ÇUKUROVA UNIVERSITY
INSTITUTE OF NATURAL AND APPLIED SCIENCES

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UPGRADING OF A SPECULARITE ORE FOR INDUSTRIAL USAGE

DEPARTMENT OF MINING ENGINEERING

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ABSTRACT

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In this study, the upgrading of a specularite ore from the Feke district in Adana, Turkey was investigated applying shaking table, wet magnetic separation, microwave heating followed by wet magnetic separation, roasting followed by wet magnetic separation, falcon concentrator, anionic and reverse cationic flotation methods. As a result of the X-Ray Diffraction (XRD) analyses, it was determined that the ore mainly contains specularite, goethite, hematite, and some other iron oxide minerals along with quartz and mica as gangue minerals. According to XRF analysis, the ore contains 57.90 % Fe. It was possible to produce concentrates (> 62 Fe) suitable for industrial usage by any methods above. Under the optimum conditions, for shaking table, wet magnetic, microwave heating, roasting, falcon concentrator, reverse anionic, and reverse cationic flotation methods, Fe grades (%) were 64.47%, 68.90%, 68.90%, 69.10%, 63.93%, 63.04%, and 68.95% respectively.

Key Words: Specularite, Gravity separation, Magnetic separation, Reverse flotation, Roasting, Upgrading, Experimental design.

ÖZ

YÜKSEK LİSANS TEZİ

ÖRNEK BİR SPEKÜLARİT CEVHERİNİN ENDÜSTRİYEL KULLANIM İÇİN KALİTE ÖZELLİKLERİNİNİ İYİLEŞTİRİLMESİ

Ammar Mahdi Ahmed AL-DHUBAIBI

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Bu çalışmada, Adana bölgesinden alınan sepkülarit cevherine, sallantılı masa, yaş manyetik ayırma, mikrodalga ısıtma sonrası yaş manyetik ayırma ve kavurma sonrası yaş manyetik ayırma, Falcon konsantratörü, ve ters anyonik ve katyonik flotasyon yöntemleri uygulanarak zenginleştirilebilirliği araştırılmıştır. X-Işını Kırınımı (XRD) analizleri sonucunda cevherin esas olarak spekülarit, goetit, hematit ve diğer bazı demir oksit mineralleri ile gang mineralleri olarak kuvars ve mika içerdiği tespit edilmiştir. Ayrıca, XRF analizine göre, cevher %57,90 Fe içerir. Deneysel çalışmalar sonucunda bu cevherden endüstriyel kullanıma uygun konsantre üretmek (> 62 Fe) mümkün olmuştur. Optimum koşullar altında, sallantılı masa, ıslak manyetik ayırma, mikrodalga, kavurma, falcon konsantratör, ters anyonik ve katyonik flotasyon yöntemlerinden Fe tenörü (%) olarak %64,47, %68,90, %68,90, %69,10, %63,93, %63,04 ve %68,95 değerleri sırasıyla elde edilmiştir.

Anahtar Kelimeler: Spekülarit, Gravite ayırma, Manyetik ayırma, Ters flotasyon, Falcon konsantratörü, Kavurma, Deney tasarımı.

EXTENDED ABSTRACT

In this study, the enrichment of specularite ore obtained from Feke district of Adana in Turkey was investigated applying shaking table, wet magnetic separation, microwave heating followed by wet magnetic separation, and roasting followed by wet magnetic separation, falcon concentrator and reverse anionic and reverse cationic flotation methods. Sample characterization by XRD showed that the presence of a variety of minerals mainly; specularite, goethite, hematite, and iron oxide minerals along with quartz and mica as gangue minerals.

This study aimed to investigate the possibility of the enrichment of a specularite ore to produce iron concentrate with greater than 62% Fe grades that can be used by the industry by shaking table, wet magnetic separation, microwave heating followed by wet magnetic separation and roasting followed by wet magnetic separation, falcon concentrator, anionic and reverse cationic flotation as well as to investigate the operating of the parameters on which they depend on the grade and recovery of iron concentrate under the others fixed conditions.

In shaking table method upgrading the specularite sample was carried out using two-particle size fractions (-150+74 μm and -74+0 μm) and table tilt angle (between3°-7°). The wet magnetic separation method was performed at a constant magnetic field intensity of 0.75 T in two stages. In the first stage, samples were classified to five different particle size fractions (-2000+1000 μm, -1000+500 μm, -500+150 μm, -150+74 μm and -74 μm) and the optimum particle size fraction was determined. The second stage was performed at 7 different magnetic field intensity between 0.25 T and 0.85 T using the optimum particle-size fraction sample (-150 μm). In microwave heating followed by wet magnetic separation, the main parameters included; microwave power (0.52 - 0.90 KW), exposure time (1-3 min) and magnetic field intensity (0.25-0.85 T) whereas in roasting followed by wet magnetic separation, the main parameters included; temperature (450-650°C), exposure time (5-10 min) and magnetic field intensity (0.25-0.85 T). Heating and

roasting treatment methods were aimed to increase the magnetic susceptibility of the iron ore. The parameters range that in falcon concentrator tests were between 20 to 30 psi pressure, 35 to 55.33 frequency, and 75 to 425 μm particle size. In anionic flotation, the main parameters included; corn starch depressant dosage (500 – 10000 g/ton), Aero 343 collector dosage (500-1500 g/ton), and a froth collection time (1-3 min). Reverse cationic flotation performed according to four main parameters included; corn starch depressant dosage (400 –600 g/ton), Aero 3030C collector dosage (200-400 g/ton), X-133 frother dosage (50 –100 g/ton) and a froth collection time (2-4 min).

According to experimental results, a saleable iron concentrate (> 62 Fe) was obtained applying any above mentioned, besides, it is suitable for industrial use. However, at the optimum conditions (-150+74 μm particle size, 3° slope angle, 12 L/min water flow rate and 175 strokes/min shaking speed) of shaking table separation method, iron concentrate grade was obtained with 62.17% Fe (with 90.17% recovery). In wet magnetic separation, the maximum grade of iron concentrate grade was 68.90% Fe (with 22.23% recovery) at 0.25 T magnetic field intensity, -150 μm particle size and 20% solid/liquid ratio while the highest value of the iron concentrate recovery was 67% Fe (with 95.49% recovery) at 0.75 T magnetic field intensity and particle size of -74 μm and 20% solid/liquid ratio. In microwave heating followed by wet magnetic separation, maximum iron concentrate grade was 68.90% Fe (with 27.28 % recovery) at conditions of 0.9 KW microwave power, 2 min exposure time, 0.25 T magnetic field intensity, -150+38 μm particle size and 20 % solid/liquid ratio while the maximum iron concentrate recovery was 82.23% (with 66.06% Fe grade) at conditions of 0.72 KW microwave power, 3 min exposure time, 0.85 T magnetic field intensity, -150+38 μm particle size and 20 % solid/liquid ratio. In roasting followed by wet magnetic separation method, the highest value of iron concentrate grade was 69.10 % Fe (with 41.16 % recovery) at conditions of 450 °C temperature, 10 min exposure time, 0.25 T magnetic field intensity, -150+38μm particle size and 20 %

solid/liquid ratio while the maximum iron concentrate recovery was 85.95% (with 62.26% grade) at conditions of 650 °C temperature, 10 min exposure time, 0.85 T magnetic field intensity, -150+38 μm particle size and 20 % solid/liquid ratio. In Falcon concentrator test, 63.93 % Fe (with 68.06% recovery) iron concentrate grade was obtained from conditions of 30 psi pressure, 35 Hz frequency, - 75 μm particle size, and 20 % solid/liquid ratio. In reverse anionic flotation method, the optimum conditions for the best iron concentrate grade were 63.04 % Fe (with 96.85% recovery) and that was at conditions of 5250 g/ton depressant dosage, 1000 g/ton collector dosage, 600 g/ton frother dosage, 2 min froth collection time at pH 12.5 and -106+38 μm particle size while the highest iron concentrate recovery (98.96% with 62.26% Fe grade) was obtained at optimum conditions: 5250 g/ton depressant dosage, 1500 g/ton collector dosage, 600 g/ton frother dosage, 1 min froth collection time and -106+38 μm particle size. In reverse cationic flotation method, the maximum iron concentrate grade was 68.95 % Fe (with 81.9% recovery) at conditions of 400 g/ton depressant dosage, 300 g/ton collector dosage, 100 g/ton frother dosage, 2 min froth collection time, 3 pH and -106+38 particle size while the maximum iron concentrate recovery was 90.57 % (with 64.69 % Fe grade) and that was obtained at optimum conditions: 500 g/ton depressant dosage, 400 g/ton collector dosage, 150 g/ton frother dosage, 3 min froth collection time, 3 pH and -150+38 μm particle size.

GENİŞLETİLMİŞ ÖZET

Bu çalışmada, Adana bölgesinden alınan sepkülarit cevherine, sallantılı masa, ıslak manyetik ayırma, mikrodalga ısıtma sonrası ıslak manyetik ayırma ve kavurma sonrası ıslak manyetik ayırma, Falcon konsantratörü, ve anyonik ve ters katyonik flotasyon yöntemleri uygulanarak zenginleştirilebilirliği araştırılmıştır. X-Işını Kırınımı (XRD) analizleri sonucunda cevherin esas olarak spekülan, goetit, hematit ve diğer bazı demir oksit mineralleri ile gang mineralleri olarak kuvars ve mika içerdiği tespit edilmiştir.

Bu çalışmanın temel amacı, sanayi tarafından kullanılabilecek spekülarit cevherinden demir konsantresi tenörüne (>%62 Fe) yararlanma yeteneğini sallantılı masa, ıslak manyetik ayırma, mikrodalga ısıtma ardından ıslak manyetik ayırma, kavurma ardından ıslak manyetik ayırma, falcon konsantratör, anyonik ve ters katyonik flotasyonu ve ayrıca diğer sabit koşullar altında demir konsantresinin tenörüsine ve geri kazanım verimine bağlı oldukları parametrelerin etkisini araştırmak.

Sallantılı masa ayırma yönteminde spekülarit cevherlerinin zenginleştirmesi iki tane boyutuna (-150 + 74 μm ve -74 μm) ve masa eğim açısına (3-7 arasında) göre yapıldı. Tüm testler 12 L / dk su akış debisinde ve 175 dev/dk tambur dönme hızında gerçekleştirilmiştir. Yaş manyetik ayırma yöntemi iki aşamaya göre yapılmıştır. İlk aşamada numuneler, 0.75 T sabit manyetik şiddetinde ve %10 katı oranında beş farklı boyutta (-2000+1000 μm, -1000+500 μm, -500+150 μm, -150+74 μm and -74 μm) tane boyutuna göre ve 0,75 T sabit manyetik alan şiddetinda gerçekleştirilmiştir. İkinci aşama, optimum tane boyutlu numune (-150 μm) ve ağırlıkça%20 katı oranı kullanılarak 0.25 T ile 0.85 T arasında 7 farklı manyetik alan şiddetinde gerçekleştirildi. Mikrodalga ısıtma ardından ıslak manyetik ayırmada ana parametreler; mikrodalga gücü (0.52- 0.90 KW), maruz kalma süresi (1-3 dakika) ve manyetik alan şiddeti (0.25-0.85 T), oysa

kavurma ardından ıslak manyetik ayırmada ana parametreler; sıcaklık (450-650 ° C), maruz kalma süresi (5-10 dk) ve manyetik alan yoğunluğu (0.25-0.85 T). Mikrodalga ısıtma ve kavurma yöntemlerinin amacı, manyetik olmayan üründeki demir konsantrelerinin manyetik duyarlılığını artırmaktır. Falcon konsantratör testlerinde parametre aralığı 20 ila 30 psi basınç, 35 ila 55.33 frekansı ve 75 ila 425 um tane boyutudur. Anyonik flotasyonda ana parametreler; mısır nişastası depresan dozajı (500- 10000 g / ton), Aero 343 toplayıcı dozu (500-1500 g / ton) ve köpük toplama süresi (1-3 dakika). Dört ana parametreye göre yapılan ters katyonik yüzdürme; mısır nişastası depresan dozu (400-600 g / ton), Aero 3030C toplayıcı dozu (200-400 g / ton), köpürtücü dozajı (50-100 g / ton) ve köpük toplama süresi (2-4 dk).

Deneysel sonuçlara göre, ayrıca tüm yöntemleri uygulayarak satılabilir (>%62 Fe) bir demir konsantresi elde edilir, endüstriyel kullanıma hazırdır. Ancak, sallantılı masa yönteminin optimum koşullarında (-150 + 74µm tane boyutu, 3 eğim, 12 L/dk su akış debisi ve 175 dev/dk tambur dönme hızı) %64,47 Fe ile demir konsantresi tenörü (%90,17 verimile) elde edildi. Yaş manyetik ayırmada, demir konsantresi tenörü maksimum değeri 0.25 T manyetik alan şiddeti, -150 µm tane boyutu ve%20 katı oranında %68,90 Fe (%22,23 verim ile) iken, demir konsantresi veriminin en yüksek değeri%67 (%66,79 Fe tenörü ile) 0.75 T manyetik alan şiddeti ve -74 µm tane boyutu ve%20 katı oranında. Mikrodalgada ısıtma ardından ıslak manyetik ayırmadam, maksimum demir konsantresi tenörü olarak %68,90 Fe (%27,28 verim ile) idi 0.9 KW mikrodalga gücü, 2 dakika maruz kalma süresi, 0.25 T manyetik alan şiddeti, -150 + 38 µm tane boyutu ve%20 katı / sıvı oranı koşullarındadır ayrıca konsantre verimi 0.72 KW mikrodalga gücü, 3 dakika maruz kalma süresi, 0.85 T manyetik alan şiddeti, -150 + 38 µm tane boyutu ve% 20 katı oranı koşullarında% 82.23 (% 66,06 Fe tenör ile) idi. Kavurma ardından ıslak manyetik ayırma yönteminde, en yüksek demir konsantresi tenörü %69,10 Fe (%41,16 verim ile) 450 ° C sıcaklık, 10 dakika maruz kalma süresi, 0.25 T manyetik alan şiddeti, -150 + 38 µm tane boyutu ve %20 katı / sıvı oranı

koşullarında iken maksimum demir konsantresi verimi, 650 ° C sıcaklık, 10 dakika maruz kalma süresi, 0.85 T manyetik alan şiddeti, -150 + 38 µm parçacık boyutu ve%20 katı / sıvı oranı koşullarında%85,95 (%62,255 Fe tenörü ile) idi. Falcon konsantratörü ayırma yönteminde % 63,93 Fe (% 68.06 verim ile) demir konsantresi tenörü, 30 psi basınç, 35 Hz frekansı, - 75 µm tane boyutu ve% 20 katı / sıvı oranı koşullarından elde edilmiştir. Anyonik flotasyon ayırma yönteminde, en iyi demir konsantresi tenörü için optimum koşullar% 63.04Fe (% 96.85 verimle) ve 5250 g / ton mısır nişastası, 1000 g / ton Aero 343 toplayıcı, 600 g / ton MIBC, pH 12.5 ve -106 + 38 µm tane boyutu ve 2 dakika köpük toplama süresi iken en yüksek demir konsantresi verimi (% 89.02 demir konsantresi tenörü ile% 69,21 Fe) optimum koşullarda elde edildi: 5250 g / ton mısır nişastası, 1500 g / ton Aero 343, 600 g / ton MIBC, 1 dakika köpük toplama süresi ve -106 + 38 µm tane boyutu. Ters katyonik flotasyon ayırma yönteminde, maksimum demir konsantresi tenörü % 68,95 Fe (% 81.9 verimle) , 400 g / ton depresan dozajı, 300 g / ton toplayıcı dozajı, 100 g / ton köpük dozajı, 2 dakika köpük toplama süresi koşullarında idi iken maksimum demir konsantresi verimi% 90.57 (% 64,689 Fe tenörü ile) iken 3 pH ve -106 + 38 tane boyutu ve optimum koşullarda elde edildi: 500 g / ton depresan dozajı, 400 g / ton toplayıcı dozajı, 150 g / ton köpürtücü dozajı, 3 dakika köpük toplama süresi, 3 pH ve -150 + 38 µm tane boyutu.

