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Effect of NaOH and Thermal Treatment on Purifying Iron-Rich Hematite Ore

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Abstract

The current work provides an easy fast route to extract iron oxide from low-grade iron ore. The method deals with one simple method to get rid of three compounds silica, alumina and manganese present with iron oxide in the ore. It depends on mixing the ore with sodium hydroxide, and heating it to interact with the existing elements and not with iron. The treatment is repeated twice to improve the results. XRF, SEM, and EDAX were employed to characterize untreated and treated ore. The current method succeed to get rid of 86.4% of silica, 87.4% of Al_2O_3, and 74.6 % of MnO. 75.8 % increasing in Fe_2O_3 concentration was recorded. The ore second treatment with 1 M of sodium hydroxide and calcination temperature at 550 ºC for one hour was considered optimal condition for this study. EDAX results show an observable increase in the iron percentage after treatment (36%). These results are in a good agreement with results obtained by XRF and SEM. SEM images, before and after the treatment, show a significant difference. An increase in the concentration of spherical particulate aggregates of Fe_2O_3 was found, which represent the majority of the sample.

Keywords: iron oxide extraction, purify iron ore, sodium hydroxide, silica, alumina and manganese..

1. Introduction

The iron ores are prevalent in the Eastern and Western Deserts of Egypt, according to the Egyptian Mineral Resource Authority. The iron ore contains iron oxide deposits of a range from 38 to 55%; then they have economic value [1]. Besides, the extracted iron oxides are playing an essential role in various disciplines. The investigations of these oxides may be required for the particular utilization in real, environmental and industrial chemistry, soil science, corrosion science, mineralogy, planetology, biology and medicine [2]. Several well-known forms of iron oixdes are provided naturally in essential ores such as magnetite, hematite, goethite, limonite, siderite,...etc [3-5]. Hematite is the earlier iron oxide and is extensive in rock and soil and identified as ferric oxide, iron sesquioxide, red ochre, martite and kidney ore among others [2]. Iron (III) oxides have received much attention, not only on the industrial level but on all economic and strategic levels, since it is mainly applied in iron steel manufacture. Moreover, it is considered one of the most important materials utilized in other industries such as ceramics, glass, paints, rubber, plastic, paper, and building materials industry and more [3,6-7].

The quality and quantity of iron and steel are the main symbols of development degree and economic strength in a country [8]. Commonly, iron is recovered from high-grade ores via conventional flotation and pyrometallurgical methods, but it is not facile to recover iron from low-grade iron ores or low-grade iron ore by the conventional method [9]. The increased need for iron in the steel-making industries and the depletion of high-grade iron ores have led to the substitutional extraction of iron from low-grade ores [10-12]. Iron ores of low-grade have higher grades of silica and alumina as gangue mineral agglomerates with little amounts of phosphorus oxides. They are utilized as feed in steel manufacture; although, these impurities harmfully influence the manufacture of iron and steel [13]. In blast furnaces, a greater volume of highly viscous slag is formed due to the presence of high silica and alumina that need more flux. Consequently, removing these impurities before feeding into the steel-making operation is necessary [10]. Haga et al. [10] studied alumina and silica removal from hematite-based low-grade iron ore by wet magnetic separation of high-intensity and reverse flotation methods. Ke-qing et al. [14] utilized deep reduction and magnetic separation technique to bring out iron from oolitic iron ore. They used raw coal with a fixed carbon of 66.54% as a reductant. The extracted iron was obtained under the condition of 1200 ºC and 60 min and coal content of 30%. Gao et al. [15] separated iron and manganese from Fe-Mn ores utilizing carbothermal reduction and magnetic separation. They recorded higher temperatures (above 1200 °C) and shorter roasting reduction time during the carbothermal roasting reduction process of the Fe-Mn ore. They attributed the poor separation efficiency of Mn and Fe from Mn-Fe ore during the carbothermal reduction process was due to the formation of composite oxide phase $Mn_{1-x}Fe_xO$ and unreduced hercynite ($FeO.Al_2O_3$). All of the mentioned methods are expensive, as they consume a lot of time, equipments, and chemicals. Therefore, finding economic effective alternative methods are necessary. Sarkar. [13] used NaOH and different acids via a hydrometallurgical process to leach alumina and silica from iron ore. He informed that NaOH was the most efficacious leachant. The leaching process was conducted at low temperatures, i.e. 30, 50, 70, and 95 °C, and high

