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Separation of iron and vanadium from titanomagnetite by thermal treatment and magnetic separation

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ABSTRACT

In this paper, the extraction of iron and vanadium from titanomagnetite sources was studied by a reduction roasting process. Titanomagnetite has a spinel structure, and roasting facilitates its decomposition, making it easier to separate iron and titanium. In the roasting stage, several key parameters including temperature, particle size, and oxidation roasting were investigated for the iron separation, and a magnetic separator was used at a field intensity of 800 G to enrich the roasted product. For iron extraction, decreasing the particle size resulted in an increase in grade, and increasing temperature and oxidation did not have a positive effect on increase in grade. The highest iron grade and recovery were 65.32% and 65.77%, respectively, and were obtained with a particle size less than 38µm and a temperature of 550°C. The tailings from the magnetic separation stage contain titanium and vanadium, so the separation of vanadium was investigated using salt roasting at a temperature of 900°C and a roasting time of 2 h followed by leaching with water. The highest grade and recovery of vanadium oxide were obtained at 67.51% and 69.4%, respectively.

Keywords: *Roasting, Titanomagnetite, Iron, Vanadium.*

1. Introduction

Today, with the increasing use of metals and their oxides in domestic industries, as well as the availability of rich metal resources in Iran, the need to achieve extraction technology and self-sufficiency in production is felt more than ever. Titanomagnetites are widespread resources that are usually a source of iron, titanium, and vanadium. The structure and composition of titanomagnetites vary, and this requires diverse methods for extracting metals from these sources Zhang et al., 2019; Chen et al., 2020).

In addition to titaniferous deposits, titanomagnetite is found in coastal sands (placer) that are of volcanic origin and are economical to use due to their low cost of exploitation (Chen et al., 2011). The structure of titanomagnetite contains complex vanadium and has the chemical formula [$Fe_{0.23}$ $(Fe_{1.95}Ti_{0.42})O_4$, $Fe_2O_3.FeTiO_3$] (Yang et al., 2020). Vanadium in titanomagnetite is isomorphous with iron minerals, with most of the vanadium present as spinel chalconite (FeV_2O_4) associated with magnetite (Han et al., 2021). Regardless of the type of feed, vanadium processing uses processes including physical concentration, salt roasting, leaching, solution purification, and precipitation (Li et al., 2019; Zhang et al., 2018). Salt roasting is carried out using sodium salts such as sodium carbonate, sodium sulfate, or sodium chloride, usually at temperatures of 800 to 1000°C. Based on reactions 1 to 5, the vanadium produced in salt roasting reacts with added sodium salts such as sodium carbonate to form water-soluble sodium vanadate compounds such as sodium metavanadate ($NaVO_3$). Vanadium solubility and recovery are increased (Gilligan & Nikoloski, 2020; Yang et al., 2020).

$$FeO.V_2O_3 + 0.5O_2 \rightarrow Fe_2O_3.V_2O_3 \quad (1)$$

$$Fe_2O_3.V_2O_3 + 0.5O_2 \rightarrow Fe_2O_3.V_2O_4 \quad (2)$$

$$Fe_2O_4.V_2O_3 + 0.5O_2 \rightarrow Fe_2O_3.V_2O_5 \quad (3)$$

$$Fe_2O_3.V_2O_5 \rightarrow Fe_2O_3 + V_2O_5 \quad (4)$$

$$V_2O_5 + Na_2CO_3 \rightarrow 2NaVO_3 + CO_2 \quad (5)$$

Since titanomagnetites contain elements such as titanium, vanadium, and iron, studies have been conducted to separate these elements (Chen et al., 2011; Yang et al., 2016; Han et al., 2021;). Safdar et al. (Safdar et al., 2020) developed a new method for extracting TiO_2-enriched materials from vanadium-titanomagnetite (VTM) concentrates, which combines partial carbon reduction and mild acid leaching. By optimizing the reduction at 1000°C for 3 h with 6% carbon and leaching with 0.2 mol/L H_2SO_4 at 80°C for 3–4 h, a rutile residue containing 72.2 wt% TiO_2 was obtained, which provides a valuable material for titanium extraction, while iron and vanadium were recovered from the leaching solution. Maldybayev et al. (2024) examined the processing of titanomagnetites using low-temperature treatment and magnetic separation, showing that with the use of a reducing agent, chloride additives, and calcium fluoride at 1200°C for 60 minutes, iron is effectively extracted, with the magnetic fraction yielding 75.5% and iron extraction reaching 97.8%, while the titanium dioxide content in the non-magnetic fraction increased to 65%.