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LIST OF ABBREVIATIONS

%	: Percentage
°	: Degree
°C	: Centigrade
µm	: Micrometera
BF	: Blast Furnace
Cl	: Chlorine
Cm	: Centimetere
CMC	: Carboxymethyl celluloses
Cu	: Copper
D	: Iron Oxide
Fe	: Iron
G	: Gauss
G	: Gram
G	: Gothite
g/ton	: Gram/ton
GS	: Gravity Separation
H	: Hematite
Hz	: Hertz
KW	: Kilowatt
L	: Litre
L/min	: Litter/minute
M	: Mica
mg	: Milligram
MIBC	: Methyl isobutyl carbinol
ml	: Millilitre
Mm	: Millimetre

Mn	: Manganese
Mt	: Million tons
Na	: Sodium
P	: Phosphor
Pb	: Lead
pH	: Power of Hydrogen
Q	: Quartz
Rpm	: Revolutions per minute
S	: Sulphur
s/min	: Strokes/minute
SEM	: Scanning Elecron Microscopy
T	: Tesla
Ti	: Tianium
Wt	: Mass fraction
XRD	: X-Ray Diffraction
XRF	: X-Ray Fluorescence
Zn	: Zinc

1. INTRODUCTION

The mining industry is a major force in the world economy, occupying a primary position at the start of the resource supply chain. Nonetheless, its role in contributing to different countries ' national economies varies widely, and is neither well known nor well understood (Dorin et al., 2014).

Nowadays, the mining sector has played the biggest role in the industrialization. The situation of a country in terms of mining assets; is important for both long-term development plans and mining policies. The mining sector has twofold importance due to its direct contribution to the economy and especially the inputs it provides to the manufacturing sector (ICMM, 2012).

The iron and steel industry has an important role in developing technology. A country's production and consumption of iron and steel are closely related to that country's economic power and development. The reason for the close relationship between the developments observed in the iron and steel industry and the process of development is because iron and steel products provide input to all industries (ICMM, 2012). The world iron and steel industry have been significantly developing in the past 30 years. This led to a rapid increase in demand for iron ore (Xiong et al., 2015).

The metals and mining sector is an industry devoted to the site and extraction of metal and mineral reserves around the world. Global metal and mineral reserves are mined for the benefit and then used in jewellery, manufacturing applications, and investments. The aim of mining activities which are one of the factors that directly affect the economy of every developing and industrializing country is to find the minerals needed by various industries and to bring them to the place and to offer them to the consumers. The ores located above and below the ground can sometimes be of high grade; such ores can be sold directly to various industries and metallurgical plants by performing very simple operations (Orel and Ümit, 1967). In the economic literature, Ricardo's

conventional wisdom is that high grade; low cost, mineral deposits are mined first. Because the quality of the ores is presumed to be gradually decreasing over time, non-renewable resource extraction models have historically focused on reserve size. (Marvasti, 2000).

Since the excessive extraction of high-grade iron ore reserves has resulted in the rapid depletion of these ores, the need to extract and upgrade low-grade iron ores to more economically viable products are growing (Mhonde et al., 2017) and this is done by the so-called beneficiation which is the process by which the size of the ore is reduced and the gangue is separated from it, they are sold to smelting or other parts of the industry (Tolun, 1960). An iron ore is the primary iron source for iron and steel industries throughout the world. Hence, it is essential for steel production, which in turn is essential for maintaining a strong industrial base. In steelmaking, all iron ore (nearly 98%) is used. An iron ore is mined in approximately 50 countries “including Turkey”. Australia and Brazil are between the world's largest iron ore mine producers, Australia and Brazil producing 900 million metric tons and 490 million metric tons, respectively, in 2018 (U.S. Geological Survey). The main objective for mineral processing of any ore is to separate the concentrate from the gangue with high grade and recovery as possible. In the iron ores, the major gangues include (Si, Al, and P). Separation of iron concentrate from these complex gangues represents the main problem and it is kinda difficult. To separate iron concentrate from these complex gangues and to prepare it for industrial usage, iron ore processing goes through several stages that include: crushing, screening, grinding, classification and concentration. In addition to the conventional beneficiation methods of the iron ores, there are some chemical beneficiation that includes carbothermal roasting reduction (Zhu et al., 2020) and microwave roasting reduction (Chen et al., 2017).

In this study, the objective was to investigate of the ability to benefit specularite ore to iron concentrate grades that can be used by the industry (>88% Fe_2O_3) by shaking table, wet magnetic separation, microwave heating followed by

wet magnetic separation and roasting followed by wet magnetic separation, falcon concentrator, reverse anionic and reverse cationic flotation. Besides, finding out the effective separation parameters for high Fe_2O_3 grades and recoveries was another aim.

1.1. General Information

Iron is estimated to account for 32.07% of the Earth's mass, its elemental abundance varies between about 5% of the Earth's crust and as much as 80% of the planet's core (Morgan and Anders, 1980). The most commonly used iron-bearing minerals contain iron compounds as follows: hematite, Fe_2O_3 (70% Fe); magnetite, Fe_3O_4 (72.4% Fe) and of much less importance are: limonite, $2Fe_2O_3{\cdot}3H_2O$ (60% Fe); siderite, $FeCO_3$ (48.3% Fe); pyrite, FeS_2 (46.6% Fe) (Biswas, 1981; Filippov, 2014). Most of the iron ores in the world are in the form either magnetite (Fe_3O_4)) or hematite (Fe_2O_3) (Yang et al., 2017). In iron ores, the content of iron decreases according to the number of impurities present in the ore. More specifically, ores with Fe contents more than 65 % are classified as high-grade ores; 62-64% medium (or average) grade ores, while ores with Fe contents less than 58% are considered to be low-grade ores (Sparks and Sirianni, 1974; Uwadiale, 1989; Cheng, 1999).

Table 1.1 shows details of common iron ores and associated gangue minerals present in many deposits of iron ores (Waychunas, 1991). The three most common iron ore minerals are magnetite, hematite, and goethite, which together account for an estimated > 99 % of the iron minerals found in the world's seaborne-traded iron ores in 2012. The chemical composition of direct-ship and upgraded iron ores can be classified into high-grade hematite ores with 64–67 % Fe and low SiO_2 and Al_2O_3, primarily from Brazil and West Africa (e.g., Simandou); many Australian and other iron ores with ~60–62 % Fe; high-LOI martite-goethite Australian ores; and high-LOI Australian CID iron ores (Clout et al, 2015).

Table 1.1. Definitions of common iron ore and gangue mineral.

Iron Ore minerals	Chemical formula	Description
Hematite	Fe_2O_3	Iron oxide
Magnetite	Fe_3O_4	Primary iron oxide
Goethite	FeOOH	Most abundant iron oxyhydroxide: yellow, brown and while
Maghemite[a]	$Fe^{3+}[Fe^{3+}_{1.67}[\ ]_{.33}]O_4$	Intermediate iron oxide
"Kenomagnetite"[b]	Fe_{3-x} (X) $_xO_4$	Intermediate metal-deficient phase
"Martite"	Fe_2O_3	Hematite that has replaced primary magnetite
"Hydrohematite"[c]	$Fe_{(2-x)/3}(OH)_xO_{3-x}$	A defect solid solution where OH^- ions replace oxygen
Gangue mineral	**Chemical formula**	**Description**
Quartz	SiO_2	The common oxide of silicon
Kaolinite	$Al_4(Si_4O_{10})(OH)_8$	Clay
Gibbsite	$Al(OH)_3$	Clay
Minnesotaite[d]	$(Mg, Fe)_3Si_4O_{10}(OH)_2$	Fe-Mg silicate in iron formations
Stilponmelane	$(K,NA,Ca)_{0.6}(Mg,Fe_2+,Fe^{3+})_6Si_8Al(O,OH)_{27}$ 2-4H_2O	Common silicate in iron formations
"Chlorite"[e]	$(Fe, Al, Mg)_3(Si,Al)_2O_5(OH)_4$	Silicate
Pyrite	FeS_2	Iron sulphide
Pyrolusite	MnO_2	Manganese dioxide
Siderite	$FeCO_3$	Fe-carbonate
Ankerite	(Fe, Mg, Mn) $(CO_3)_2$	Ca-Fe-Mg-Mn carbonate

(X) represents cation vacancies. Formulae are from Howie et al., (1992); (a) From Waychunas (1991); (b) From Morris and Wolff, (1985); (c) From Wolska and Szajda, (1985); (d) From Miyano, (1987) and (e) From Klein and Bricker (1977).

Concentrates of magnetite and hematite ores used for direct reduction purposes are generally higher in grade (64-67% Fe) with low impurities or >67% Fe grade with very low impurities, which reflects more comprehensive beneficiation, usually with fine grinding followed by separation methods like magnetic separation or sometimes gravity or reverse flotation to reduce impurities. Higher-grade reserves are consumed and lower-grade reserves are evaluated for both direct-ship and beneficiated products due to the continuous decline of the chemical composition of many fine iron ore products with time. It is expected that the grades of iron ore products will continue to decline in the future, therefore higher-grade reserves for both direct-ship and beneficiated products will slowly be substituted in the future with beneficiated fine ores and magnetite concentrates derived from Banded-Iron Formation (BIF) (Clout et al., 2015). Common gangue minerals and elements that may have a negative effect on downstream processing were identified by Clout (1998). Table 1.2 shows the effects of deleterious gangue and minor/trace elements on downstream process performance.

1.1.1. Iron Ore Deposits

There are many types of deposits of iron ores that have historically been important in the production of iron ores and other types of metalliferous deposits where iron ore is produced as a by-product, but none are as important as iron formation ores. (Clout and Simonson, 2005).

Iron formation-hosted iron ore deposits account for the majority of the world's current production and above 31.000 Mt of resources of high-grade hematite iron ore. Based on the geological age and texture of the iron formation host, the iron formations were subdivided into three subtypes (Gutzmer and Beukes, 2009). Generally, the largest deposits of iron ore are found where paleoenvironmental conditions allowed the largest iron formations to accumulate. High-grade economic raw materials do not form, however, without upgrade the iron formation, usually, that grade increasing from 30 wt.% iron content in an

unenriched iron formation to 56–68 wt.% iron content in high-grade hematite and martite- goethite ores (Clout et al, 2015). According to Fitch Solutions Macro Research (in its report that was released in November 2019), global iron ore production will grow from 2,850 Mt in 2019 to 3,119 Mt in 2028. This growth would be mainly driven by India and Brazil (Business Standard, 2019).

Table 1.2. Effect of deleterious gangue and minor/trace elements on downstream process performance.

Deleterious phase/ element	**Process stage**	**Effects**
Sideritic carbonates	Sintering and pelletizing	Lowers strength of sinter and pellets due to increased porosity once carbonates are calcined
Clays (>5%)	Sintering and pelletizing	Lowers strength of sinter and pellets due to increased melt viscosity due to Al_2O_3
Alkalis (e.g., K_2O>0.09% and Na)	Sintering, pelletizing, BF	Lowers melt temperature, corrosion of BF refractory bricks
P (>0.08%)	Metal production	Removal cost
Base metals and heavy metals	BF, sintering	Removal costs, especially reprocessing of base and heavy metal-rich dust
Al_2O_3 (>2.8%)	Sintering, BF	Higher levels progressively increase melt viscosity and hence increase fuel rates
SiO_2 (>5%)	Sintering, BF	Increased use of limestone since the ratio of CaO/SiO_2 must be fixed
Mn (>0.9%)	Steelmaking	Excess levels require dilution with low Mn-bearing ores to maintain steel properties
Cu (>100 ppm)	Sintering,steelmakig	Catalyzes dioxin formation during sintering, must be diluted with low Cu ores to maintain steel quality
Cl (>500 ppm)	Sintering, BF	Increased dioxin (toxic) and NO_x emissions, reduces the efficiency of electrostatic dust precipitators and increases BF refractory wear
S(>0.08%)	Sintering, BF	Increased So_x emissions and higher MgO levels required to partition S into BF slag
Ti(>1.0%)	Sintering and pelletizing	The lower physical strength of pellets and sinter

According to reports submitted by 64 countries to the World Steel Association, global crude steel production reached 151.5 Mt in October 2019. Global production decreased by about 2.8% compared to October 2018 (Fig.1.1) (www.recycling-magazine.com).

Due to the inadequate level of reserves, iron ore production in Turkey has not changed significantly over the years. Turkey produced iron ore of 4.7 Mt in 200 8, compared to 4.6 million tons in 2005. Turkey is dependent on imports of iron ore because of the low iron ore production. Turkey's iron ore imports increased by 18 % in 2012 and reached 7.8 Mt. The value of imports of iron ore was 1.15 billion dollars. In 2012, Turkey's total iron imports from Brazil reached 4.6 Mt, which constitutes 58% of Turkey's total iron ore imports. Sweden accounted for 15%, Russia constituted 9.2% and Ukraine constituted 8.9%. South Africa, Canada, and other countries constituted the remaining 8.4% of the imports (www.celik.org.tr.com). According to Mineral Research and Exploration (2017), iron ore reserves in Turkey are found in the provinces of Balıkesir, Sakarya, Kırsehir, Adana, Hatay, Kayseri, Sivas, Erzincan, Malatya and Bingöl, Turkey has an estimated 82.5 Mt of iron ore reserve. Turkey’s crude steel production for September 2019 was 2.7 Mt, down by 6.9% in September 2018. A significant decrease in the production of crude steel in Turkey is observed this year during the first nine months of 2019 compared to the same period last year despite the increasing global demand for the steel industry, which solutions must be worked to increase the production of crude steel in Turkey by increasing the extraction and processing of the existing crude in mines inside Turkey to meet the shortage, especially since a large part of iron ore used in the steel industry in Turkey is imported from abroad.

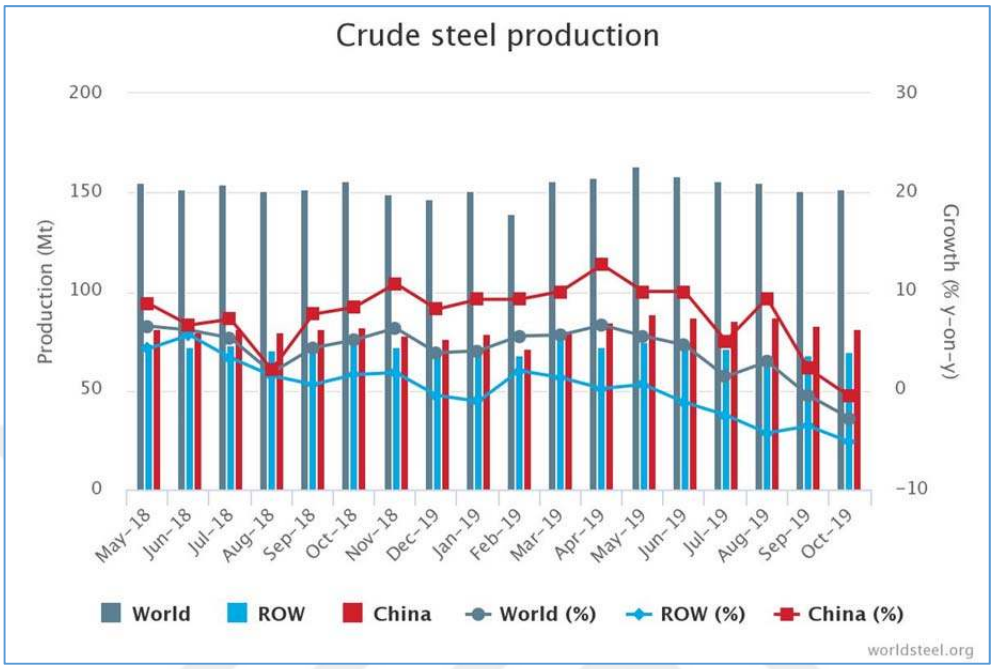

Figure 1.1. Crude steel production in the world and china.

1.1.2. Separation Methods of Iron Ores

Iron ores; according to the formation methods, simple separation methods such as (crushing, screening, classification, discharge, manual extraction) can be applied to product to sell the saleable products. However, Fe gain efficiency of these simple separation methods not a large proportion compared to those that need to be enriched by other separation methods. Therefore, these simple methods are supported by several beneficiation methods such as magnetic, gravity, and flotation methods when the reserve is sufficient. Gangue minerals such as quartz, calcite, dolomite, clay, feldspar, and carbonaceous materials may associate with iron ore can also contain some other minerals such as silica, phosphorus, potassium, zinc, sulfur, and sodium (Astrup, 1998). Problematic elements like P, K, and Na will reduce the profitable value of iron ore to a point where the ore is valueless (Davies, 1978).