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concentrations of NaOH. He recorded optimal conditions at 95 °C and 5 M of NaOH for 2h, which prompted us to conduct the experiments at room temperature (more favourable condition), in addition to employing low concentrations of NaOH. Removal of manganese was not included by Sarkar's research and utilization of ore containing lower impurities of silica and alumina was conducted. The current experiments were conducted at room temperature

The current research aims to design a simple method to get rid of three compounds; silica, alumina and manganese in one step. The method depends on mixing iron ore with sodium hydroxide and heating to interact with the elements present with iron that have the ability to interact with sodium hydroxide and not with iron, then washing and separating the dissolved compounds in one step and we get rid of three impurities present with iron. The optimization process was conducted to obtain an optimized condition of purifying process by studying the effect of NaOH concentration and calcination temperature.

2. Experimental

2.1 Materials

The iron ore, Hematite, was collected from Bahariya Oasis, Egypt. The chemical composition of the ore was described in Table 1. The utilized sodium hydroxide pellets were laboratory reagent grade (Fluka ≥ 97 %).

Table 1, the elemental composition of the ore before treatment

Main Constituents	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	K_2O	Na_2O	P_2O_5	BaO	LOI
Percentage %	18.18	10.05	39.20	8.11	0.13	1.50	0.09	0.67	0.14	3.65	18.23

2.2. Sample Preparation and separation procedure

Nine samples were prepared to investigate the proposed method. Sample A was for the ore as a raw material after being grinded. Table 1 shows the elemental analysis of the grinded sample using an X-ray fluorescence (XRF) spectrometer. 25 grams of sample A was homogeneously mixed with 750 ml of sodium hydroxide solution (lower concentration; 0.5 M) on three steps for about 20 minutes at room temperature followed by filtration and washing (hot water) several times, then calcinated at 350 °C for a period of one hour to obtain the dry precipitate (sample B).

The experiment was repeated by taking the precipitate, sample B, adding to sodium hydroxide solution (the same concentration), on three steps for about 20 minutes at room temperature followed by filtration and washing (hot water) several times, then calcined at 350 °C for a period of one hour to obtain the dry precipitate (sample C).

25 grams of sample A was added to sodium hydroxide solution (the same volume and concentration), on three steps for about 20 minutes at room temperature followed by filtration and washing (hot water) several times, then calcinated at 550 °C for a period of one hour to obtain the dry precipitate (sample D).

The experiment was repeated by taking the precipitate, sample D, adding to sodium hydroxide solution (the same volume and concentration), on three steps for about 20 minutes at room temperature followed by filtration and washing (hot water) several times, then calcinated at 550 °C for a period of one hour to obtain the dry precipitate (sample E).

25 grams of sample A was homogeneously mixed with 750 ml of a higher concentration of sodium hydroxide solution (1M). The temperature was raised on three steps for about 20 minutes followed by filtration and washing (hot water) several times, then calcinated at 350 °C for a period of one hour to obtain the dry precipitate (sample F).

The experiment was repeated by taking the precipitate, sample F, adding to 750 ml sodium hydroxide solution (the lower concentration), on three steps for about 20 minutes at room temperature followed by filtration and washing (hot water) several times, then calcinated at 350 °C for a period of one hour to obtain the dry precipitate (sample G).

25 grams of sample A was homogeneously mixed with 750 ml of a higher concentration of sodium hydroxide solution. The temperature was raised on three steps for about 20 at room temperature minutes followed by filtration and washing (hot water) several times, then calcinated at 550 °C for a period of one hour to obtain the dry precipitate (sample H).

The experiment was repeated by taking the precipitate, sample H, adding to 750 ml of sodium hydroxide solution (the lower concentration), on three steps for about 20 minutes at room temperature followed by filtration and washing (hot water) several times, then calcinated at 550 °C for a period of one hour to obtain the dry precipitate (sample I). The chemical composition analysis of all samples was determined by XRF technique.