Processing, concentrating, and purifying placer titanomagnetite mines ores has both technical and economic justification, enabling the simultaneous extraction of strategic and high-value-added elements. The purpose of this project was to assess the feasibility of increasing the grade of placer minerals with the approach of simultaneous extraction of iron and vanadium. The use of the roasting method, especially at lower temperatures or under reducing conditions using reducing gases (such as CO or H_2), or under oxidizing conditions, is a less expensive

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and simpler process that can optimize the extraction efficiency and achieve more desirable results.

2. Material and methods

2.1. Material

The titanomagnetite used in this research was obtained from the placer deposit mine of Bazman (Sistan and Baluchestan, Iran). The chemical composition of the representative ore was characterized by X-ray diffraction (XRD) (Asenware, AW/XDM 300, China), X-ray fluorescence (XRF) (pw1410, Netherlands), thin section and polished section (Kaywa polarizing microscope) analyses. Also, the solutions taken from the leaching experiments were analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES, PerkinElmer, Optima 7300DV, USA).
circuit.

2.2. Method

In this process, the samples were first crushed to particles smaller than 500 μm. In the reductive roasting process, the parameters of temperature, particle size, and oxidation were investigated. Temperatures of 550 to 750 °C, oxidation temperatures of 700 to 1100 °C, and particle sizes of 38, 75, and 200 μm were examined. It should be noted that the particle size entering the furnace was constant and the effect of particle size was investigated after reduction. Methane gas (0.5 L/min) was used along with a certain percentage of air (1-3%) and 2% nitrogen. After the reduction stage, re-crushing was performed and magnetic separation was performed using a Davis tube with an intensity of 800 G.

On the tailings of the magnetic separation step, a salt roasting step was performed using sodium chloride at a temperature of 900 °C for 2 hours. In the salt roasting process, a 5 M sodium chloride solution was mixed with a pulp density of 50% (W/W) and the resulted mixture kept at 80 °C for 2 hours until the water was completely evaporated, then it was placed in the oven and heated. After the roasting process was leached in water at 50 °C until the vanadium dissolved.

3. Results and discussion

3.1. Sample characterization

In order to characterize various properties of the titanomagnetite ore, several chemical and physical analyses were conducted. XRF analysis showed that the amount of Fe_2O_3 was 45.32% and V_2O_5 was 0.045% (Table 1).

Microscopic images of thin sections were prepared using transmitted light with plane-parallel light (PPL). As can be seen in Fig. 1, they show that the sample contained magnetite, titanomagnetite, and tremolite - actinolite. Degree of liberation studies showed that magnetite and titanomagnetite were seen in the higher fractions as inclusions. As the sample particle size decreased, the degree of freedom increased, so that in the fraction of -53 +38 mμ, the degree of freedom reached 80%.

3.2. Effect of oxidation

One possible method to break the iron-titanium bond in titanomagnetite and ilmenite samples is oxidation at particles smaller than 500 mμ. Therefore, all samples were oxidized by the fixed bed furnace at temperatures of 700, 800, 900, 1000, and 1100 °C with compressed air at a flow rate of 2 L/min. Subsequently, XRD analysis was performed on the obtained samples.

The results of XRD analysis of oxidation at different temperatures are shown in Fig. 2. The results indicated that at temperatures below 1000 °C, practically no significant change occurred and the phase was still hematite and titanomagnetite. The reason for the greater presence of hematite in these samples was due to the oxidation of magnetite to hematite at this temperature, although titanomagnetite remained unchanged at lower temperatures. As the temperature increaseed to 1000°C, almost no trace of titanomagnetite was seen in the XRD analysis, and iron was almost entirely in the hematite phase.

Studies have shown that magnetite is converted to weakly magnetic or non-magnetic phases, such as hematite and maghemite, which reduces iron recovery during magnetic separation. Furthermore, titanium is not effectively released from the iron phase, leading to a decrease in iron grade in the concentrate and overall process efficiency (Jena et al., 2015). Therefore, reduction roasting should be performed after this step. Oxidative roasting, by converting iron phases, such as Fe_3O_4 to Fe_2O_3 and creating a uniform structure, allows the subsequent reduction stage with H_2 and CO gases to proceed with higher efficiency and faster kinetics (Li et al., 2021).