This is why the presence of such impurities (phosphorus, silica, Al_2O_3, Cu, Zn, Pb, As) in iron ores causes various problems in the granulation and smelting processes in the blast furnace and limits the exploitation of the iron ore. Therefore, iron ore is enriched in different separation methods to increase the concentrate iron ratio and to remove these unwanted impurities. Conventionally, beneficiation of iron ore involved the use of different sequence of steps such as crushing, grinding, concentrating, or separating by size or weight such as screen and/or specific gravity, such as a hydraulic classifier or a magnetic classifier and concentration using reagents of flotation, such as froth flotation (Forciea, 1980). According to Forciea, 1980, the ore is ground in ball mill at a size of about - 300 μm. It is then transferred straight to a magnetic separator, where all the material that is released and nonresponsive is discarded. The magnetic concentrate, which usually contains between 10% to 30% silica, is then transferred to the flotation process. In high-grade iron ore which charged directly to a furnace, removes silica and alumina is necessary to decrease energy costs. Especially when we know that silica in the kiln requires extremely high temperatures. Both silica and alumina build up as a coating in the kilns, decreasing the effectiveness of the kilns over time (www.cdeglobal.com).

1.1.3. Gravity Separation Methods

There are some reasons for using gravity separation (GS); to reject waste as a pre-concentration step, recover malleable and/or friable heavy coarse minerals from circulating loads of the grinding circuit, to clean low weight Recovery bulk concentrates, to scavenging plant tailings, to pre-concentrate heavy minerals to minimize downstream processing costs, to concentrate some heavy minerals, to generate a concentrate of valuable metals that can go directly to a refinery rather than a smelter (Balasubramanian, 2017). To put it simply, the separation of several minerals by gravity is the product of their relative movement in response to gravity and one or more other forces, which often involves the resistance of a viscous fluid

to motion. The factors that are important in determining the relative motion of a particle in the fluid include the specific gravity, size, weight, and shape of the particle not only in absolute terms but also relative to all other particles in the system. It is a characteristic of gravity separation devices that, for the particles to travel relative to each other, must be held slightly apart at some stage of the process, or ' dilated' by the superimposed force. The operation of gravity concentration devices is explained essentially by four mechanisms: density; flow film; stratification and horizontal shear (Falconer, 2003).

The shaking table is a gravity separation device that has been used for many years. The design of this device did not change much, the change was through multi-deck tables (up to three levels) which led to an increase in capacity relative to floor area. Shaking tables are usually only used during cleaning stages due to their low potential. There are different kinds of wet tables, including the Wilfley, Deister, and Holman, designed to handle either fine feed or coarse. Variables include: the angle of the deck, length of, frequency of stroke, splitter positions, feed rate, and density separation. The advantages of shaking tables include (a) highly selective, with high upgrading ratio if used correctly, and (b) the ability to observe separation and to make adjustments. The disadvantages include (a) low capacity, (b) large floor area requirements, (c) needs frequent operator attention, checking and adjustment, and (d) feed requires sizing (Falconer, 2003).

Falcon Concentrator is a combination of a sluice and a continuously operating centrifuge. Capable of operating at a high rotational speed and therefore “gravity” force, it enables the separation of fine particles with various specific gravity. The bowl the heavier particles react more to the forces acting upon them than the lighter particles. This results in the migration of the heavier particles into the surface in contact with the bowl within the slurry stream, while the lighter particles tend to move with water to the top of the slurry. The separation then takes place by removing the lower (higher specific gravity) portion of the slurry through a lip/slot collection, through which the flow is regulated by several orifices that

open and close in a controlled manner, removing the concentrate from the mainstream that discharges to the tails (Fig.1.2). The Falcon concentrator variables include; (a) speed of rotation or spin; (b) pulse frequency of concentrate orifices; (c) feed rate and density (Falconer, 2003). The particles of the feed streams are subjected to the gravitational force of up to 300 G's and are separated along the smooth spinning rotor wall according to effective specific gravity. Advantage of Falcon concentrator include; (a) able to treat particles in size down to 15–20 μm, (b) relatively simple mechanically and robust, (c) relatively high capacity and (d) relatively low operator attention. While the disadvantages of this concentrator include: (a) generally low upgrading ratio, (b) unable to see separating surfaces and (c) requires feed to be screened to less than opening size of concentrate orifices to prevent blinding (Falconer, 2003).

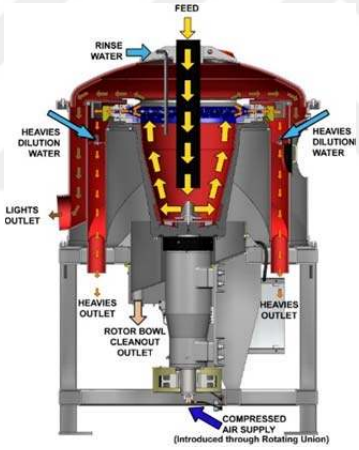

Figure 1.2**.** Partial cross-section of a Falcon continuous separator.

1.1.4. Magnetic Separation Methods

Due to its many benefits such as high efficiency and low operating costs in addition to being environmentally friendly, magnetic separation technology plays an important role in the process of upgrading low-grade iron ores. So, it is easier to upgrade specularite with this separation method compared to martite and hematite (Xiong, 2015). Various materials that exist on Earth have various magnetic

properties, ranging from strongly magnetic, weakly magnetic, zero magnetic, and antimagnetic. Magnetic susceptibility can represent these minerals. Magnetic separation methods depend on the difference between the magnetic susceptibility to iron minerals and quartz. Table 1.3. displays the basic groupings of magnetic separators used most frequently (Das, 2008). From the table 1.3., noted that the magnetic susceptibility of specularite ore is weak when compared to the magnetite ore that has a high magnetic susceptibility. There are methods recently used such as magnetic roasting that aimed to increase magnetic susceptibility/magnetic properties of iron ores such as hematite and goethite. Reduction roasting includes the conversion of iron oxides such as hematite and goethite in the presence of carbonaceous reductants to magnetite (a ferromagnetic product) that can be separated from the gangue by magnetic separation methods at a low magnetic field intensity (Roy et al., 2020). Microwave irradiation treatment recently gained more interest in research into mineral processing due to its selective effect and low power consumption. Microwave roasting depends on the material’s ability to convert electromagnetic energy to heat while the material's ability to absorb microwave energy is dependent on its dielectric constant (Lovás et al., 2001). Comparing with iron ores, transparent material such as silica is not a good absorbent. Good absorption of the iron ore of the microwave leads to increase in surface temperature in shorter time compared to conventional roasting. (Barani et al., 2011).

In this research, wet magnetic separation was applied depending on the difference in the magnetic susceptibility of both iron concentrations and gangue present in the specularite ore. In addition to applying microwave heating and roasting to rejected-products to increase their magnetic properties and then applying wet magnetic separation to obtain iron ore concentrates with a high grade and recoveries as possible.

Table 1.3. Magnetic susceptibilities of iron ore-related minerals ((Das, 2008)

Mineral	Susceptibility (10^{-6} m^3/kg)	Chemical formula
Magnetite	625–1156	Fe_3O_4
Martite	6.2–13.5	Fe_2O_3
Hematite	0.6–2.16	Fe_2O_3
Specularite	3.7	Fe_2O_3
Limonite	0.31–1.0	$2Fe_2O_3 \cdot 3–4H_2O$
Siderite	0.7–1.5	$FeCO_2$
Ilmenite	0.34–5	$FeTiO_3$
Quartz	0.0025–0.126	SiO_2
Feldspar	0.063	$KAlSi_3O_8$, Na $AlSi_3O_8$
Calcite	0.0038	$CaCO_3$
Spodumene	0.82	$LiAl(Si_2O_6)$
Chlorite	0.38–1.13	$(Mg, Fe)_5Al(AlSi_3O_{10})(OH)_8$
Garnet	0.79–2	$Mg_3Al_2(SO_4)_3$
Biotite	0.5–6.5	$K(Mg,Fe)_3(Si_3AlO_{10})(OH,Fe)_2$
Olivine	0.17	$(Mg, Fe)_2SO_4$
Pyrite	0.34	FeS_2
Apatite	0.007–0.142	$Ca_5(PO4)_3F$

In general, a magnetic separator is classified as low intensity if its maximum field intensity is less than approximately 2000 G (H= 1.6 x 105 A / m, B=0.20 T). High magnetic separators (HIMS) have field strengths of 10 to 20 kilogauss in general. Low-intensity magnetic separators (LIMS) are used to treat highly paramagnetic and ferromagnetic minerals like iron and magnetite. High-intensity magnetic separators (HIMS), such as hematite, are used to treat weakly magnetic minerals (Table 1.4) (Das, 2008).

Table 1.4. Basic groupings of common magnetic separators (Das, 2008).

Wet magnetic separators	***Dry magnetic separators***
Low: Drum separators	*Low: Magnetic pulleys*
Bowl traps	*Rectangular suspended magnets*
Magnetizing coils and blocks	*Magnetic drums-radial pole types*
Demagnetizing coils	*Magnetic drums-axial pole types*
	Plate magnets
	Grate magnets
High: High-intensity separators	*High: Induced roll magnetic separators*
	Crossbelt magnetic separators
	Ring-type magnetic separators

1.1.5. Flotation

Flotation is a selective concentration and can be used for specific separations from complex ores like copper-zinc or lead-zinc. Initially conceived to treat sulfides of copper, lead, and zinc. The flotation field has now expanded to include platinum, nickel, and gold-hosting sulfides, and oxides such as hematite and cassiterite, oxidized minerals such as malachite and cerussite, and non-metallic ores such as fluorite, phosphates, and fine carbon (Wills et al., 2015). Recently, the flotation method has only been used for hematite iron ores. There are two ways for flotation of iron ores that include direct flotation or reverse flotation. In the former, iron oxide is floated. Most widely used are anionic reagents like petroleum sulfonates or fatty acids. Structurally, these reagents have negatively charged ionic -heads- attached to organic (tails) of long chains. Adsorption of these substances to surfaces of mineral particles bearing iron causes them to float with air bubbles. Conversely, it is possible to float waste rock (mainly silica) with cationic reagents (positively charged heads) if the basicity is kept high (pH 11-12) and other organic depressants, such as synthetic organic polymers or starch, are added to prevent the iron oxide from floating. This is commonly known as the reverse flotation. Usually, silica is floating as a flotation reagent using an amine. But "reverse"

flotation using anionic collectors also applies to activated rock minerals (e.g., silica C_2 + activation) where the iron-bearing minerals are depressed (Vidyadhar, 2007). The reverse cationic route is by far the most widely utilized method. Quartz is floated with ether amines ($R–(OCH_2)_3–NH_2$) partially neutralized with acetic acid. The degree of neutralization is an important parameter. Higher neutralization degrees enhance the collector solubility but impair the flotation performance. (Araujo et al., 2005).

The process of recovering material from the pulp by flotation consists of three mechanisms: (a) selective attachment to air bubbles (or "true flotation"); (b) entrainment in the water which passes through the froth; (c) physical entrapment between particles in the froth attached to air bubbles (often referred to as "aggregation") (Wills et al., 2015). The most important mechanism is the attachment of valuable minerals to air bubbles and represents the majority of particles that are recovered to the concentrates. Although true flotation is the dominant mechanism for recovering valuable mineral, the efficiency of separation between valuable mineral and gangue also depends on the degree of physical entrapment and entrainment. Unlike true flotation, which is chemically selective to the mineral surface properties, both gangue and valuable minerals alike can be recovered by entrainment and entrapment. Only if they are water-repellent, or hydrophobic, can the mineral particles attach to the air bubbles. The air bubbles can only continue to support the mineral after they have reached the surface. Having reached the surface, the air bubbles can only continue to support the mineral particles if they can form a stable froth, otherwise, the air bubble will burst and drop and particles drop to the bottom of the cell. To achieve these conditions, the various chemical compounds known as flotation reagents must be used (Wills et al., 2015). In the flotation process, some reagents such as frother, collector, and depressant are usually added to the flotation process with the particular dosage to achieve an optimal process condition (Haryono et al., 2017) and to achieve effective flotation (Vidyadhar, 2007). These reagents are added to the flotation

system to change their interfacial properties of the solid-liquid-gas system (Srdjan, 2017).

The selection of which reagents should be applied and the design of processes during the iron ores flotation with complicated silicates should rely on the mineralogical composition of the ore and is connected to the crystal chemistry properties of the mineral deposit and the process flowsheet economics (Uwadiale, 1992, De Melo et al., 2017). The collector is the most important reagent used in the flotation processing of iron ore. Collector groups can be divided into two main groups, which include: (a) ionising and (b) non-ionising (Fig. 1.3) (Bulatovic, 2007).

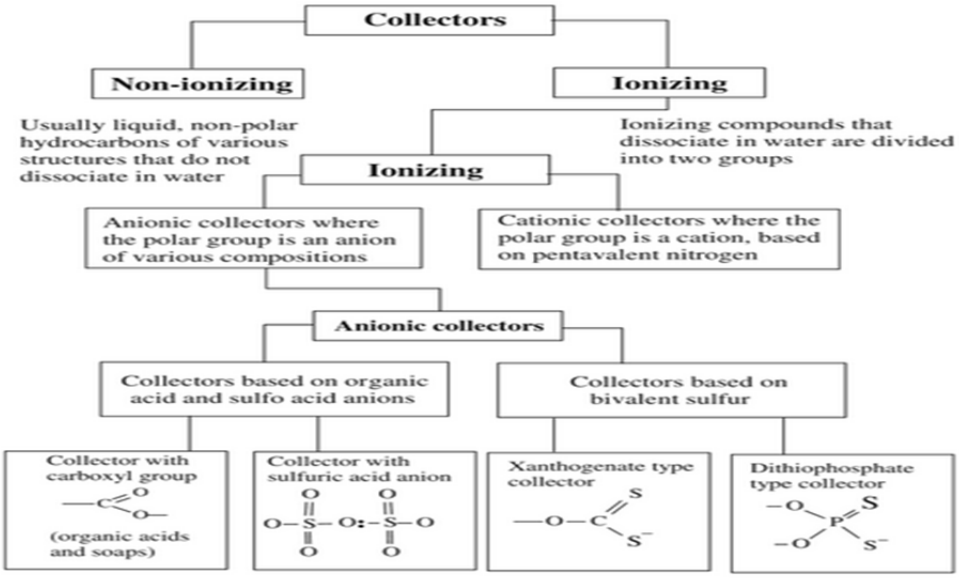

Figure 1.3**.** Classification of flotation collectors

Depression is also used to increase the selectivity of flotation by rendering certain minerals hydrophilic (water-avid), thus preventing their flotation sulfides (Wills et al., 2015). In the flotation of iron ores, starches are the universal depressants of iron oxides. Starch may be extracted from several vegetable species such as corn, wheat, cassava, potato, rice, arrowroot) (Araujo, 2005). Corn starch is

widely preferred in the flotation of metallic ores due to its good depressant properties and their low cost (Yang et al., 2017; Araujo et al., 2005; Kar et al., 2013). Frothers are compounds specifically used to create flotation froth. Most collectors used in the processing of iron ore, however, produce adequate froths. The general types of these frothing collectors include; long-chain carboxylates, sulfonates or sulfates, and amines. Pine oil, aliphatic alcohols, polypropylene glycol, polypropylene glycol alkyl ethers of polypropylene glycol, and cresylic acids are the most widely used frothers in iron ore flotation in practice. Five to eight carbon atoms, such as mixed amyl alcohols, methyl isobutyl carbinol (MIBC), and certain heptanols and octanols are the preferred alcohols (Vidyadhar, 2007). In the selective flotation of the minerals from most ores, modifying agents are usually necessary (Rao, 2014). Generally, the modifying agents are inorganic or organic additives (Vidyadhar, 2007). Significant progress has been made in increasing the selectivity of iron ore flotation over the past decade: this has largely been accomplished through the judicious use of old and new modifying agents (Vidyadhar, 2007).

2. LITERATURE REVIEW

Aydin (2009), in his study, investigated the suitability of beneficiation of three samples of low-grade hematite ores from Eğmir-Balıkesir, Turkey (57.44%, 40.22% and 26.63% Fe) by using conventional gravity separation equipment, jigs and shaking tables, and high-intensity magnetic separator. In this study, the experimental results showed that both high-intensity magnetic separation and gravity-based separation were effective in the concentration of low-grade hematite ores. The highest iron concentrate obtained by magnetic separation was 47.21% with 86.66 recovery % from 39.34% Fe of feed whose size -3 mm.