2.3 Techniques

The chemical composition of the ore sample was determined through the elemental analysis by using x-ray fluorescence (XRF Axios, sequential WD-XRF spectrometer, PANalytical 2005, Netherland). Grinding, pressing, and bead preparation of the investigated samples were carried. The microscopic pictures were taken from scanning electron microscope, JEOL-SEM (Quanta FEG 250), hyphenated with energy dispersive X-ray spectrometer (EDX). The spectra of elemental analysis by EDX were obtained on TEAM® software.

3. Results and discussions

3.1. Effect of calcination temperature on the purification process

XRF analysis was employed to determine the elemental composition of the ore and the purified samples. Table 2 shows all the elemental composition of the ore before and after some purification steps. It is obvious that the major compositions of the ore are Fe_2O_3 (39.2 %), SiO_2 (18.18 %), Al_2O_3 (10.05 %), and MnO (8.11%), respectively. It shows the effect of temperature and treatment frequency (for the same sample) on the efficiency of separating iron from its ore at the lower concentration of NaOH. The first treatment showed success in reducing the percentage of unwanted oxides associated with iron and increasing the percentage of iron oxide in the ore as a result of decreasing the other oxides, for both temperatures 350 °C and 550 °C. More superior, the second treatment, repeating the same experiments on the obtained product, showed more desirable results than that of the first one, for both temperatures 350 °C and 550 °C. Increasing the calcination temperature from 350 to 550 °C had a positive effect on getting rid of unwanted materials by a larger percentage, especially after the second treatment. Fig 1(a-d) shows the effect of temperature on increasing the percentage of iron oxide, and decreasing SiO_2, Al_2O_3, and MnO, in the sample after two consecutive treatments of the same sample and under the same conditions. For the second treatment, a successful removal of SiO_2 (56.8% and 70.3%), and of Al_2O_3 (44.5 % and 56.8 %), and of MnO (57 % and 69.5 %) were obtained at 350 and 550 °C, respectively. Moreover, Fe_2O_3% was increased to 43.5% and 54%, at 350 and 550 °C, respectively.

Table 2 the elemental composition of the ore before and after two consecutive treatments of the same sample and under the same conditions at calcination temperature 350 °C and 550 °C.

Main Constituents	Before treatment (A)	First treatment 350 °C (B)	Second treatment 350 °C (C)	First treatment 550 °C (D)	Second treatment 550 °C (E)
SiO^2	18.18	10.33	7.85	10.83	5.40
Al_2O_3	10.05	6.33	5.58	5.19	4.34
Fe_2O_3	39.20	40.98	56.24	56.37	60.53
MnO	8.11	4.78	3.46	4.65	2.47
MgO	0.13	0.09	0.09	0.09	0.08
CaO	1.50	1.21	1.29	1.33	1.25
K_2O	0.09	0.07	0.09	0.08	0.07
Na_2O	0.67	0.61	0.75	0.74	0.75
P_2O_5	0.14	0.05	0.05	0.06	0.03
BaO	3.65	3.30	3.48	3.66	3.57
LOI	18.23	22.10	20.7	20.45	21.46

3.2. Effect of NaOH Concentration on the purification process

Table 3 shows the elemental composition of the untreated and treated ore by different concentrations of NaOH at different temperatures. Fig 2 shows that the higher concentration of sodium hydroxide represents an effective role in increasing the iron oxide percentage in the raw material, in addition to decreasing the percentage of silica, alumina, and manganese oxide. It also becomes clear that increasing the temperature to 550°C in combination with the high concentration led to obtaining distinctive results, reaching a removal percentage of silica, alumina and manganese of 77.6 %, 75.3 % and 65.4%, respectively. It also increased the iron concentration to reach 65.4 %, as shown in the figure. Further experiments were done to optimize the results.

