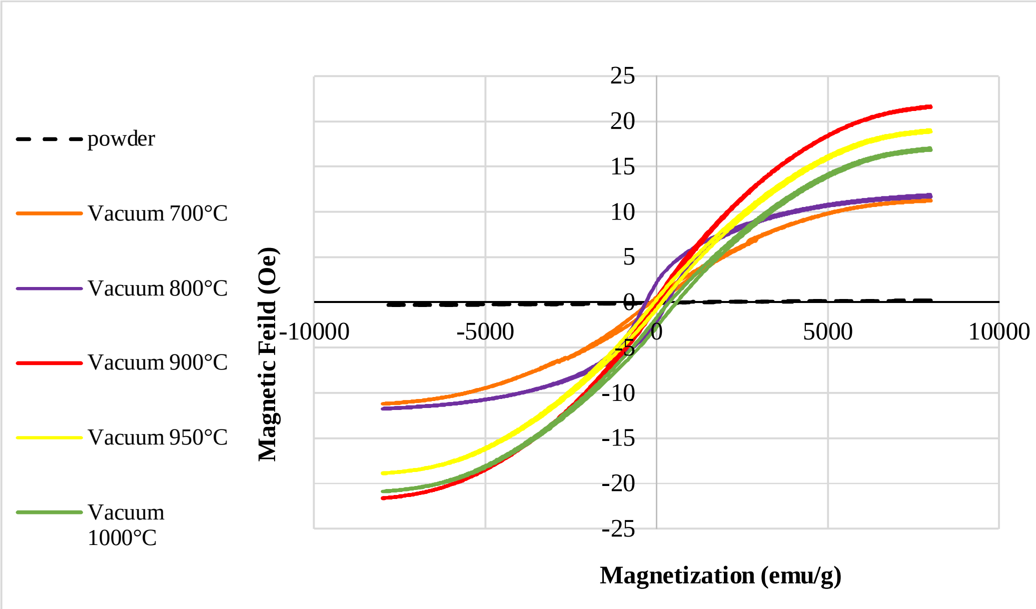

Figure 7. Hysteresis loops of powder and heat treated at 700, 800, 900, 950, and 1000 ˚C in vacuum.

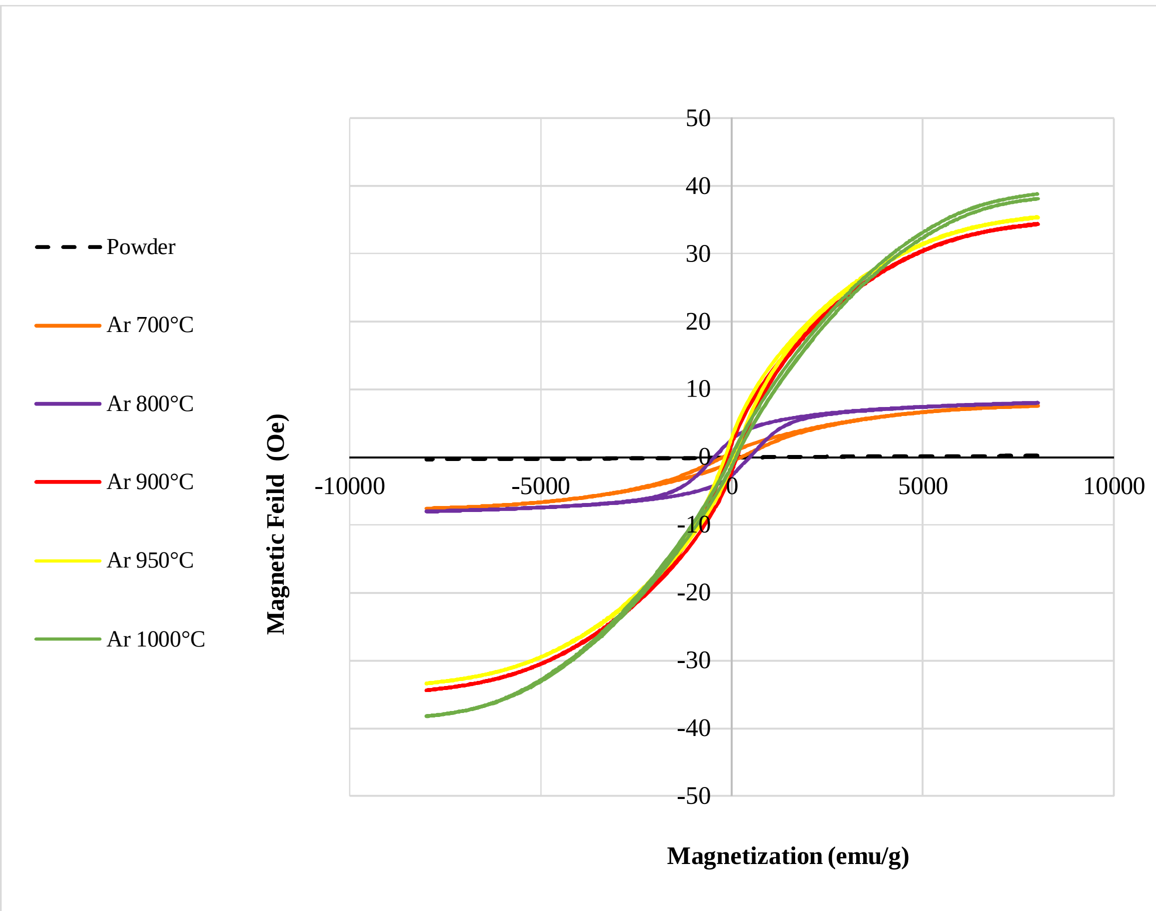

Figure 8. Hysteresis loops of powder and heat treated at 700, 800, 900, 950, and 1000 ˚C under Argon

3.5. Effect of furnace type on saturation magnetization of red mud

Comparison of VSM changes at different temperatures in muffle furnace, tube furnace under vacuum, and Argon is shown in Figure 10.