Lin et al. (2009), in this study, experimental research was conducted on magnetic hematite concentrates employing anionic reverse flotation method in the flotation column. The optimal conditions had been obtained at the circulation of the feeding quantity of 160 g/min, the pressure of 0.055 MPa, the collector dosage of 800 g/ton, and the starch dosage of 900 g/ton. The results show that it can be obtained the iron ore concentrate grade of 67.37% Fe due to the advantages of good selectivity and high recovery.

Turrer and Peres (2010), studied flotation depressants in reverse cationic flotation of iron ores for other mineral systems. The reagents that tested included corn starch, six carboxymethylcelluloses (CMC), three anionic lignosulfonates, guar gum, and four humic acids. Most of these depressants have poor selectivity. The silica content was increased in the flotation concentrate due to depressing both the hematite and quartz flotation.

Wang et al. (2011), investigated the iron ore concentrate grade and recovery for an iron ore contain specularite and hematite by several processes including gravity separation, high-intensity magnetic separation, high-intensity magnetic-gravity separation, and high intensity magnetic - reverse flotation. The results showed that the high-intensity magnetic-reverse flotation process can

produce an iron ore concentrate grade of 66.62 % Fe with 58.38 % recovery from an iron ore assaying about 35.00 % Fe.

AbdulRahman et al. (2012) investigated the responses of Nigerian iron ore deposit to magnetic separation a case study of Agbaja iron ore deposit which contains principally of goethite, with minor hematite, magnetite, siderite, kaolinite, and quartz. Particle size analysis was carried out over the range of 250μm. The result shows that the recovery of 76.7% concentrates (magnetic) and 10.7% tailing (non-magnetic) with the other 12.7% of the ore been lost in the process.

Seifelnassr et al. (2012), investigated the effectiveness of gravity and magnetic concentration techniques for the beneficiation of Wadi Halfa iron ore in Northern, Sudan, it is a low-grade type of ore with high silica content, more than 45% SiO_2 and iron content of about 35% Fe. About 64% Fe with the recovery of about 70% was achieved by these two separation methods that were prepared in the second stage of processing via a high-intensity magnetic separator.

Seifelnassr et al. (2013), investigated the amenability of low-grade iron ore for upgrading by gravity and high-intensity magnetic separation methods. The chemical content of this ore was 36% Fe and 48% silica. The results showed that the optimum degree of grind is around 150 μm and at this particle size by using two-stage separation, roughing, and cleaning, it was possible to obtain an iron concentrate grade of 64% with the recovery of 72% Fe grade.

Roy and Das (2013), carried out a study for, the physical processing of low-grade (28% Fe) iron ore was investigated. The results showed that iron ore with 28% Fe and 55% silica could be successfully upgraded to 65% Fe using physical beneficiation with about 20% recovery and a process flow consisting of tabling, wet magnetic separation, and flotation methods gives a suitable iron concentrate for pellet production from this ore.

Lima et al. (2013), tested the understanding of the mechanisms of reagents interaction for the quartz collector and iron oxides depressant to improve the separation selectivity at high amine dosages in cationic flotation system in three

different size ranges. In this study, the results showed that at -150+45 μm particle size, enhanced quartz depression as a consequence of an increase in amine dosage was observed. The effect is clearer at pH = 9.5 and higher conditioning speed (2000 rpm).

Kar et al. (2013) studied four different starches, namely; soluble starch, corn starch, potato starch, and rice starch with different characteristics as depressants for hematite in cationic flotation using dodecylamine as the collector. According to the results they obtained, all the starches have effective depressant action for hematite at neutral to slightly alkaline pH. from the flotation studies of hematite and quartz mineral, with the presence of soluble starch and dodcylamine as the collector, quartz is floated better than hematite. In addition to that, the flotation of natural iron ore indicated that the maximum iron recovery can be obtained by using soluble starch as the depressant and the flotation of natural occurring banded iron ore using different starches has indicated that it is possible to obtain concentrate with 63–65% Fe grade with 85–88% recovery.

Omran et al. (2014) while investigating the effect of microwave treatment on Egyptian iron ore samples. In that study, during roasting processing, the samples were changed the microwave exposure time and power, as well as their reflection on the magnetic properties of iron ore before and after the roasting processing with the microwave, was analyzed using different methods. The results showed that at larger particle sizes and longer exposure times the microwave was more efficient. Besides, compared to the untreated sample with the recovery of 39.54% the iron recovery increased to 97.95% in the microwave treated sample. These results indicated that microwave radiation has a significant effect on hematite's magnetic properties through the formation of new and more magnetic phases that allow its separation from non-magnetic minerals.

Abaka-Wood et al. (2016), have investigated the effect of the physicochemical properties including; zeta potential, contact angle, and floatability of three sample ores (monazite, hematite, and quartz) by the micro flotation

method. In this investigation sodium oleate, sodium dodecyl sulfate, and hydroxamic acid were used as collectors. The results showed that the contact angle of the minerals are depended on the collector type used and the contact angle of the monazite, hematite, and quartz were 49°, 47°, and 22°, respectively which mean that monazite has more floatability than hematite and quartz under the same conditions.

Rath et al. (2016), he studied tests of reduction roasting and magnetic separation of complex Indian iron ore with 57% Fe which could not be upgraded to 61% Fe using a flow sheet consisting of traditional unit operations such as scrubbing, jigging, magnetic separation or flotation due to an inadequate liberation of iron oxide and the fine dispersion of silica particles within the hematite grains. In this study, the ore was subjected to reduction roasting with conventional and microwave roasting and then it was followed by magnetic separation. The results showed that compared to conventional roasting, the reduction using the microwave roasting process could be achieved considerably in a shorter time. In both the processes, it was possible to obtain a high-grade concentrate of 63-65% Fe with the recovery of 85–90%.

Yang et al. (2017), investigated the solubilities of four corn starches (as depressant) with different amylose/amylopectin contents which were measured at different temperatures and pH in the flotation system for hematite ore. The results showed that the starch solubility was enhanced with the increase of temperature or pH. The flotation tests showed that mineral recovery was decreased by increasing starch solution preparation temperature or pH. The depressing ability of corn starch for hematite and quartz was positively related to its solubility.

Yu et al., (2017) studied the beneficiation of low-grade hematite ore fines containing carbonates with magnetization roasting and magnetic separation. The results showed that at a roasting temperature of 800 °C for 8 min, the hematite and siderite are almost completely converted into magnetite by 8 wt% coal. The highest magnetic iron concentrate 65.4 wt% with an iron recovery of 92.7% was obtained

under the optimized conditions of 800 °C temperature, 12% coal to ore ratio, 8 min roasting time, and 38 μm particle size. The results also show that due to the selective conversion of hematite and siderite into magnetite caused by magnetization roasting, which facilitates their separation from non-magnetic minerals, the magnetic susceptibility and magnetism concentration of hematite ore can be dramatically increased.

Vapur et al. (2018), this study investigated the effects of iron ore roasting (hematite-magnetite conversion) on iron ore beneficiation through high-intensity wet magnetic, low-intensity dry magnetic and Falcon gravity separators separation methods to the roasted and unroasted ores at the optimum test conditions. The results in this study indicated that there are positive effects of roasting in terms of concentrate recovery for the wet magnetic separation and gravity separation methods while dry magnetic separation experiments, the desired saleable iron ore concentrates were not achieved.

3. MATERIALS AND METHODS

3.1. Materials

3.1.1. Preparation of Specularite Sample

Specularite ore used in this study was obtained from Feke district of Adana in Turkey. Approximately 200 kg of specularite ore extracted from the ore bed was taken and brought to the Mneral Processing Laboratories of Mininf Engineering Departmetn Çukurova University, Faculty of Engineering and Architecture. The sample preparation flowsheet is given in Fig. 3.1.

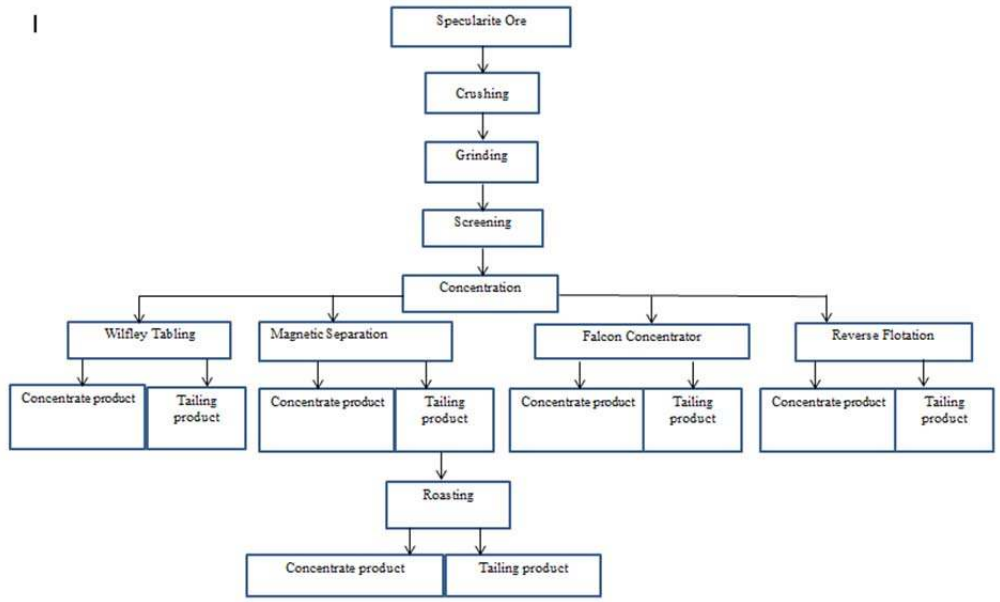

Figure 3.1. The sample preparation flow chart.

Fig. 3.2. showed the microscope images of specularite ore sample under the reflected light microscope (Nikon-YS100). The microscope images were taken for different particle size and these microscope images were (a) -38 µm, (b) -74 µm, (c) -150+74 µm, (d) -500+150 µm, (e) -1000+500 µm and (f) -2000+1000 µm. In the microscopic images, white-colored particles are quartz minerals while red,

black, and dark gray colored particles are iron minerals such as goethite, hematite, and specularite.

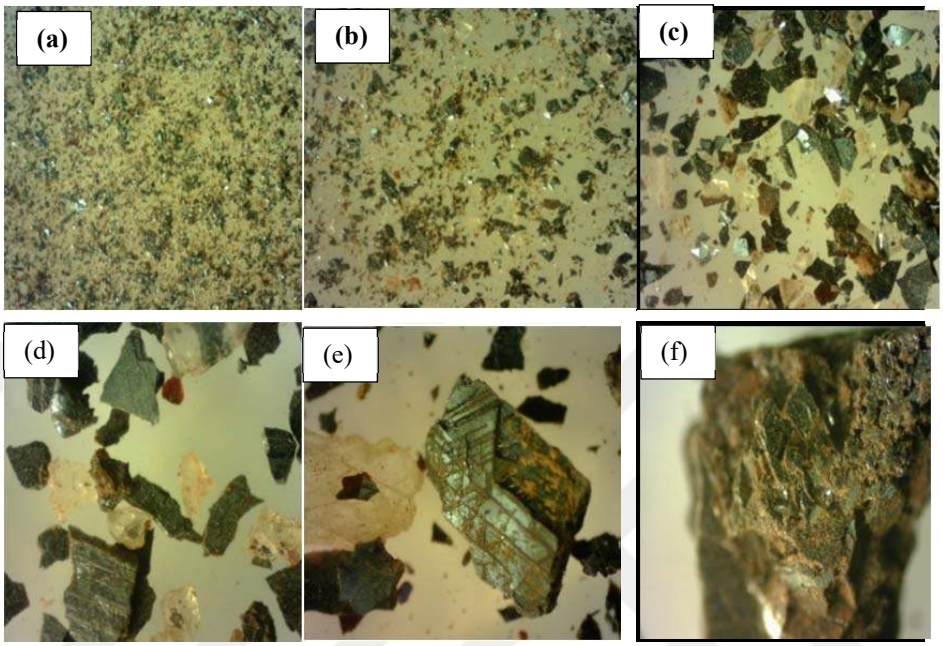

Figure 3.2**.** Microscope images of the different sized samples.

The specularite sample, which had the maximum particle size of 95 mm, was crushed under to 10 mm using a laboratory-type jaw crusher (Fig. 3.3. (a)). The crushed sample was and ground for 12 min, 18 min, 24min, 30 min and 36 min using a laboratory grinding ball mill (Fig. 3.3.(b)). (The charge volume was 45-50 % of the internal volume of the mill with 15 kg balls in various diameter) at 60 rpm drum speed as the optimum rotating value in dry conditions.

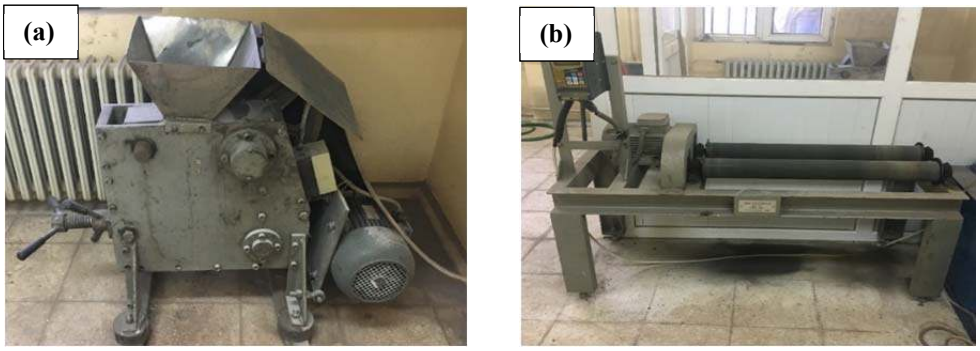

Figure 3.3. Laboratory-type jaw crusher (a) and ball mill rotator(b).

In all the experiments of the methods that were used in this research, it was taken the sample obtained from that was ground for 12 min at 60 rpm drum speed. Then samples were sieved by RETSCH Sieve Shaker AS 200 (Fig. 3.4) for 12 min into different degrees of fineness: -2000+1000 µm, -1000+500 µm, -500+150 µm, -150+74 µm, -150 µm, -75 µm, -74 µm, -150+38 µm and -106 + 38 µm, -425+250 µm and -250+75 µm. In flotation tests (reverse anionic and reverse cationic flotation), -38+0 µm sized materials were rejected. The general particle size distribution of the ground sample for12 min is shown in Fig. 3.5. The Fe contents of the each particle size fraction after sieving were also determined and they were shown in Fig 3.6. as well. The particle size distribution of the sample for the 18, 24, 30, and 36 minutes are shown in Fig. 3.6.

Figure 3.4**.** RETSCH Sieve Shaker AS 200 used in experiments.

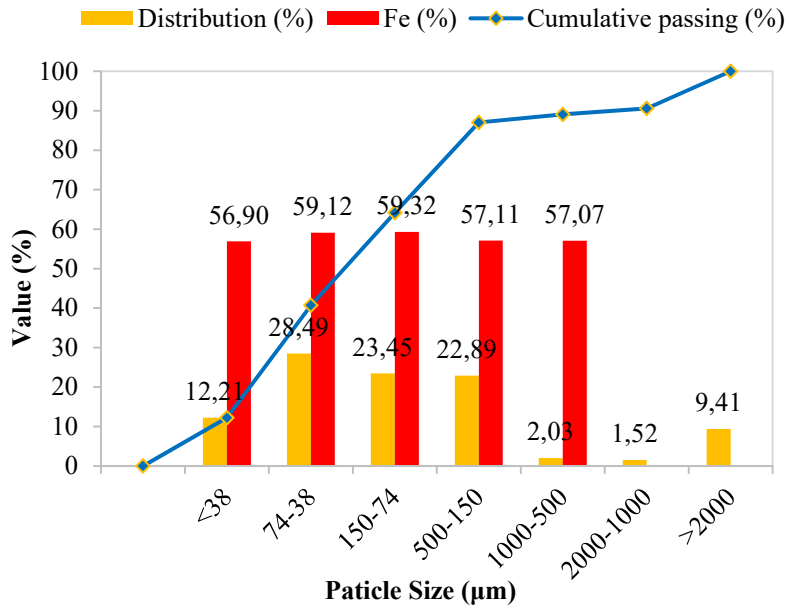

Figure 3.5. The particle size distribution of the sample (12 minutes ground).