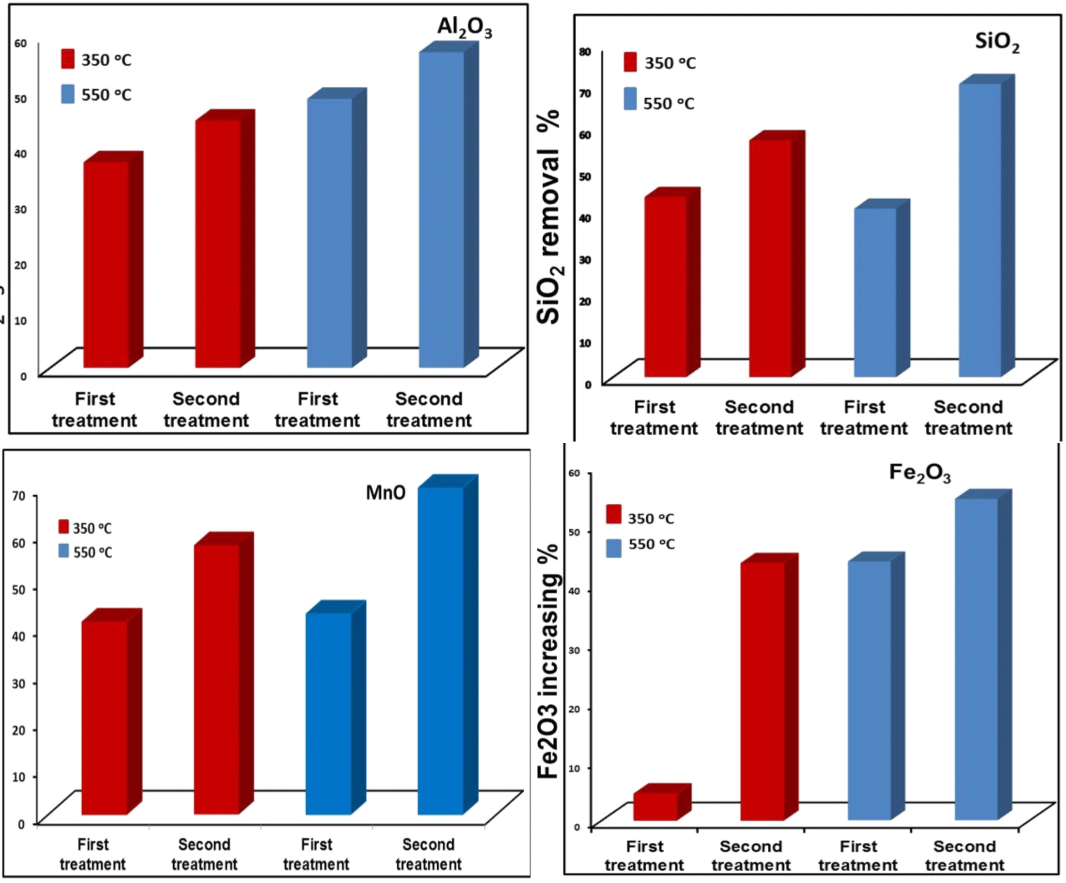


Fig.1 Removal % of SiO_2, $Al_2O_3$and MnO and increasing % of Fe_2O_3 at 350 and 550 °C for the first and second treatment

According to Bayer process (a process used to produce Al_2O_3 from bauxites), Al_2O_3 and SiO_2 can be leached by NaOH solutions as following [16]:

$Al_2O_3 . 2SiO_2 . 2H_2O + 2NaOH . 5H_2O$

$2Al(OH)_4^-(aq) + 2Si(OH)_4^0(aq) + 2Na^+(aq)$

$Al_2O_3(s) + 2NaOH(aq) + 3H_2O$

$2Al(OH)_4^-(aq) + 2Na^+(aq)$

$SiO_2(s) + NaOH(aq) + H_2O$

$Si(OH)_3^-(aq) + 2Na^+(aq)$

Mazzocchitti et al. [16] showed that F_2O_3 has low solubility in NaOH solutions which causes the F_2O_3 to be non-leachable in highly alkaline NaOH solutions. These investigations are in agreement with our XRF and SEM results, as iron is concentrated in the final residual solids.

Table 3 the elemental composition of the ore before and after treatment with different NaOH concentration at different temperatures.