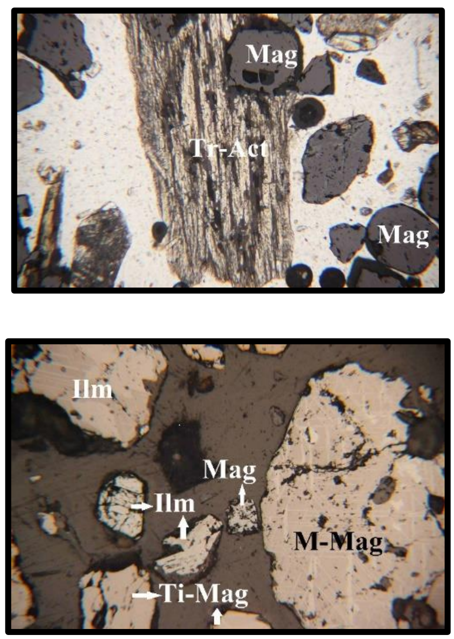

Fig. 1. Microscopic images of the titanomagnetite sample with polarizing microscope (Mag: magnetite, Ti-Mag: titanomagnetite, I: ilmenite, and Tr-Act: tremolite – actinolite).

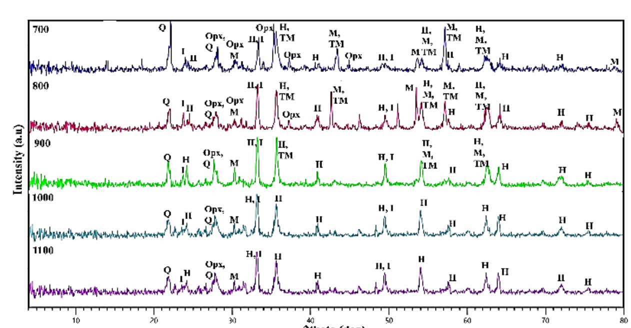

Fig. 2. XRD results of the sample after oxidation at different temperatures (M: magnetite, H: hematite, Q: quartz, TM: titanomagnetite, Opx: orthopyroxene, and I: ilmenite)

Table 1. The chemical composition of the representative titanomagnetite ore sample.

Element	Na_2O	K_2O	CaO	MgO	Al_2O_3	Fe_2O_3	MnO	SiO_2	SO_3	P_2O_5	TiO_2	V_2O_5	LOI
Content (% Wt)	2.34	0.98	3.36	3.98	6.89	45.32	0.21	25.62	0.56	0.23	6.95	0.045	2.99

After performing oxidation tests, samples were reduced by methane gas at five temperatures of 550, 600, 650, 700, and 750 °C. The results of sample reduction by methane showed that the hematite obtained at 600 °C was completely converted to magnetite (Fig. 3).

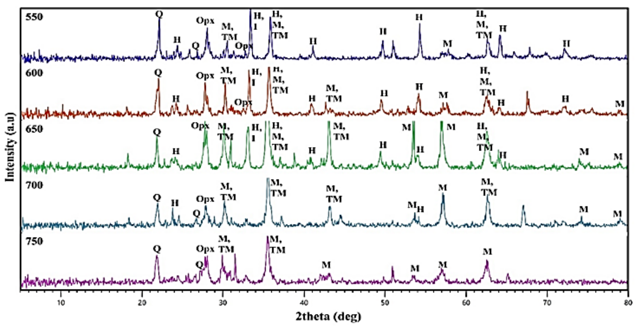

Fig. 3. XRD results of the sample after the reduction process at different temperatures with the oxidation process at 1000 °C (M: magnetite, H: hematite, Q: quartz, TM: titanomagnetite, Opx: orthopyroxene, and I: ilmenite)

In Table 4, the results indicated an increase in iron content in the magnetic separation concentrate sample by more than 59%. The iron content in the concentrate did not change significantly with the increase in temperature reduction and remained approximately constant at 58-59%. The titanium content in the tailings at reduction temperatures of 550, 600, and 650°C was higher than the titanium content in the concentrate, indicating the relative separation of iron and titanium from each other.