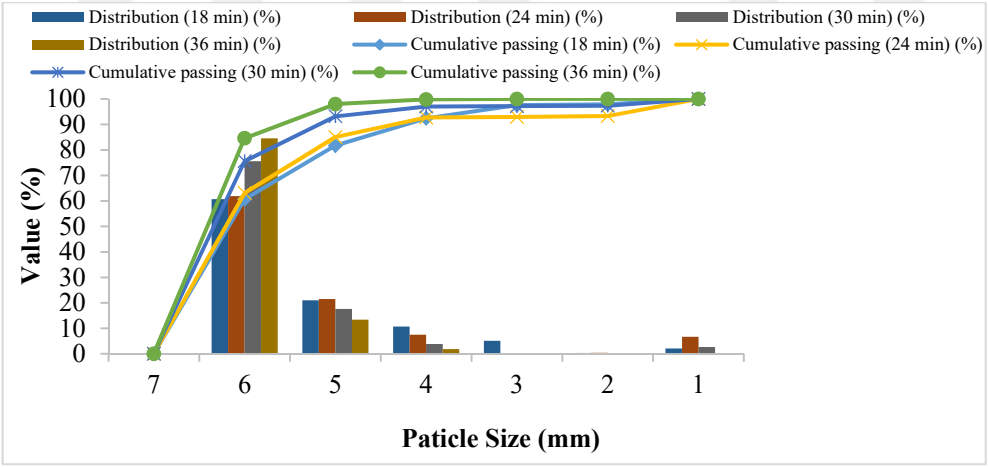

Figure 3.6. The particle size distribution of the sample ground for 18, 24, 30 and 36 min.

3.1.2. Chemical Analysis of Specularite Ore

The chemical analysis of the samples have been determined by XRF analysis method using Minipal-4 Panalytical device. The results of the chemical analysis have been shown in Table 3.1. The sample contains a high ratio of Fe_2O_3. Besides, the results of the analysis showed high content of both silica and calcium oxide. According to chemical analysis for the specularite ore, there are no impurities that have negative effects on sintering, blast furnace, or steelmaking such as Al_2O_3, K_2O, Na, Zn, Pb, Cu, Cl, S, Ti, P, Mn (>0.9%) and Cu. The mineral phases of the sample have been revealed by XRD analysis method. Rigaku Minflex XRD device has been used for this purpose. XRD patterns have been interpreted with the PDXL software with the current database (Fig. 3.7.). After each experiment in all the methods that were used in this study, both concentrate and tailings were filtered, dried at 105 °C and then weighed by analytical balance type AND GR-200 to calculate the recovery of the iron ore concentrate to evaluate the efficiency of the parameters on which they depend on the beneficiation test by using the following equation: Where R is the recovery of the iron ore concentrate; F is the weight of the feed solids (g), C is the weight of the concentrate (g), f is a metal grade in the feed (%), c is the metal grade in the concentrate (%) (Wills et al., 2015; Rao, 2016).

$$\% \text{ Recovery} = R = \frac{C\,c}{Ff} \times 100 \quad (1)$$

Table 3.1. Chemical analysis of the specularite sample

Content	Fe_2O_3	SiO_2	CaO	SO_3	La_2O_3	MnO	Cr_2O_3	CuO	Rb_2O	MoO_3	V_2O_5
%	82.78	8.11	8.46	0.3	0.11	0.02	0.02	0.04	0.03	0.01	0.02

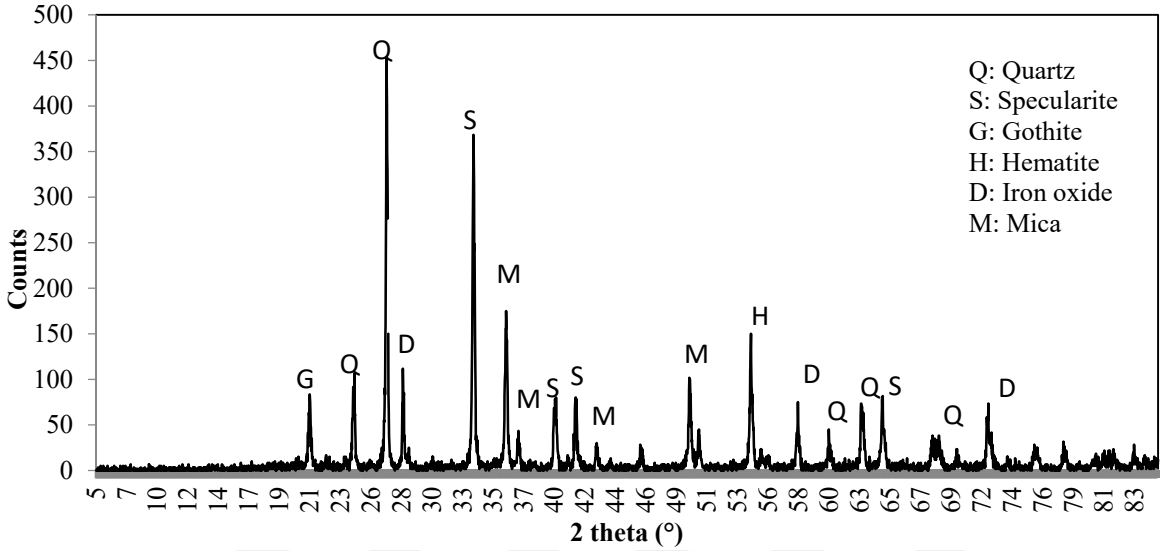

Figure 3.7. XRD analysis of the specularite ore.

3.2. Methods

The concentration of a specularite ore was investigated by applying physical and physiochemical separation techniques. Physical separation techniques included shaking table, wet magnetic separation, microwave heating, and roasting followed by magnetic separation and Falcon concentrator while the physiochemical separation techniques were comprised of reverse anionic and reverse cationic flotation. In this study, Box Behnken test design, which is one of the response surface methodologies, was used to evaluate microwave and conventional roasting process followed by wet magnetic separation, Falcon concentrator separation, reverse anionic and cationic flotation experiments result. Box Behnken test design was preferred due to the presence of more variables in the selected separation methods compared to other separation. It is also possible to determine the active parameters more quickly with fewer experiments in this design. The models were

created by using Design Expert 11 software. Table 3.2. shows methods and its experimental design based on the Box Behnken Design (BBD).

Table 3.2. Methods and experimental design based on the Box Behnken Design.

Method	**Factors and Ranges**				**Run Order**
Microwave Heating	Microwave Prower (0.54-0.9 KW)	Exposure Time (1-3 min)	Magnetic Feild Intensety (0.25-0.85 T)	-	15
Roasting	Temperature (450-650 C)	Exposure time (5-15 min)	Magnetic Feild Intensety (0.25-0.85 T)	-	15
Falcon Concentrator	Pressure (20-40 psi)	Frequency (35-55.33 Hz)	Particle Size (450-75 µm)	-	15
Anionic Reverse Flotation	Depressant (500-10000 g/ton)	Collector (500-1500 g/ton)	Collection Time (1-3 min)	-	15
Cationic Reverse Flotation	Depressant (400-600 g/ton)	Collector (200-400 g/ton)	Frother (50-150 g/ton)	Collection Time (2-4 min)	24

3.2.1. Shaking Table Experiments

Shaking table experiments were carried out using laboratory Wilfley type shaking table (Dimensions: 50x100xX35 cm (W in x L x W out)) Fig. 3.8. In this separation method upgrading the specularite ore was according to particle size and table tilt angle. The sample was dry sieved to two particle sizes: -74+0 µm and -150+74 µm. The table tilt angles were applied as 3°, 4°, 5°, and 7°. All the tests performed at 12 L/min water flow rate and 175 strokes/min shaking speed. Two products were obtained namely into concentrate (heavy minerals) and tailing (light minerals) products.

Figure 3.8**.** Wilfley laboratory type shaking table used in experiments.

3.2.2. Wet Magnetic Separation Experiments

Tests were carried out using laboratory type high intensity wet magnetic separator (Eriez L4-20). Beneficiation of specularite samples using this separation method divided into two stages. The first stage, samples were prepared according to particle size at five different size fractions: -2000+1000 µm, -1000+500 µm, -500+150 µm, -150+74 µm, -74 µm and separation tests carried out at a fixed magnetic field intensity of 0.75 T. All the tests were performed at 10% solid ratio by weight (25 g solid/225 ml water). The second stage was performed at 7 different magnetic field intensity; 0.25 T, 0.35 T, 0.45 T, 0.55 T, 0.65 T, 0.75 T and 0.85 T using optimum particle-sized sample (-150µm). This size was selected based on the results of the experiments of the first stage and the experiments in second stage were performed at a 20% solid ratio by weight (40 g solid/160 ml water).

3.2.3. Roasting-Magnetic Separation Experiments

Roasting encompasses reduction roasting studies of rejected-products that contain a high amount of the iron oxides that were obtained as a tailing (rejected-products) from the preliminary beneficiation test using magnetic separation. Rejected-products were subjected to microwave heating and conventional roasting to treat and to increase magnetic susceptibility of iron oxides which then was followed by magnetic separation to evaluate the efficiency of the magnetic field

intensity on the beneficiation test. The aim of these methods to increase the overall beneficiation recovery of iron concentrate.

3.2.3.1. Preliminary Beneficaion Experiments

Preliminary beneficiation of the samples was carried out by using magnetic separation, which was carried out using laboratory type high intensity wet magnetic separator (Eriez L4-20). Magnetic separation experiments performed at 3 different magnetic field intensity; 0.25 T, 0.55 T, and 0.85 T using optimum particle-sized sample (-150+38 μm) at 20% solid ratio by weight (40 g solid/160 ml water). Particle size was selected based on preliminary experiments.

3.2.3.2. Microwave Heating Followed by Wet Magnetic Separation Experiments

In the microwave roasting process, the non-magnetic product was treated in a kitchen type microwave oven (LG WAVEDOM, 2.45 GHz). Rejected products of the previous separation were treated in the oven for variable power levels and exposure times. The range of the microwave power was 0.54–0.90 KW while the exposure time was 1 to 3 min. The range of the microwave power and exposure time was selected based on preliminary experiments. In each test, 40 g of the non-magnetic sample was put in a quartz pan. It was approximately 10 mm in diameter and 40 mm in height. To minimize the impact of the field pattern variation in the microwave the quartz pan was placed in the same central position and microwave roasting started. After roasting up, the samples were then allowed to cool to room temperature in the microwave oven. The magnetic separation test was performed at 3 different magnetic field intensity; 0.25 T, 0.55 T, and 0.85 T and the experiments were performed at 20% solid ratio by weight (30 g solid/120 ml water)

3.2.3.3. Roasting Followed by Wet Magnetic Separation Experiments

In the roasting test, 40 g of non-magnetic product was mixed completely with 4 g of high-quality coal (10 %) have particle size -150 µm and it was put in a crucible made from aluminum and placed in a conventional furnace (Protherm brand) preheated to the desired temperature. The tests were performed under normal atmospheric pressure conditions. Non-magnetic product was treated in the oven for different temperature and exposure times. The range of the temperature was 450-650 °C while the exposure time was between 5-15 min. The temperature and exposure time range was selected based on preliminary experiments. The magnetic separation test was performed at a magnetic field intensity of; 0.25 T, 0.55 T, and 0.85 T, and the experiments were performed at a 20% solid ratio by weight (30 g solid/120 ml water). The factors and responses which were analyzed by using DOE,

3.2.4. Falcon Concentrator Experiments

Falcon concentrator test was carried out in the Mineral Processing Laboratories of the Department of Mining Engineering, İnönü University by (using Sepro-L40R3.0 Lenze Falcon concentrator) device (Fig. 3.9). It had the following specifications: maximum 75% solids by wt. %, 0–300 kg/hr feeding capacity, <1.7mm particle size, 4–20 L/min fluidization water, and 0–300G centrifugal force. The frequency was between 35-55.33 Hz and the pressure was ranged 20-40 psi, the particle size was dry sieved into three different fractions of -425+250 µm, -250+75 µm and -75µm. All the experiments performed at a 20% solid ratio by weight (100 g solid/400 ml water). In Falcon separator test, the experiments were analyzed by box Behnken design. In the Box Behnken tests, the variables were as the following; X1: water pressure (psi), X2: frequency (Hz), X3: maximum particle size (µm).

Figure 3.9**.** Falcon separator used in the study.

3.2.5. Reverse Anionic Flotation Experiments

The reverse anionic flotation tests were carried out in a batch Denver type flotation machine equipped with a volume of 1 L cell and operating shown in Fig. 3.20 at a fixed impeller speed of 1200 rpm. The solid/liquid ratio was 10% by weight and pH was 12.5 in all experiments. PH values were increased by the addition of sodium hydroxide granulated NaOH (pharm quality-pure grade >99 %) solution to the flotation cell. Comparing to acidic medium, the flotation of iron oxides and silica was stronger in the alkaline medium. As pH increases, the surface charge of oxides increases. The surface charges aim to become more negative, according to the previous studies (Fuerstenau et al.,1956; Smith et al., 1973). For these reasons, and based on the preliminary experiments, pH was adjusted at 12.5 for all experiments in this method. Methyl isobutyl carbinol (MIBC) was used in the experiments as frother was added to the flotation cell at a fixed dosage of 600 g/ton for all experiments. MIBC dosage and the particle size were selected based on the preliminary tests. MIBC was added to the cell one min before froth collection. The froth product was collected at certain time intervals. The particle size was -106+38μm. Corn starch is widely preferred in the flotation of metallic ores due to its good depressant properties and their low cost (Yang et al., 2017; Araujo et al., 2005; Kar et al., 2013). The dosages of corn starch were between 500 – 10000 g/ton. The collector in these experiments was Aero 343. Collector dosages ranged from 500-1500 g/ton. Conditioning time with iron depressant was 2 min

and the depressant was added directly to the flotation cell before adding the collector to leave enough time to react with iron for depressing. Conditioning time was 4 min for collector reagent. The collectors were added directly in the flotation cell after depressant conditioning time.

3.2.6. Reverse Cationic Flotation Experiments

The reverse cationic flotation test was carried out at room temperature using a batch Denver type flotation machine with 1 L cell volume. The impeller speed was 1200 rpm. The solid/liquid ratio was 10% and the pH value was 3 which was adjusted by additional H_2SO_3 (grade >99 % in liquid form) directly to slurry in the flotation cell. Because of the preliminary tests, the results of the experiments performed at pH 3 showed better results than other values. The particle size was -150+38 μm in all the experiments and this particle size was selected based on a preliminary test. Corn starch was used as a depressant which conditioned for 5 min which added to flotation cell after adjusting of pH. The range of corn starch dosages was between 400 to 600 g/ton. The cationic collector was Aero 3030C (2-Ethylhexanol/cationic surfactant in liquid form). The collector was conditioned for 3 min and it was added directly to the flotation cell after depressant was conditioned. The collector range dosages were from 200-400 g/ton. The frother was X-133 (OrePrep X-133 Frother) and was added directly to the slurry 1 min before froth collection time. In preliminary experiments for the same conditions, X-133 frother showed better results than MIBC (methyl isobutyl carbinol) frother. Therefore, it was chosen accordingly. The range of depressant, collector, and frother dosages were selected based on the results of the preliminary experiments. The froth collection time was between 2 and 4 min.

4. RESULTS AND DISCUSSIONS

4.1. Shaking Table Experiments

In this study, the table tilt angle and particle size were considered as the main variable parameters in the shaking table separation method. The effect of the table tilt angle and particle size on the grade and recovery of iron ore concentrate was investigated and the results showed that the table tilt angle and particle size have significant effects on the grade and recovery of the iron ore concentrate. Generally, at different particle sizes, the effect of tilt angles on the grade of iron concentrate was less, while the effect of tilt angles on the recovery of iron concentrate was greater. At -74 μm particle size, the grade of iron ore concentrate was decreased by increasing the tilt angle until the tilt angle of 4° after that, the grade of iron ore concentrate was increased by increasing the tilt angle. At -74 μm particle size and 3° table tilt angle, iron ore concentrate grade was at its highest value of 68.83 %. At -74 μm, iron concentrate recovery was increased by increasing of the tilt angle until the tilt angle of 4 after that iron concentrate recovery was decreased by the increase of the tilt angle due to the water pushing a significant amount of the iron-bearing particles towards the tailing section instead of quartz with increasing table tilts, in contrast, the grade of iron ore concentrate was increased. At this particle size, the maximum of iron concentrate recovery was 32.42 % at the tilt angle of 4°. Fig. 4.1 shows the effect of the table tilt angle on grades and recovery of the iron ore concentrate at a particle size of -75 μm. Fig. 4.2 shows the effect of the table tilt angle on grades and recovery of the iron ore concentrate at a particle size of -150+74 μm. From the Fig. 4.2, at a particle size of -150+74μm, in general, the grade and recovery of iron ore concentrates were decreased with increasing tilt angle until the tilt angle of 5°. Then the grade and recovery of iron ore concentrate were increased with an increasing tilt angle. As is

evident from the Fig.4.2, the effect of the tilt angle on the grade of iron concentrate was less compared to its effect on the recovery of iron ore concentrate.