Main Constituents	Before treatment (A)	Lower conc NaOH $350^{o}C$ (B)	Higher conc NaOH $350^{o}C$ (F)	Lower conc NaOH $550^{o}C$ (D)	Higher conc NaOH $550^{o}C$ (H)
SiO_2	18.18	10.33	6.7	10.83	4.07
Al_2O_3	10.05	6.33	4.09	5.19	2.48
Fe_2O_3	39.20	40.98	56.55	56.37	64. 84
MnO	8.11	4.78	4.65	4.65	2.78
MgO	0.13	0.09	0.08	0.09	0.00
CaO	1.50	1.21	1.24	1.33	0.98
K_2O	0.09	0.07	0.08	0.08	0.05
Na_2O	0.67	0.61	0.75	0.74	0.62
P_2O_5	0.14	0.05	0.05	0.06	0.76
BaO	3.65	3.30	3.54	3.66	2.85
LOI	18.23	22.10	22.43	20.45	20.09

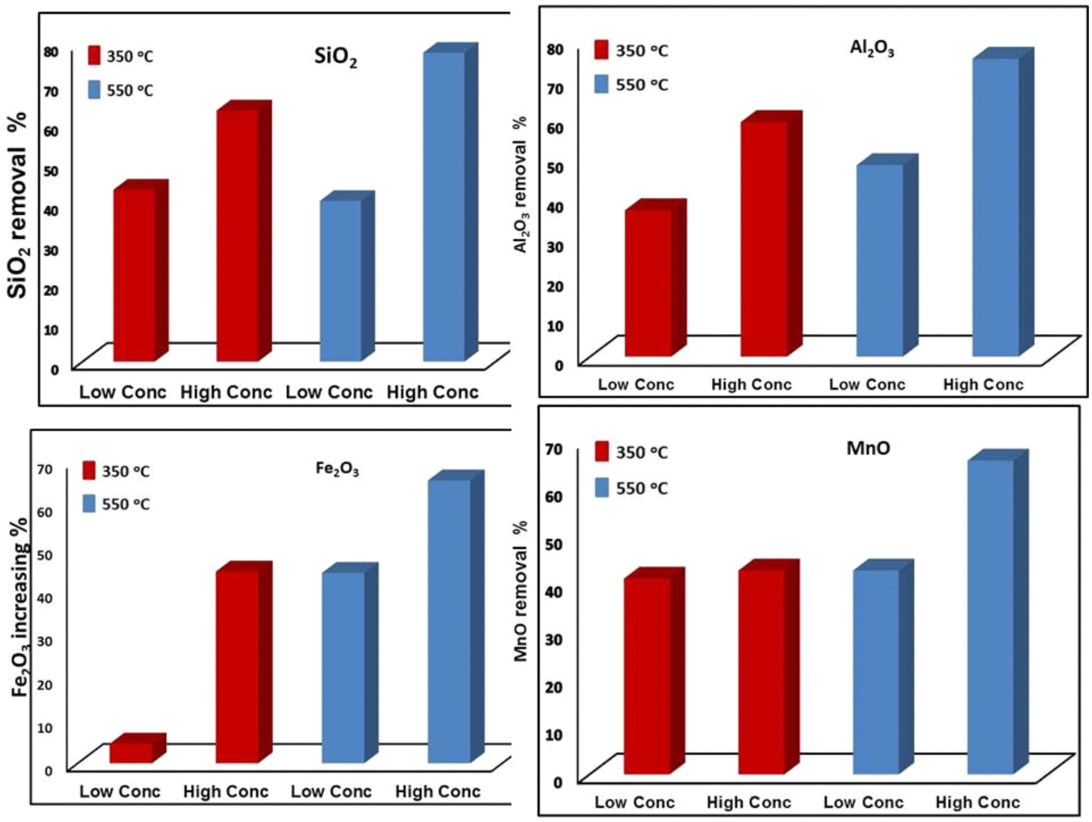


Fig.2 Removal % of SiO2, Al2O3 and MnO and increasing % of Fe2O3at 350 and 550 oC with different concentrations of NaOH

3.3. Optimization process

The samples treated with the higher concentration of NaOH at different temperatures were undergone another treatment with the lower concentration of NaOH at different calcination temperatures. Table 4 shows XRF analysis of the mentioned samples before and after the treatments.