However, with increasing reduction temperatures (700 and 750°C), the titanium content in the concentrate increased (more than 5%), which means that the iron and titanium bond did not separate at high reduction temperatures. At reduction temperatures of 750°C, the vanadium content in the concentrate increased and reached 0.25%. These results indicated the relative separation of iron in the concentrate phase and the accumulation of titanium in the tailings. However, the presence of vanadium in the concentrate indicates a correlation between iron and vanadium, which did not changed with oxidation and reduction. The correlation between iron and vanadium is consistent with previous studies (Yang et al., 2020). Iron recovery at temperatures of 550, 600, 650, 700 and 750°C was 86.92, 85.90, 86.10, 81.40 and 80.32%, respectively.

Pre-oxidation creates cracks and porous structures in the iron ore particles, which improves the permeability of the reducing gas (Zheng et al., 2021).

In previous studies, the effect of roasting temperature during suspension magnetic roasting of ores containing siderite and hematite was investigated. The results showed that at temperatures below 550°C, the phase transformation was incomplete, and the magnetic properties were poor. At around 610°C, the transformation of hematite and siderite to magnetite was almost complete, resulting in the highest iron recovery and grade. A further increase in temperature led to the formation of non-magnetic phases, such as Fe_3O_4, which negatively affected the magnetic separation performance (Chen et al., 2022).

3.3. Effect of reductive temperature

Reduction tests were conducted directly at temperatures of 550, 600, 650, 700, and 750°C with methane gas to investigate the effect of the presence or absence of the oxidation process on factors, such as iron recovery, titanium and vanadium recovery, and titanium separation from iron. The results obtained are shown in Fig. 4.

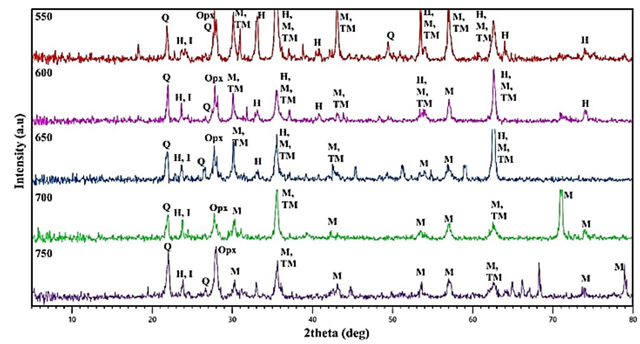

Fig. 1. XRD results of the sample after the reduction process with methane gas at different temperatures (M: magnetite, H: hematite, Q: quartz, TM: titanomagnetite, and I: ilmenite).

In Table 3, an increase in iron grade of up to 59% can be seen. However, by comparing the results of these two tables, it can be seen that the oxidation process did not considerably affect the increase in the iron grade in the magnetic separation concentrate sample. The iron content in the concentrate did not change significantly with the increase in temperature reduction and remained approximately constant at 58-59%. The titanium content in the concentrate was higher than its content in the tailings, indicating the effectiveness of oxidation in breaking the bond between iron and titanium. The titanium grade in the concentrate at all reduction temperatures ultimately reached 5.5%, while in tailings it was below 3%. At reduction temperatures of 700 and 750 °C, the vanadium grade in the concentrate increased to more than 0.2%. These results indicated the relative separation of iron in the concentrate phase and the accumulation of titanium in the tailings. The presence of vanadium in the concentrate indicated a correlation between iron and vanadium, which remained changed with oxidation and reduction. Iron recovery at temperatures of 550, 600, 650, 700 and 750°C was 85.62, 87.47, 80.23, 84.31 and 78.32%, respectively.

The effect of roasting temperature on iron recovery and magnetic separation was discussed. The results showed that at temperatures lower than 540°C, the roasting process was not able to fully convert hematite (Fe_2O_3) to magnetite (Fe_3O_4), leading to lower iron recovery. However, at 540°C, the conversion was complete, increasing the magnetic properties of the ore (Yu et al., 2018).

Table 2. XRF results of oxidized and reduced samples after magnetic separation.