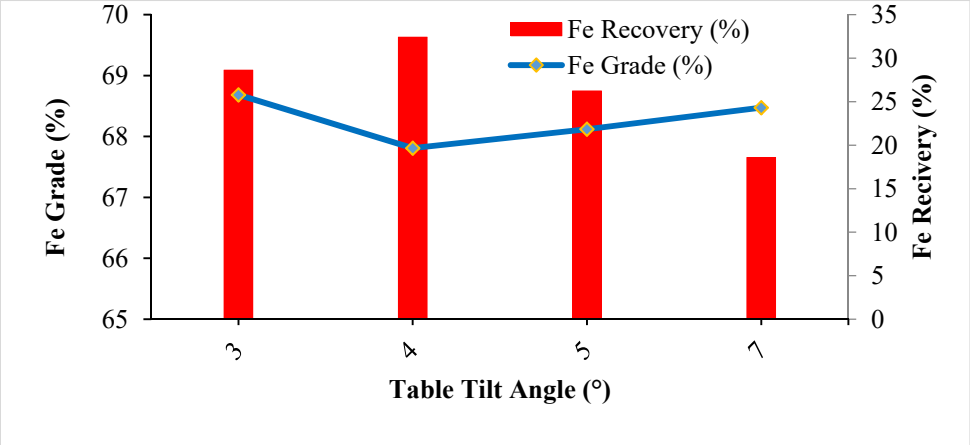

Figure 4.1. Effect of table tilt angles on the grades and recovery of the iron concentrate at -74 µm particle size.

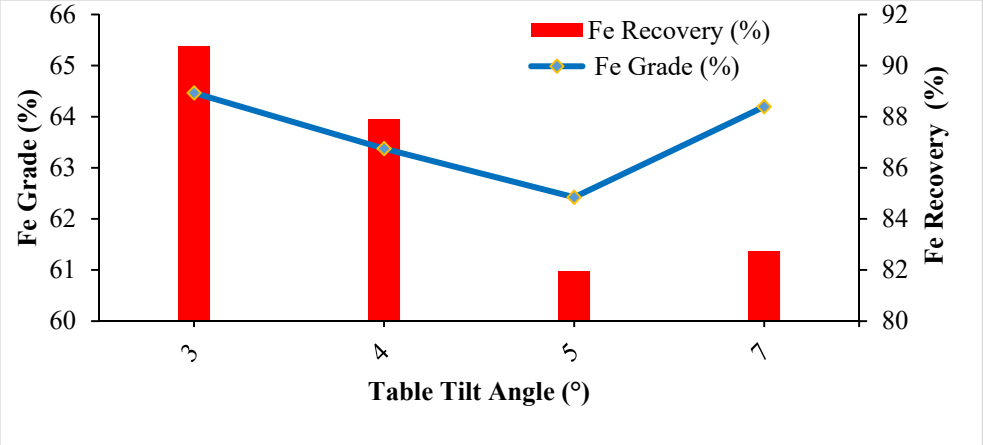

Figure 4.2. Effect of table tilt angles on the grades and recovery of the iron concentrate at -150+74 µm particle size.

When comparing the effect of the table tilt angle on the grade and recovery of iron ore concentrate at the particle sizes -74 µm and -150+74µm, it was noted

that the grades of iron concentrate were higher at a particle size -74µm due to the increase of the degree of liberation as the size fraction decreases, but for the same reason, there was a significant loss of iron-bearing particles in the part devoted to tailings. The liberated iron minerals should, therefore, report to the concentrate product instead of the tailings. But the ore particles below size limits are unable to travel through downstream before a particle at the top of the fluid film settles at the deck surface; therefore, the ore particles below these size limits are invariably lost in the tailings. When the size distribution of ore has a significant number of heavies (iron-bearing mineral particles) below these limit particle size, these heavies are lost during the processing (Roy, 2009). The problem of losing fine valuable minerals during the processing is not only in the shaking table process but in general, a significant amount of fine iron concentrates are lost during the processing in various conventional methods (Surkov et al., 2008). Therefore, at a particle size of -74 µm, recoveries of the iron ore concentrate were low compared to that at a particle size of -150+74µm. At a particle size of -150+74µm, the grades of iron concentrate were lower than that at -74+0µm, but the recoveries of iron concentrate were significant, especially with a decrease of the table of tilt angle. In -150+74µm, the highest iron concentrate grade was 64.47 % Fe at 3° tilt angle, 12 L/min water flow rate and 175 strokes/min shaking speed while the maximum iron ore concentrate recovery of 90,74 % was at the same conditions.

4.1.1. Wet Magnetic Separation Experiments

Parameters affecting the performance of the wet intensity magnetic separator are mostly particle size and magnetic field intensity. The effect of each operating parameters on grade and recovery of iron ore concentrate was discussed in the following subtitles.

4.1.2. Effect of Particle Size on Grade and Recovery of the Iron Concentrate

Wet magnetic separation experiments were carried out to investigate the effect of particle size on the specularite upgrading in wet magnetic separation as shown in Fig. 4.3. The results showed that, at a fixed magnetic field intensity of 0.75 T and a solid/liquid rate of 10% by weight, the particle size had a significant effect on both grade and recovery of iron concentrate. Generally, when the particle size of the specularite sample was reduced, iron ore concentrate grade gradually increased and reached to highest value (67.85 % Fe) at the particle size of 150 µm. Then, iron concentrate began decreasing slightly when the particle size was decreased.

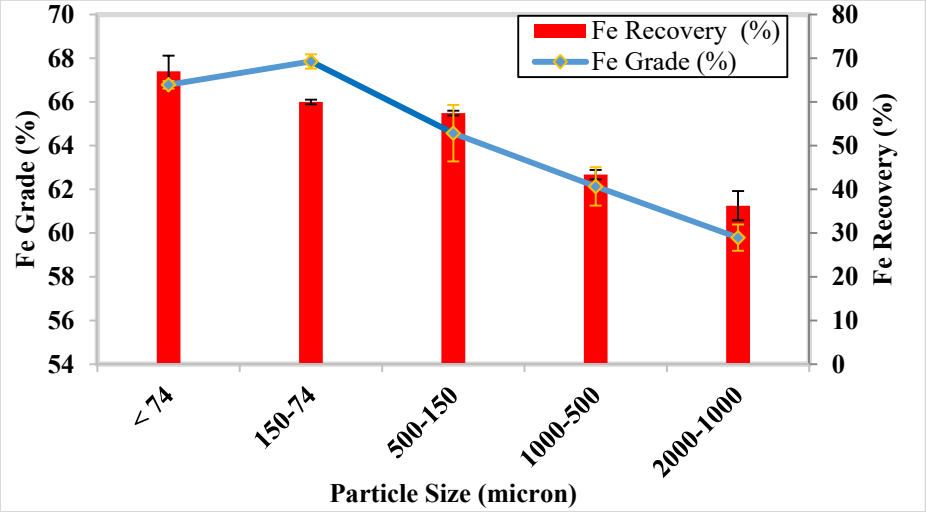

Figure 4.3. Grades and recoveries at different particle sizes.

The particle size of the samples also had a particular effect on the iron ore concentrate recovery as illustrated in Fig.4.3. At particle size of -2000+1000 µm, iron concentrate recovery was at its lowest value as 36.26%. When particle size reduced from 2000µm, iron ore concentrate recovery significantly increased. A maximum recovery (67%) achieved at the particle size of -74µm. In these

experiments, the efficiency of the wet magnetic separator to attract iron-bearing particles in large quantities and selectivity was increased by decreasing the particle size at magnetic field strength 0.75 T. Therefore, both the grade and recovery of the iron concentrate were increased by decreasing the particle size of the sample.

4.1.3. Effect of Magnetic Separation on Grade and Recovery of the Iron Concentrate

Results of wet magnetic separation experiments were seen in Fig. 4.4. After the optimum particle size range was determined as -150+0 µm, wet magnetic separation tests were performed at 0.25 T, 0.35 T, 0.45 T, 0.55 T, 0.65 T, 0.75 T, and 0.85 T magnetic field strengths.

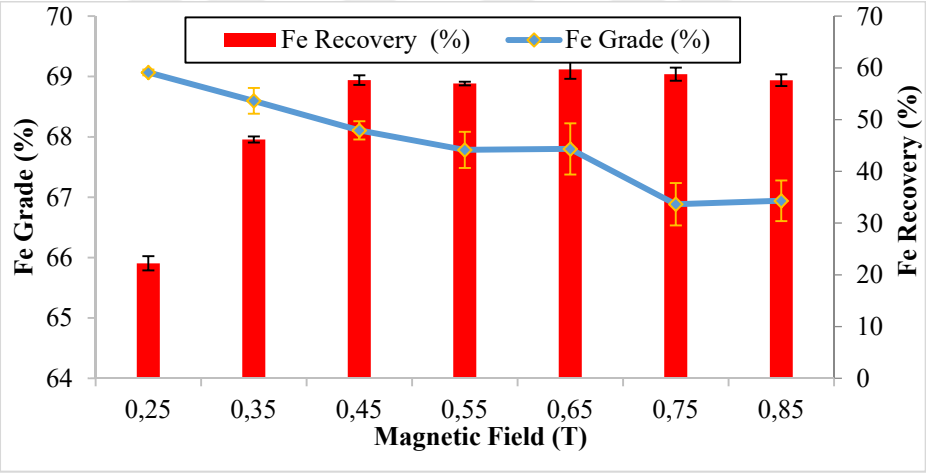

Figure 4.4. Concentrate grades and recovery at different magnetic field intensities.

From Fig. 4.4., it was concluded that there was a significant effect of the magnetic field intensity on both iron ore concentrate grade and recovery by using wet magnetic separation. In general, iron concentrate grades were decreased as the magnetic field intensities increased. When magnetic field intensity was low, the ability of the separator to attract the particles containing iron-quartz reduced. Only

liberated iron-bearing particles were attracted at low magnetic fields and this caused increasing ability of the separator to upgrade the sample but in return, recovery decreased. At a magnetic field intensity of 0.25 T, iron concentrate grade was at its maximum value of 69.07% Fe. After this point, iron ore concentrate grade began to decrease slightly with increasing magnetic field intensity due to the attraction of more quartz-iron bearing (non-liberated) particles. At a magnetic field intensity of 0.55 T, iron concentrate grade was increased so slightly by increasing the magnetic field intensity from 67.30% to 67.80% Fe at a magnetic field intensity of 0.65 T. Iron concentrate grade began to decrease slightly to 66.89 % Fe at a magnetic field intensity of 0.75 T which is considered at the lowest value of the iron ore concentrate grade.

4.2. Roasting Experiments

4.2.1. Preliminary Beneficiation Experiments

Preliminary beneficiation experiments were carried out by using wet magnetic separation to explore the effect of magnetic field intensity on the specularite upgrading in wet magnetic separation as shown in Fig. 4.5. The results showed that, at an optimum particle size of -150+38 μm and a solid/liquid rate of 20% by weight, the magnetic field intensity had a significant effect on both grade and recovery of iron concentrate. Generally, when the magnetic field intensity was reduced, iron concentrate grade gradually increased and reached to the highest value of 68.74 % Fe at the magnetic field of 0.25 T while the iron ore concentrate recovery was increased as magnetic field intensity increased. The highest value of the iron ore concentrate recovery was 76.55 % at the magnetic field intensity of 0.85 T. In this study, rejected product at the highest iron ore concentrate grade of (68.74% Fe) which was obtained at 0.25 T magnetic field, -150+38 μm particle size and 20% solid/ liquid ratio respectively was taken to the roasting process to obtain high grade and recovery of the iron ore concentrate as possible. At this highest grade, the iron concentrate grade of the rejected product was 80.62 %

Fe_2O_3 (56.39% Fe) while the content of the SiO_2 and CaO were 11.2% and 7.6 %, respectively. SEM was used to investigate the surface morphology of rejected product that was taken from the preliminary beneficiation. The SEM images of the rejected product are shown in Fig. 4.6. As shown in fig. 4.6, the micrograph obtained for rejected product particles shows that these particles are irregular in shape and size and they are distributed irregularly with a particle size between -150+38 µm.

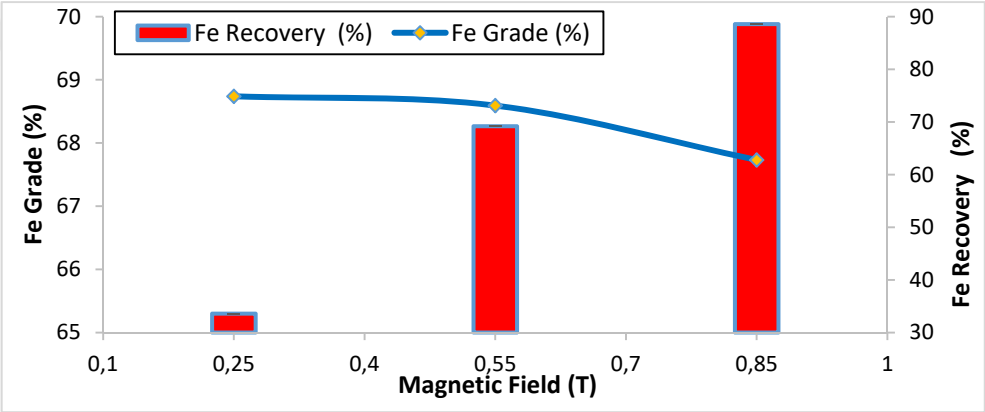

Figure 4.5. Concentrate grades and recoveries at different magnetic field intensities.

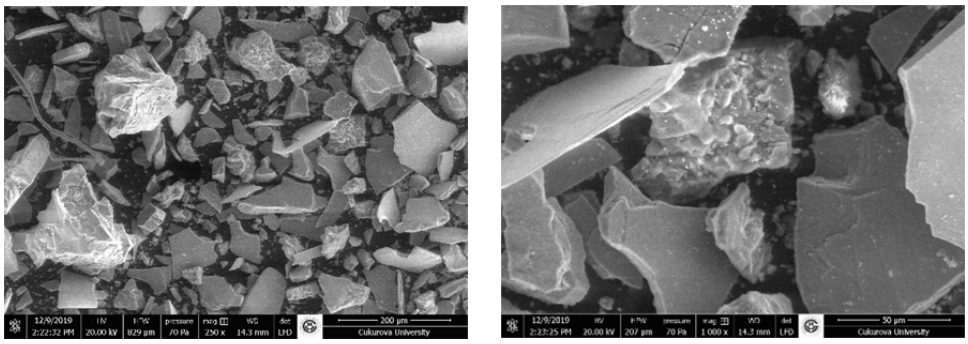

Figure 4.6. SEM images of the rejected product at 0.25 T magnetic field, -150+38 µm particle size and 20% solid/ liquid ratio

4.2.2. Microwave Heating of the Non-Magnetic Product Experiments

Microwave roasting experiments of the non-magnetic product were carried out to investigate the effect on their magnetic properties. The microwave heating process was performed depending on the difference between the absorption properties of the iron-bearing minerals and gangue to microwave in the sample. The non-magnetic product was subjected to microwave power of 0.54, 0.72, and 0.9 KW with an exposure time of 1, 2, and 3 min to increase it is magnetic susceptibility. The effects of microwave roasting and exposure times on the phase properties of a non-magnetic product in the microwave roasting process were characterized by the magnetic hysteresis (M–H) curves. The magnetic hysteresis (M–H) of the original and roasted rejected products for 0.54 KW and 0.9 KW microwave power for 2 min exposure time is shown in Fig. 4.7. The magnetism of the original rejected product, at 0.54 KW microwave power, and at 0.9 KW microwave power (for 10 min exposure time) was measured by PPMS (Physical Property Measurement System). Quantum Design (Model: PPMS DynaCool-9) device was used for this purpose. The microwave heating had a strong effect on the saturation magnetism of the rejected product as shown in the M–H curves. It is seen that the increase in roasting microwave power led to an increase in the saturation magnetism of the rejected product. Table 4.1. Shows the parameters (microwave power, exposure time, and magnetic field intensity) as factors while grade and recovery of the iron ore concentrate as the response in the test of microwave heating followed by magnetic separation for all experiments, which were analyzed in Box-Behnken test design. The effect of each parameter on the grade and recovery iron ore concentrate was discussed in the following headings.