Figure 3 shows that the best results obtained were for the sample of the second treatment at calcination temperatures 500 ^{o}C with the lower concentration of sodium hydroxide (sample I). We get rid of 86.4% silica, 87.4% Al_2O_3, and 74.6 MnO. The concentration of iron oxide after treatment is 68.9 % instead of 39.2 % in the original ore (table 4) which means an increasing in concentration by 75.8 % over the original concentration, as shown in fig 3. However, Lanmu and Zhibao, 2014, demonstrated that silica solubility is favored at low temperatures [17]. Mazzocchitti et al. [16] used pressure caustic for dissolution of Si and Al. They informed that the dissolution of Si and Al at 200 °C has a faster rate than that at 120 °C, as well as avoiding formation of a secondary phase. The optimized time was chosen to be one hour at 200 °C. These results are largely consistent with the results of this study.

Table 4 the elemental composition of the ore before and after Optimization process

Main Constituents	Before treatment (A)	Higher Conc NaOH 350 ^{o}C (First treatment (F))	Lower conc NaOH 350 ^{o}C (Second treatment (G))	Higher conc NaOH 550 ^{o}C (First treatment (H))	Lower Conc NaOH 550 ^{o}C (Second treatment (I))
SiO2	18.18	6.7	4.28	4.07	2.47
Al2O3	10.05	4.09	3.20	2.48	1.27
Fe2O3	39.20	56.55	61.55	64. 84	68.90
MnO	8.11	4.65	2. 58	2.78	2.06
MgO	0.13	0.08	0.08	0.00	0.00
CaO	1.50	1.24	0.97	0.98	0.86
K2O	0.09	0.08	0.07	0.05	0.05
Na2O	0.67	0.75	0.76	0.62	0.77
P2O5	0.14	0.05	0.05	0.76	0.02
BaO	3.65	3.54	3.31	2.85	2.70
LOI	18.23	22.43	23.04	20.09	20.88

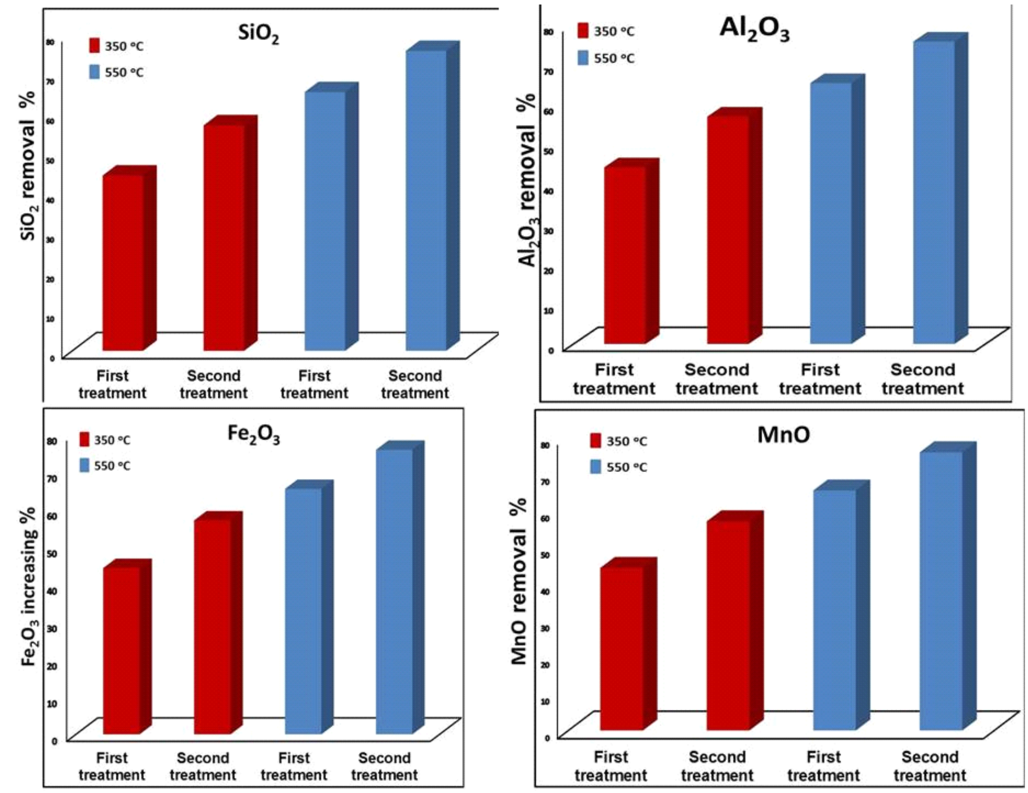


Fig.3 First and second treatments for removal % of SiO_2, $Al_2O_3$and MnO and increasing % of Fe_2O_3 at different temperatures with different concentrations of NaOH