The furnace has a great influence on the reduction process, as you can see in Figure 10, there is no high saturation magnetization in the muffle furnace even at high temperatures. The reason for this is that in the muffle furnace, even when covered porcelain crucibles are used, oxygen cannot be prevented from entering the furnace chamber. Therefore, as the temperature increases, regeneration takes place but at the same time as the temperature decreases, due to the presence of oxygen, the samples are oxidized again. But this does not happen in the tube furnace, because first we create a vacuum, then we vent the vacuum by opening the argon gas, this is repeated 5-6 times to finally ensure the absence of oxygen inside the tube.

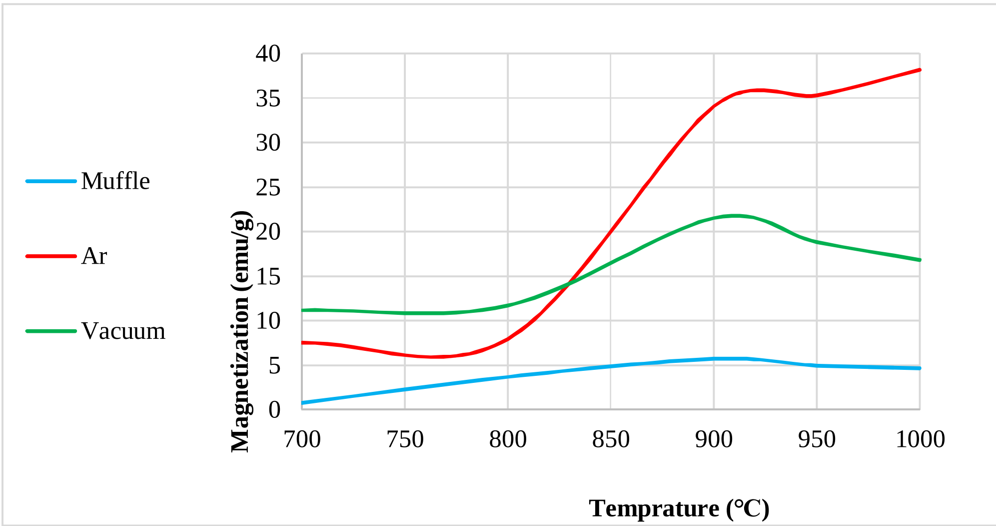

Figure 9. Effect of furnace type on saturation magnetization of powder.

4. Conclusions

The following results were obtained:

1. The important factors affecting iron recovery from red mud include temperature, type of additive, reduction agent, and furnace due to reduction.
2. In the reduction tests in the tube furnace under Argon, the magnetization rate increased significantly from 0.239 to 38.205 with increasing temperature. But under vacuum, the measure of magnetization increased up to the temperature of 900 ˚C from 0.239 to 21.538 emu/g and decreased from then on to 16.834 emu/g.
3. By comparing the results obtained in the muffle and tube furnace, it was concluded that the tube furnace with saturation magnetization 38.205 emu/g is much more suitable for reduction than the muffle furnace with saturation magnetization 4.978 emu/g.
4. By comparing the results obtained in the tube furnace under vacuum and Argon, it was shown that the reduction under Argon gas with saturation magnetization 38.205 emu/g was better than under vacuum with saturation magnetization 21.538 emu/g.
5. Based on the optimum reaction conditions at 1000 ˚C roasting for 60 min in a tube furnace with Argon gas and the magnetic field intensity is about 1000 Gauss , magnetic separation concentrate can be obtained with 89.9% recovery of iron.

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استحصال آهن از گل قرمز بوکسیت به روش تشویه احیایی

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چکیده:

گل قرمز، باطله جامد مهم با خاصیت قلیایی بودن قوی است که در طی استخراج آلومینا در فرآیند بایر حاصل می‌شود. ذخیره جهانی گل قرمز بیشتر از 4 بیلیون تن است و دفع آن به عنوان یک باطله جامد همواره یک مشکل زیست محیطی جدی بوده است. این باطله به دلیل محتوای بالای ترکیبات فلزی با ارزش از جمله آهن یک منبع بالقوه در نظر گرفته می‌شود. در این پژوهش به بررسی استحصال آهن موجود در گل قرمز به روش تشویه احیایی پرداخته می‌شود، همچنین فاکتورهای اصلی تاثیر گذار نیز بررسی می‌شود. این روش‌ها شامل احیا در کوره‌های مافل و تیوبی و فاکتورهای مهم دما، عامل احیا، نوع افزودنی می‌باشد. تشویه احیایی نمونه‌ها در کوره تیوبی، با گاز آرگون و خلاء، مخلوطی از گل قرمز، گرافیت و کربنات سدیم در دمای 700 تا 1000 درجه سانتی گراد منجر به تشکیل Fe_3O_4 شد. اندازه گیری‌های مغناطیسی نشان داد که مغناطش اشباع از 0,239 به 38,205 emu/g به دلیل تشکیل Fe_3O_4 افزایش یافت. اعمال شدت میدان مغناطیسی حدود 1000 گاوس منجر به بازیابی آهن 89,9٪ شد.

کلمات کلیدی: گل قرمز، تشویه احیایی، کوره مافل، کوره تیوبی، مغناطیس سنج نمونه ارتعاشی.