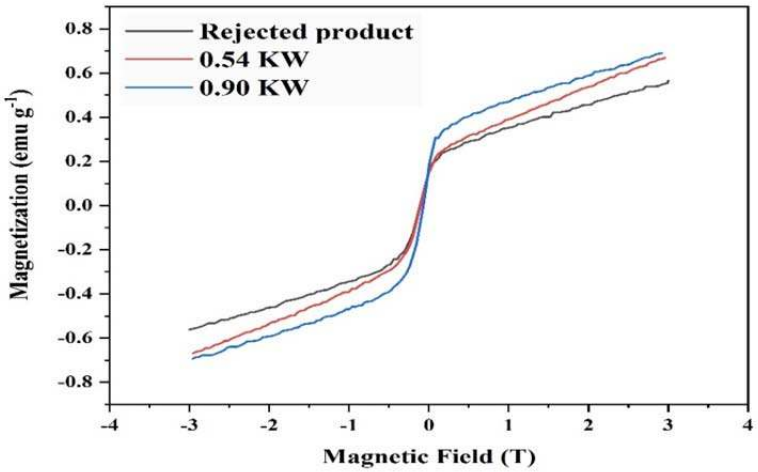

Figure 4.7. M–H curves at different microwave power and exposure time.

Table 4.1. Results of Box-Behnken test design of microwave heating experiments

	A: Microwave power (KW) (X_1)	B: Exposure Time (min) (X_2)	C: Magnetic field intensity (T) (X_3)	Fe Grade (%)	Fe Recovery (%)
1	0.54	2	0.25	66.53	72.27
2	0.54	2	0.85	66.64	80.53
3	0.54	3	0.55	68.07	63.56
4	0.54	1	0.55	66.31	68.50
5	0.9	1	0.55	67.80	65.25
6	0.72	2	0.55	67.56	69.32
7	0.72	2	0.55	67.25	75.18
8	0.72	3	0.25	68.79	33.17
9	0.9	2	0.85	65.79	84.90
10	0.9	3	0.55	66.72	80.54
11	0.9	2	0.25	68.90	28.98
12	0.72	2	0.55	66.71	83.04
13	0.72	3	0.85	66.06	87.37
14	0.72	1	0.85	66.23	77.53
15	0.72	1	0.25	68.89	24.04

4.2.2.1. Effect of Microwave on Grade and Recovery of the Iron Concentrate

The effect of microwave power on iron concentrate ore grade was observed and the results obtained as shown in Fig. 4.8. The results showed that the microwave powers had a significant effect on the iron ore concentrate grade. At 0.54 KW microwave power, the grade of iron ore concentrate was significantly increased with increasing exposure time while it was decreased very slightly with increased intensity of the magnetic field. At this microwave power, the maximum iron content (68.07 % Fe) was obtained at 3 min exposure time and 0.55 T magnetic field.

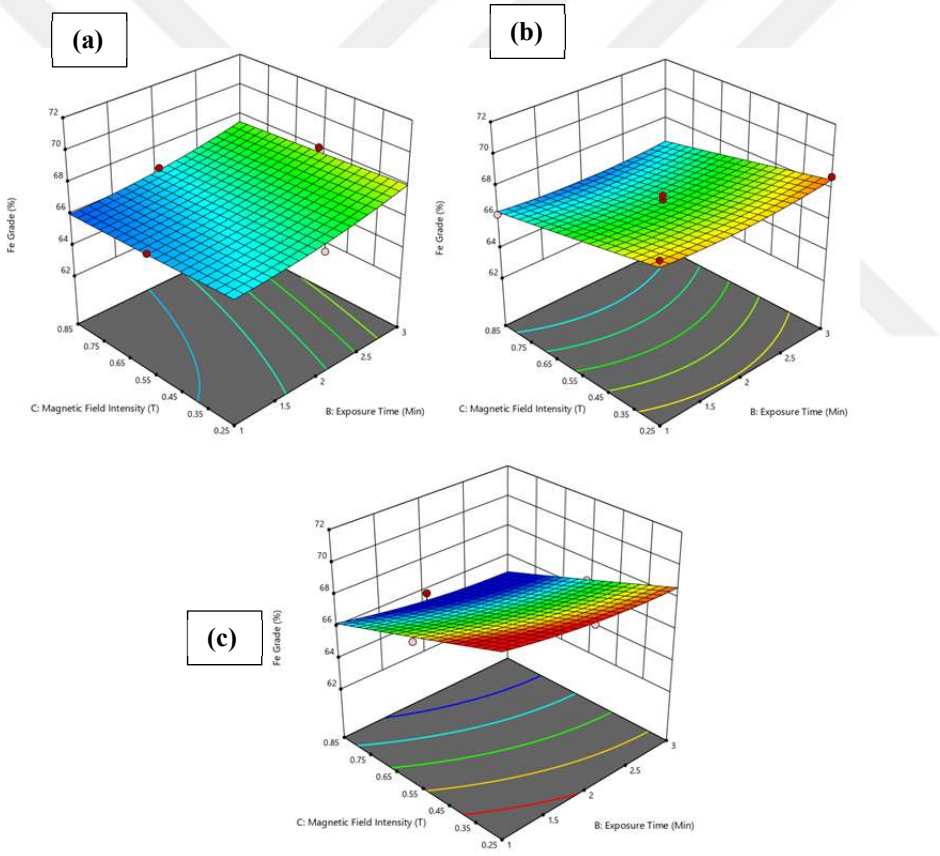

Figure 4.8. Effect of 0.55 KW (a), 0.72 KW (b), and 0.9 KW (c) microwave power on the iron concentrate grade.

When the microwave power of 0.72 KW, the iron concentrate grade was decreased when the magnetic field was increased while exposure time did not affect the grade of the iron concentrate, therefore, the grade was deceased and then increased very slightly with increasing exposure time. Comparing with 0.54 KW microwave power, when microwave was 0.72 KW, the grades of iron concentrate were higher especially at 0.25 T magnetic field intensity and the highest value of iron concentrate grade at this microwave power was 68.90 % Fe. At this point, the exposure time and magnetic fields were 1 min and 0.25 T respectively. When microwave power was 0.9 KW, iron concentrate grade was increased with decreasing exposure time and magnetic field and the grade of iron concentrate reached its highest value of 68.90% Fe. At this point; exposure, time, and magnetic field were at their lowest values of 2 min and 0.25 T respectively. SEM images of iron ore concentrate (68.90% Fe) at 0.9 KW microwave power, 2 min exposure time, and 0.25T Magnetic field intensity in microwave heating test are shown in fig.4.9. As shown in fig. 4.9, the surface micrograph of iron ore concentrate at 68.90% Fe was irregular in size and shape.

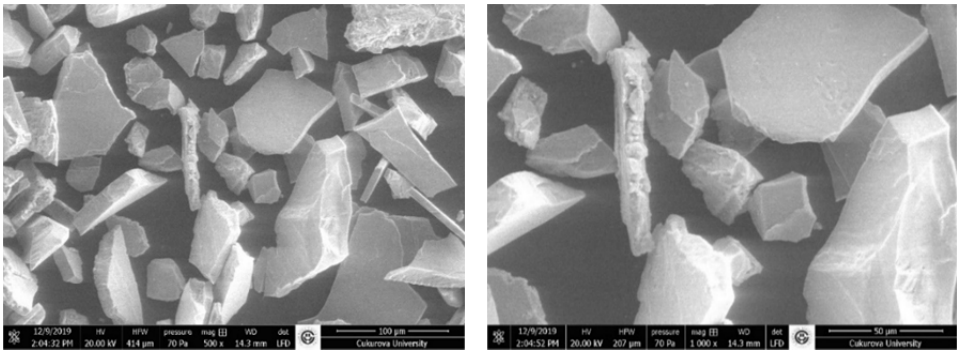

Figure 4.9. SEM images of iron concentrate (68.90% Fe) at 0.9 KW microwave power, 2 min exposure time and 0.25T Magnetic field intensity in microwave heating test

4.2.2.2. Effect of Exposure Time on Grade and Recovery of the Iron Concentrate

The effect of the 1 min, 2 min, and 3 min exposure time on iron ore concentrate grade is shown in Fig. 4.10. The exposure time had a significant effect on iron concentrate grade. Therefore, generally, at 1 min exposure time, the grade of iron concentrate was increased with increasing microwave power and decreased magnetic field intensity.

When exposure time was 2 min, the effect of the microwave power and magnetic field on the iron concentrate grade remained the same as that at 1 min exposure time expect at magnetic fields over 0.65 T, the general effect of exposure time on iron concentrate grade was negative and the iron concentrate grade was decreased with increasing microwave power. At 2 min exposure time, the grade of iron concentrate was at the highest value of 68.90% Fe. At this point, both microwave power and magnetic fields were 0.9 KW and 0.25 T respectively.

At 3 min exposure time, the iron concentrate grade was slightly increased with increasing microwave power and decreasing the magnetic field. Generally, at all exposure times, the highest grades of the iron concentrate were observed when the magnetic field was 0.25 T.

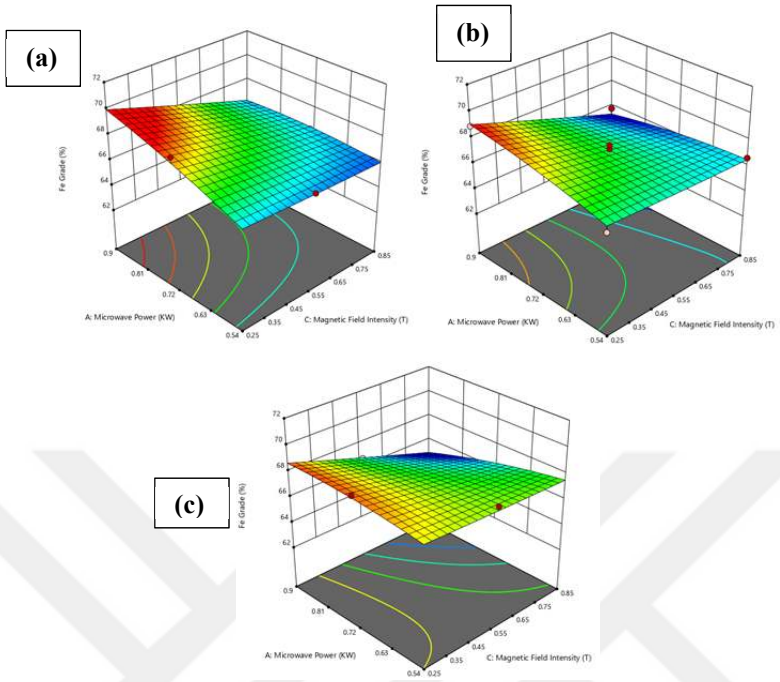

Figure 4.10. Effect of 1 min (a), 2 min (b), and 3 min (c) exposure times on iron concentrate grade.

The effect of exposure times on the iron concentrate recovery is indicated in Fig. 4.11. The results showed that, at 1 min exposure time, the recovery of iron concentrate was increased with increasing microwave power and magnetic field intensity except when the magnetic field intensities were at the lowest values the iron concentrate recovery was decreased with increasing microwave power. At 2 min exposure time, the effect microwave power and magnetic field intensity remain the same that at 1 min, and at this time the general effect on recoveries of iron concentrate was more positively compared to other exposure time as well as the recoveries were observed high values. When exposure time was 3 min, the maximum iron concentrate recovery was obtained with the 0.72 KW microwave power and 0.85 T magnetic field intensity.

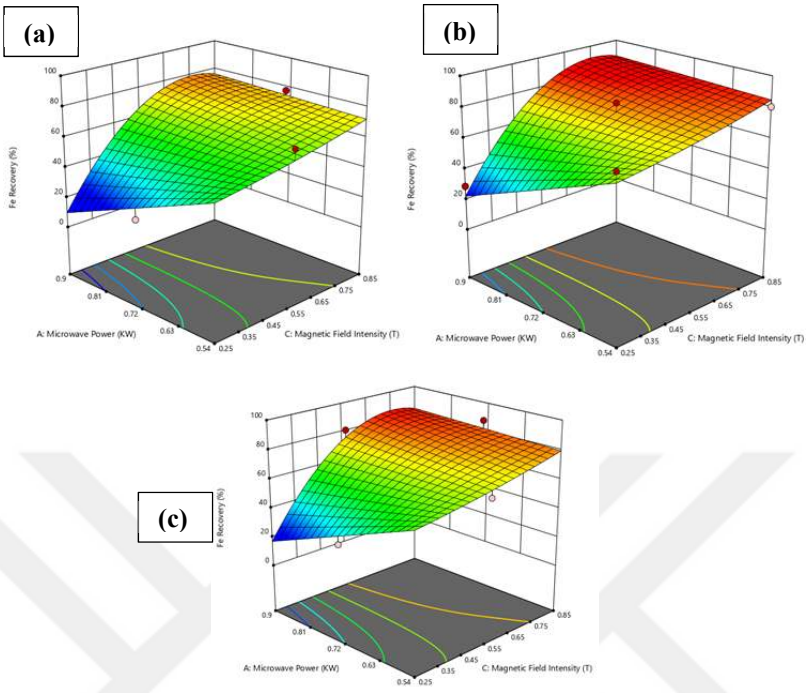

Figure 4.11. Effect of 1 min (a), 2 min (b), and 3 min (c) exposure times on iron concentrate recovery.

4.2.2.3. Effect of Magnetic Field Intensity on Grade and Recovery of the Iron Concentrate

The effect of the magnetic field intensity on the iron concentrate grade was remarkable. The magnetic field intensity had the greatest effect on the iron concentrate grade as shown in Fig. 4.12. When the magnetic field intensity was 0.25 T where 0.9 KW microwave power and 2 min exposure time were applied, iron concentrate grade reached 68.90% Fe. This is because of the magnetic separation method. At 0.25T magnetic field intensity, the concentrator only attracts iron concentrates, while the non-magnetic gangues go with the water as a tailing to the bottom of the concentrator stream. At 0.55 T and 0.85 T magnetic field intensities, the general effect of the microwave power and exposure time almost the same as that at 0.25 T but the grades of iron concentrate that were decreased with magnetic field intensities increased due to the attraction of more iron-quartz

content. The lowest values of the iron concentrate grades were at 0.85 T magnetic field intensity.

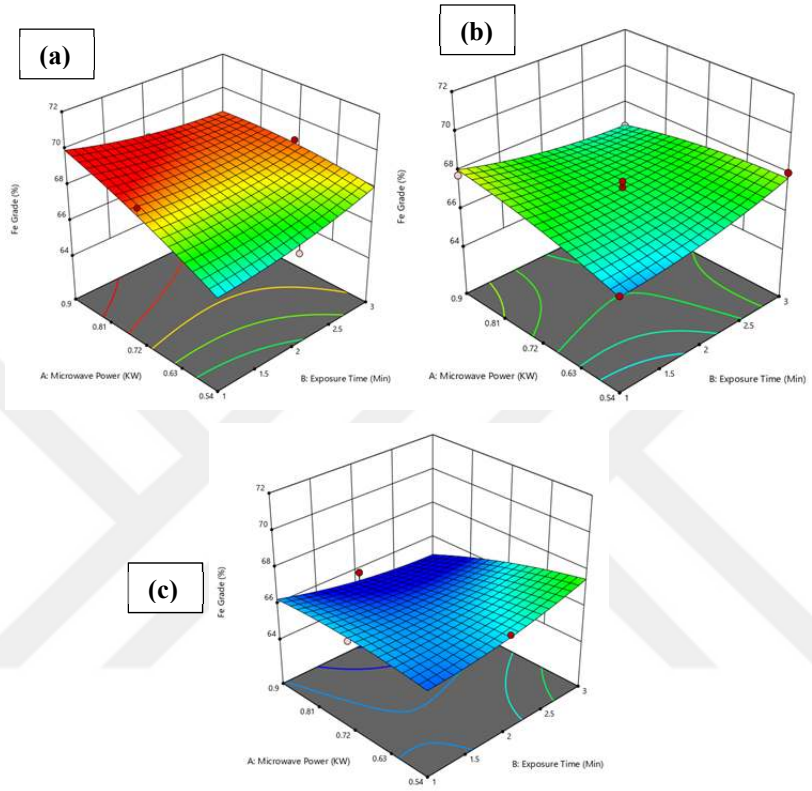

Figure 4.12. Effect of 0.25 T (a), 0.55 T (b), and 0.85 T (c) magnetic field intensity on iron concentrate grade.