3.4 SEM-EDX measurements

The raw ore was scanned by SEM-EDX to investigate morphological features and micro-chemical components before (Fig. 4 a and b) and after treatment (Fig. 4 c and d). The SEM images of untreated ore, at low and high magnifications, reveal polymorph structures include tubular, granular, irregular, and random agglomerated particles. After treatment, SEM images show a significant difference from that before treatment. We find an increase in the concentration of spherical particulate aggregates of Fe2O3, which represent the majority of the sample. The particles are in the nano scale which represent narrow size particles distribution. Bhosale et al. [18] prepared γ -Fe_2O_3 nanoparticles of nearly the same morphology. These results are confirmed by EDS analysis, Fig.5. The results shown in Fig.5a Shows that iron is the main component of the ore which accounted by about 29% from the elemental surface existence. Fig.5b shows an observable increase in the iron percentage to reach about 36% from the elemental surface existence. These results are in a good agreement with results obtained by XRF and SEM.

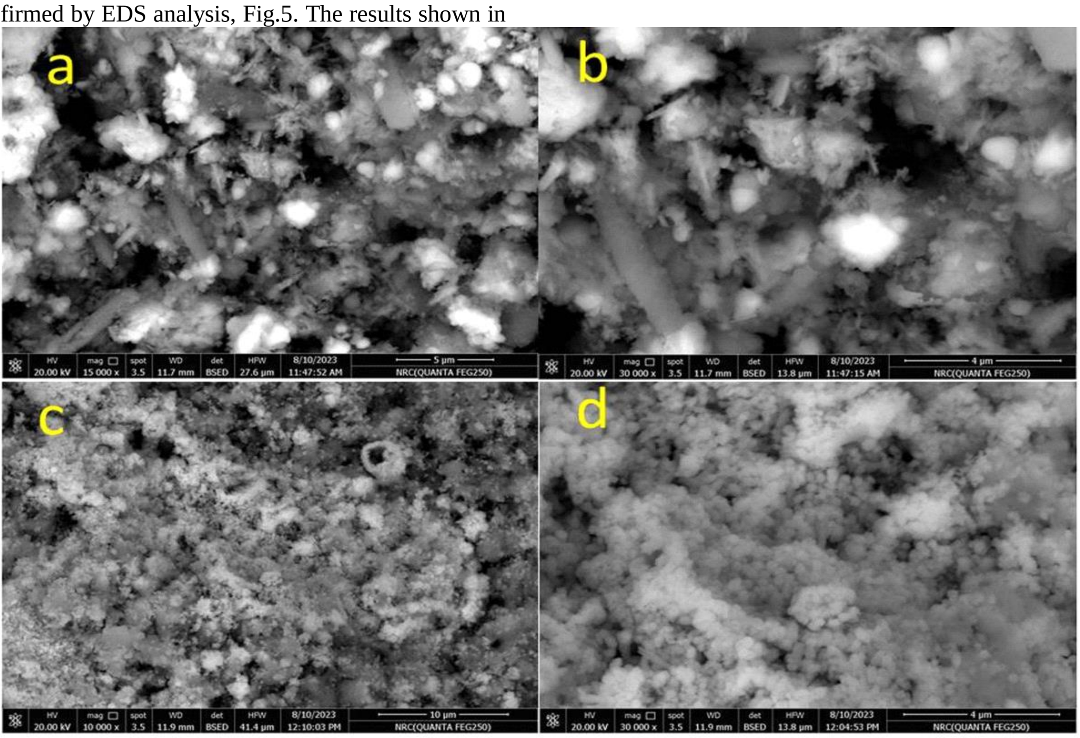
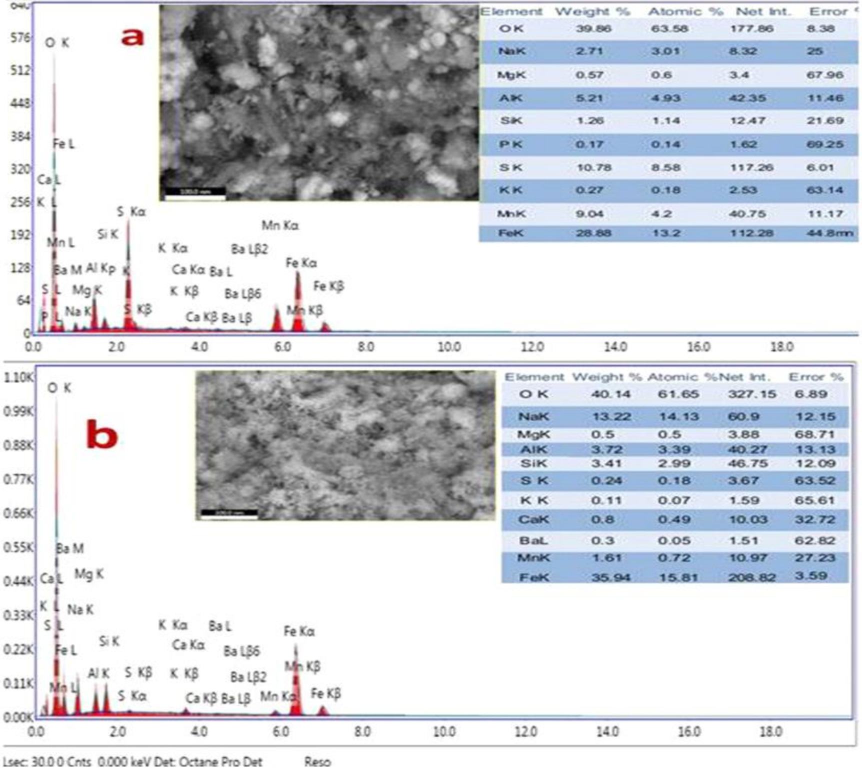