Element	Na_2O	K_2O	CaO	MgO	Al_2O_3	Fe^c	MnO	SiO_2	SO_3	P_2O_5	TiO_2	V^d	LOl
C^a 550	0.08	0.09	0.78	1.84	2.36	58.67	0.44	3.5	ND	0.42	7.1	392	1.13
T^b 550	0.99	0.56	2.75	6.63	5.65	33.41	0.47	25.81	0.11	0.31	10.43	211	1.64
C 600	0.07	0.08	0.71	1.74	2.25	59.24	0.45	3.05	ND	0.31	7.13	504	1.25
T 600	0.99	0.58	2.94	6.85	5.71	32.33	0.47	26.06	ND	0.32	10.56	241	0.96
C 650	0.08	0.07	0.7	1.76	2.25	59.38	0.45	3.14	ND	0.35	7.46	650	1.65
T 650	1.27	0.64	3.58	8.23	6.58	25.54	0.44	32.94	ND	0.39	9.39	261	0.15
C 700	0.09	0.07	0.7	1.77	2.23	58.69	0.47	3.3	0.09	0.3	9.03	1457	2.56
T 700	1.83	0.93	4.95	10.96	8.65	13.01	0.39	48.46	ND	0.36	3.95	204	0.88
C 750	0.08	0.08	0.62	1.87	2.34	59.26	0.47	3.29	ND	0.22	9.06	2431	3.26
T 750	1.95	0.9	5.35	10.75	8.84	12.14	0.42	48.55	ND	0.64	5.67	283	0.46

Table 1. XRF results of the reduced samples after magnetic separation.

Element	Na_2O	K_2O	CaO	MgO	Al_2O_3	Fe^c	MnO	SiO_2	SO_3	P_2O_5	TiO_2	V^d	LOl
C^a 550	0.09	0.08	0.6	1.68	2.13	59.04	0.4	2.66	ND	0.31	9.27	1648	-2.04
T^b 550	1.78	0.81	4.91	10.34	7.38	15.46	0.38	45.37	ND	0.57	5.05	236	0.74
C 600	0.09	0.07	0.56	1.67	2.13	59.03	0.39	2.67	ND	0.27	9.3	1924	-1.98
T 600	1.77	0.81	5.15	10.93	8.1	13.5	0.36	46.78	ND	0.52	4.19	200	1.92
C 650	0.09	0.07	0.55	1.77	2.19	59.18	`0.41	2.78	ND	0.24	9.36	1894	-2.5
T 650	1.67	0.78	4.68	9.76	7.86	18.05	0.35	42.3	ND	0.5	5.01	446	1.11
C 700	0.08	0.08	0.56	1.69	2.19	59.43	0.4	2.62	ND	0.25	9.41	2255	-2.68
T 700	1.93	0.88	5.24	11.4	8.42	12.45	0.36	50.55	ND	0.64	3.57	173	-0.88
C 750	0.09	0.09	0.56	1.77	2.23	59.19	0.4	2.81	ND	0.28	9.48	2674	-2.77
T 750	1.98	0.88	5.44	11.15	8.63	12.43	0.37	49.68	ND	0.56	3.81	212	-0.24

a Concentrate, b Tailing, c Analyzed by Titration, d Analyzed by ICP (ppm)

In the study by Chen et al. (2020), the effect of roasting temperature on iron recovery from titanomagnetite was investigated. In this process, temperature plays a crucial role in the transformation of mineral phases and the improvement of the magnetic properties of iron ore. The results showed that increasing the temperature from 450°C to 550°C facilitated the reduction and transformation of hematite (Fe_2O_3) to magnetite (Fe_3O_4), improved magnetic properties, and accelerated the magnetic separation process.

3.4. Effect of particle size

In order to investigate the effect of particle size on achieving proper separation of iron, titanium, and vanadium and to break the correlation between them, a reduction test was performed on different particle sizes and the effect of grinding the initial sample on the grade of the elements of interest was studied. For this purpose, the unoxidized sample was reduced with methane gas at 550°C and the reduced sample was crushed with a planetary mill for specific times until 100% of the sample reached particle sizes below 200, 75, and 38 µm.

By examining the results obtained from Fig. 5, it can be concluded that particle size has a great impact on the iron and titanium content after the reduction process. When the particle size reduced, the iron content in the tailings also decreased, indicating the separation of the fine iron particles under a magnetic field. Regarding the silica impurity, it can be said that the extent of separation increased in the size fractions of 75, 200, and then 38 mµ, respectively. In general, the main goal, which was to eliminate the correlation between titanium and iron and increase their grade, is possible by changing the particle size. Iron recovery at particle sizes of 200, 75, and 38 µm was 72.25%, 65.27%, and 65.77%, respectively.

It was shown that reducing particle size through grinding enhances the recovery of iron and titanium during magnetic separation, as finer particles lead to better liberation of magnetic phases from non-magnetic ones. However, once an optimal grinding time is reached, further size reduction results in the formation of ultra-fine particles, which decreases separation efficiency because these fine particles are more difficult to recover in magnetic separation circuits (Liu et al., 2022).

Fig. 5d shows the dissolution rate of V_2O_5 from tailings at different particle sizes. A maximum dissolution rate of 67.51% and a vanadium recovery of 69.4% were achieved after salt roasting and leaching at particle sizes less than 38 µm. Reducing particle size increases both grade and recovery, because smaller particles undergo more effective roasting and dissolve more easily (Han et al., 2021).

Previous studies have shown that reducing particle size to below 74 µm helps to more effectively release vanadium-containing minerals. This leads to improved magnetic separation efficiency and increased iron and vanadium recovery and grade. Furthermore, the results showed that the magnetic separation process reached its maximum efficiency when more than 85.2% of the material was ground to below 74 µm. However, the authors caution that over-grinding may result in an increase in fines, which can reduce recovery in subsequent processing steps (Xu et al., 2017).

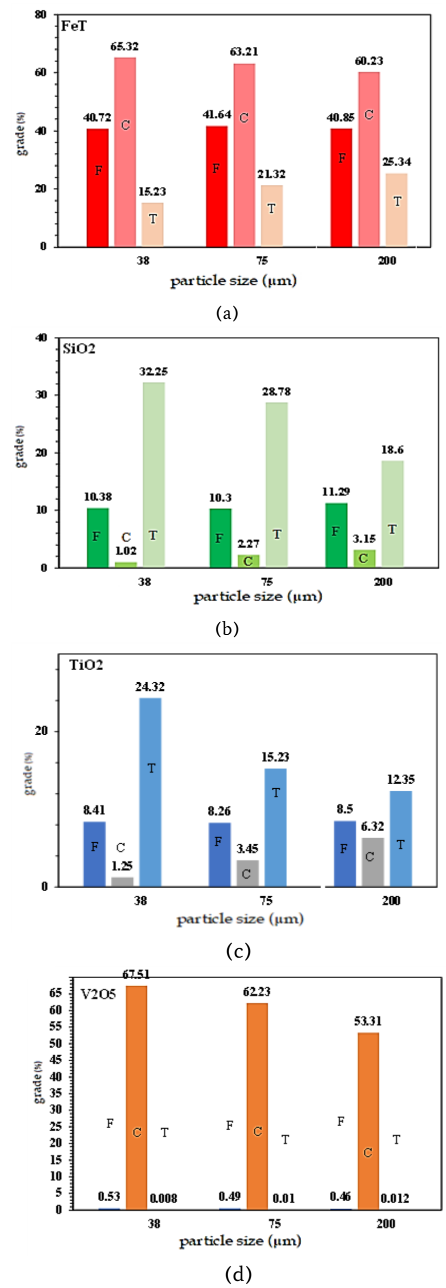

Fig. 5. The effect of particle size on a magnetic separation. a. Fe total b. SiO2 c. TiO2 d. V2O5 (F: Feed, C: Concentrate, and T: Tailing)

4. Conclusions

The development of innovative and sustainable technologies can contribute to mitigating existing challenges and optimizing the utilization of titanomagnetite resources. So, the extraction of iron and vanadium from titanomagnetite sources was investigated by the reduction roasting process and the following results were obtained:

- Increasing the temperature to 550°C had a positive effect due to the conversion of hematite minerals to magnetite and the breaking of the iron-titanium bond.
- The reduction in particle size due to the low degree of liberation of the sample resulted in an increased grade.
- Oxidation roasting had no effect on iron grade and recovery, while vanadium content decreased due to the high roasting temperature.
- The highest iron grade and recovery were 65.32% and 65.77%, respectively, obtained with a particle size smaller than 38μm and a temperature of 550°C.
- The highest vanadium oxide grade and recovery of, 67.51 and 69.4%, respectively, obtained by salt roasting at 900°C for 2 h followed by water leaching.

Acknowledgments

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