Fig. 4 SEM of the ore sample before (a and b) and after treatment (c and d) with different magnifications.



Element	Weight %	Atomic %	Net Int.	Error %
OK	39.86	63.58	177.86	8.38
NaK	2.71	3.01	8.32	25
MgK	0.57	0.6	3.4	67.96
AlK	5.21	4.93	42.35	11.46
SiK	1.26	1.14	12.47	21.69
PK	0.17	0.14	1.62	69.25
SK	10.78	8.58	117.26	6.01
KK	0.27	0.18	2.53	63.14
MnK	9.04	4.2	40.75	11.17
FeK	28.88	13.2	112.28	44.8mn

Element	Weight %	Atomic %	Net Int.	Error %
O K	40.14	61.65	327.15	6.89
NaK	13.22	14.13	60.9	12.15
MgK	0.5	0.5	3.88	68.71
AlK	3.72	3.39	40.27	13.13
SiK	3.41	2.99	46.75	12.09
S K	0.24	0.18	3.67	63.52
K K	0.11	0.07	1.59	65.61
CaK	0.8	0.49	10.03	32.72
BaL	0.3	0.05	1.51	62.82
MnK	1.61	0.72	10.97	27.23
FeK	35.94	15.81	208.82	3.59

Fig. 5 EDX of the ore sample before (a) and after optmization process (b).

4. Conclusion

Iron oxide has been successfully separated from its ores. By applying the current simple method, Fe_2O_3 concentration increases by 75.8 % and get rid of 86.4% of silica, 87.4% of Al_2O_3, and 74.6 % of MnO. The second treatment for approximately one hour with the highest concentration of sodium hydroxide at calcination temperatures 550 ºC was considered optimal for this study. According to the industrial level, this method is considered an effective, as a simple and quick process. There are no complicated steps, devices or chemicals, but continuing improvement is recommended to achieve better results.

Conflicts of Interest

There are no conflicts to declare.

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