The effect of the 0.25 T, 0.55 T, and 0.85 T magnetic field intensity on the iron concentrate grade shown in Fig. 4.13. The magnetic field intensity had the greatest effect on the iron concentrate recovery. The highest values of iron concentrate recoveries were obtained when 0.85 T magnetic field intensity at this field intensity, recoveries of iron concentrate were > 75 % and reached their highest value of 87.37% at 0.72 KW microwave power and 3 min exposure time. SEM images of iron ore concentrate (82.23% recovery) at 0.72 KW microwave power, 3 min exposure time, and 0.85 T magnetic field intensity in the microwave

heating test are shown in Fig. 4.14. As shown in fig. 4.14, the surface micrograph of iron ore concentrate at 82.23% Fe recovery was irregular in size and shape.

In the microwave heating test, the values of iron concentrate grade and recovery were 66.06 % Fe and 82.23 % respectively at optimum conditions of 0.72 KW microwave power, 3 min exposure time, 0.25 T magnetic field intensity, -150+38 µm and 20 % solid/liquid ratio**.**

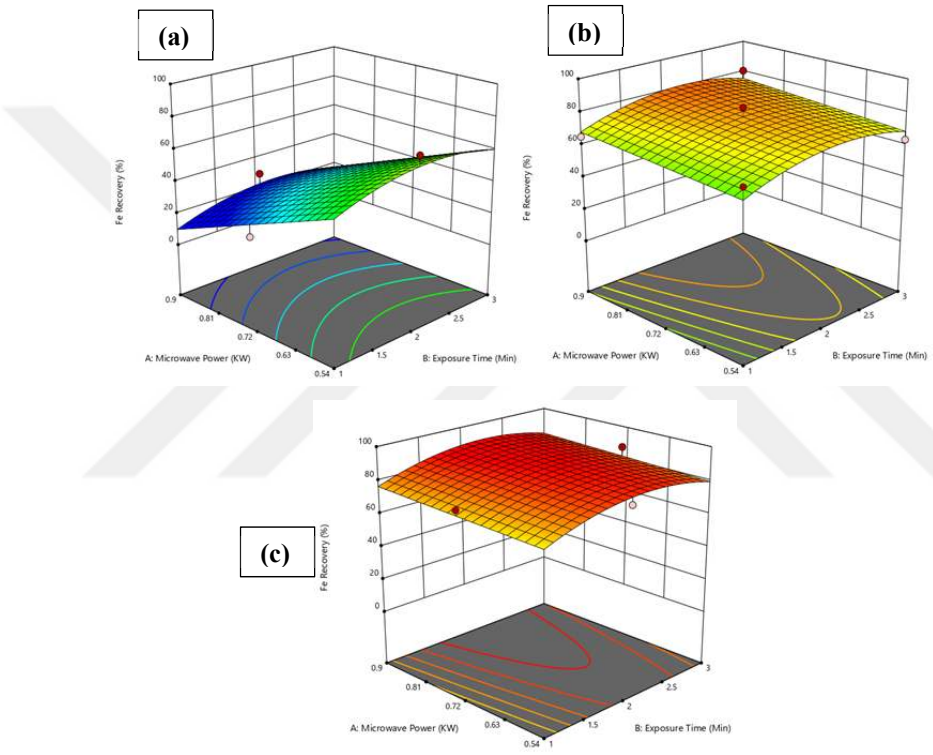

Figure 4.13**.** Effect of 0.25 T (a), 0.55 T (b), and 0.85 T (c) magnetic field intensity on the iron concentrate recovery.

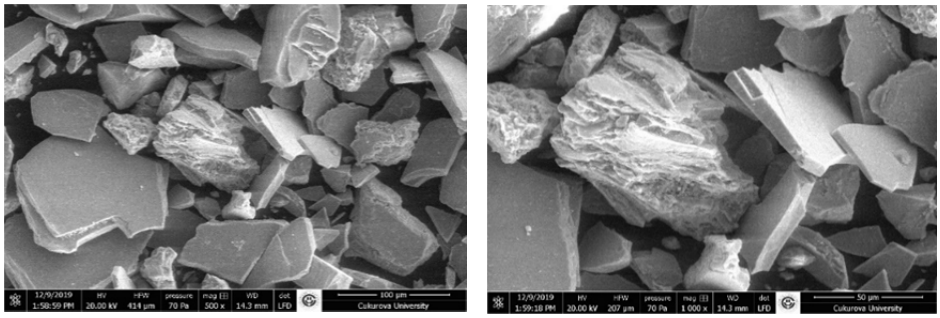

Figure 4.14. Iron concentrate (87.37% recovery) at 0.72 KW microwave power, 3 min exposure time, and 0.85 T magnetic field intensity in microwave roasting test.

Table 4.2. ANOVA for Quadratic model (Grade)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	14.34	9	1.59	5.55	0.0368	significant
X_1-Microwave power	0.3444	1	0.3444	1.20	0.3232	
X_2-Exposure Time	0.0210	1	0.0210	0.0732	0.7975	
X_3-Magnetic field intensity	8.80	1	8.80	30.66	0.0026	
X_1 X_2	2.02	1	2.02	7.03	0.0454	
X_1 X_3	2.59	1	2.59	9.03	0.0299	
X_2 X_3	0.0012	1	0.0012	0.0043	0.9504	
X_1^2	0.2090	1	0.2090	0.7283	0.4324	
X_2^2	0.3096	1	0.3096	1.08	0.3465	
X_3^3	0.0032	1	0.0032	0.0113	0.9196	
Residual	1.43	5	0.2870			
Lack of Fit	1.06	3	0.3550	1.92	0.3607	not significant
Pure Error	0.3701	2	0.1850			
Cor Total	15.77	14				

Table 4.3. Fit Statistics (Grade)

Std. Dev.	0.5357	**R²**	0.9090
Mean	67.22	**Adjusted R²**	0.7453
C.V. %	0.7970	**Predicted R²**	-0.1330
		Adeq Precision	8.4761

According to the experimental results, both of the microwave power, exposure time, and magnetic field intensity had a significant effect of the grade and recovery of the iron concentrate. The magnetic field intensity had the greatest effect on grade of the iron concentrate followed by the microwave power while the effect of exposure time on grade of the iron concentrate was the lowest. In the terms of the recovery, magnetic field intensity had the greatest effect on recovery of the iron concentrate followed by the exposure time while the effect of the microwave power on recovery of the iron concentrate was the lowest.

ANOVA for Quadratic and Reduced Cubic models was applied to iron concentrate grade and recovery recoveries respectively and was analyzed statistically. The correlation coefficients (R^2) for iron concentrate grade (Tables 4.2 and 4.3) and recovery were calculated as 0.91 (with 0.0368 P-value) and 0.92(with 0.0020 P-value) respectively. It was shown as a perfect correlation for parameters. The equations 2 and 3 show the created models: Where; X_1: microwave power (KW), X_2: exposure time (min), and X_3: magnetic field intensity (T).

$$Fe_2O_3 \text{ grade} = 67.17 + 0.2075\ X_1 + 0.0512\ X_2 - 1.05\ X_3 - 0.71\ X_1X_2 - 0.805\ X_1X_3 - 0.0175\ X_2X_3 - 0.2379\ X_1^2 + 0.2896\ X_2^2 + 0.0296\ X_3^2 \quad (2)$$

$$Fe_2O_3 \text{ recovery} = 77.31 + 3.43\ X_1 + 3.66\ X_2 + 2148\ X_3 + 11.91\ X_1X_3 - 8.95\ X_2^2 - 11.74\ X_3^2 - 13.61\ X_1X_3^2 \quad (3)$$

4.2.3. Roasting of the Rejected Product Experiments

Magnetic roasting involves controlled roasting of the ore to magnetic roasting, which includes controlled roasting of the ore to transform it into a magnetic phase, allowing for easy separation and processing in the subsequent stages. Heat treatment of other iron-bearing minerals (e.g. siderite and hematite) improved their magnetization (wills et al., 2015). In this test, roasting experiments of the non-magnetic product were carried out to investigate it affects their magnetic properties. The non-magnetic product was blended with 10% of high-quality coal that was used as an accelerator to increase the reduction rate of the iron ores. The magnetism of the original rejected product, at 450 °C and 650 °C temperatures for 10 min exposure time was measured by PPMS (Physical Property Measurement System). Quantum Design (Model: PPMS DynaCool-9) device was used for this. The effects of the roasting temperatures and exposure times on magnetic susceptibility of the rejected products were characterized by the magnetic hysteresis (M–H) curves. The magnetic hysteresis (M–H) of the original and roasted non-magnetic products at 450 °C and 650 °C temperatures for 10 min exposure time is shown in Fig. 4.15.

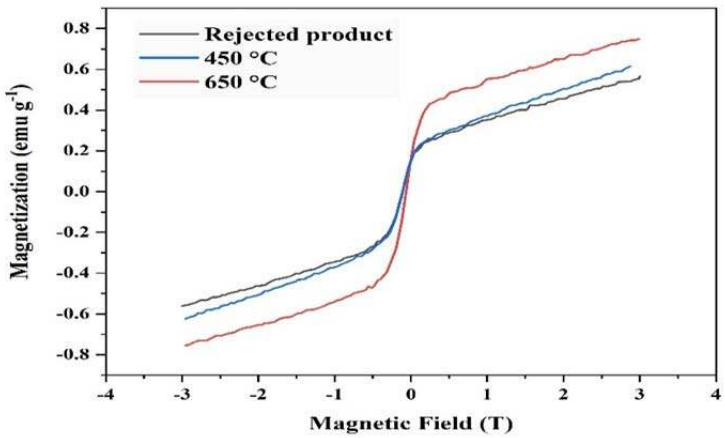

Figure 4.15. M–H curves at different temperatures for 10 min exposure time.

As seen in the M – H curves, the roasting temperature had a significant influence on the saturation magnetism of rejected products. The rise in roasting temperature has increased the saturation magnetism of rejected products. The effect of three parameters including temperature, exposure time, and magnetic field intensity on the grades and recoveries of iron concentrate was investigated in Table 4.4.

Table 4.4. Results of Box-Behnken test design of roasting experiments

	A: Temperature (°C) (X_1)	**B: Time (min) (X_2)**	**C: Magnetic intensity (T) (X_3)**	**Fe Grade (%)**	**Fe Recovery (%)**
1	550	15	0.85	64.47	82.72
2	450	10	0.85	67.73	77.56
3	650	15	0.55	61.24	84.47
4	550	10	0.55	67.66	72.34
5	650	10	0.85	62.26	85.95
6	650	10	0.25	65.28	57.35
7	450	10	0.25	69.10	41.16
8	650	5	0.55	62.44	78.97
9	550	5	0.25	68.25	39.38
10	550	15	0.25	69.02	35.82
11	550	10	0.55	66.39	63.27
12	450	15	0.55	68.47	58.71
13	550	10	0.55	67.18	60.48
14	550	5	0.85	65.32	70.27
15	450	5	0.55	67.93	58.02

4.2.3.1. Effect of Temperature on Grade and Recovery of the Iron Concentrate

Fig. 4.16. shows the effect of the temperature on iron concentrate grade at 450 °C, 550 °C, and 650 °C, respectively. At 450°C temperature, iron concentrate grade was increased with increasing exposure time while it was decreased slightly as the magnetic field increased. At 550 °C temperature, generally, the effect of exposure time and magnetic field on iron concentrate grade were same as that at 450 °C but comparing with 450 °C temperature, the iron concentrate grades were less due to negative effect of temperature on the iron concentrate grade over 500 C due to start of the melting of iron concentrates with gangues such as quartz and calcium carbonate after this temperature. At 650 °C temperature, generally, the effect of exposure time and magnetic field on iron concentrate grade remained the same at 450 °C and 550 °C temperatures, as well as the negative effect of the temperature, was increased

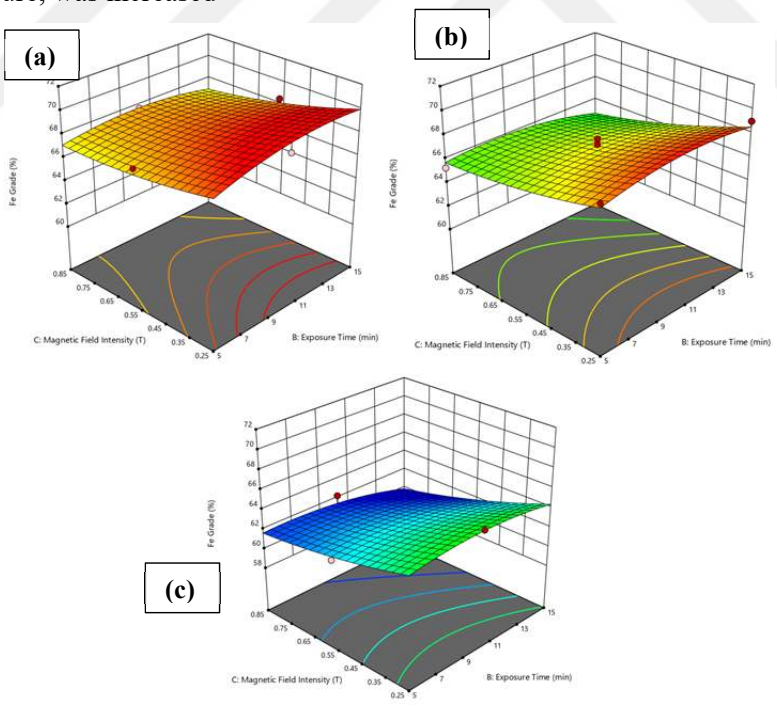

Figure 4.16. Effect of 450 °C (a), 550 °C (b), and 650 °C (c) on iron grade%

The effect of temperatures on iron concentrate recovery was observed and the results obtained as shown in Fig. 4.17. at temperatures of 450 °C, 550 °C, and 650 °C, respectively. Generally, the effect of temperature on the iron concentrate recoveries was positive and somewhat stable. However, generally, at all temperatures, the iron concentrate recovery was increased with increasing magnetic field intensity while there was no noticeable effect of exposure times at different temperatures except for the field intensity over 0.55 T there was a positive effect as the recoveries of the iron concentrate were increased with increasing exposure time. The highest value of the iron concentrate recovery was (85.95 %) at 650 °C temperature. At this point, magnetic field intensity and exposure time were 0.85 T and 10 min, respectively. The SEM of iron ore concentrate (85.95% Fe recovery) at 650 °C temperature, 0.85T magnetic field intensity, and 10 min exposure time is shown in Fig. 4.18. As shown in fig. 4.18, the surface micrograph of iron ore concentrate at 85.95% Fe recovery was irregular in size and shape.

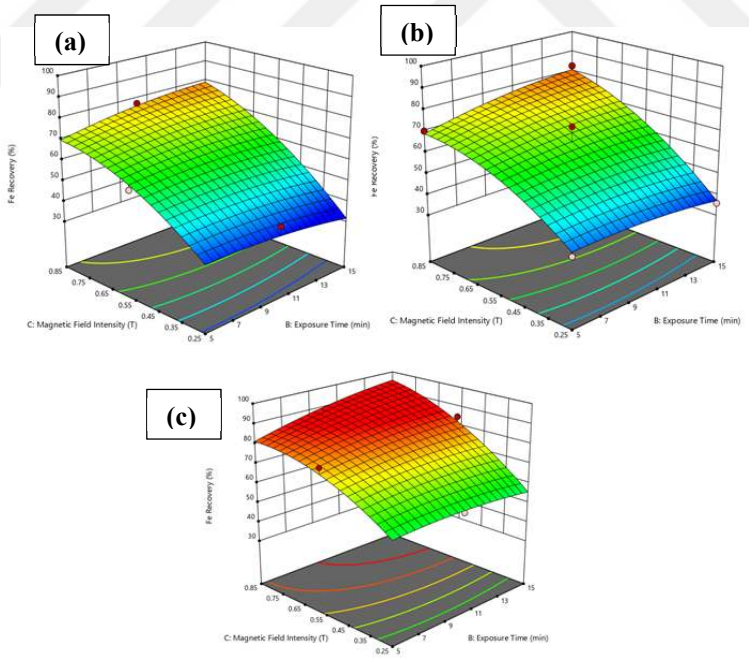

Figure 4.17. Effect of 450 °C (a), 550 °C (b), and 650 °C (c) magnetic field intensity on iron concentrate recovery.

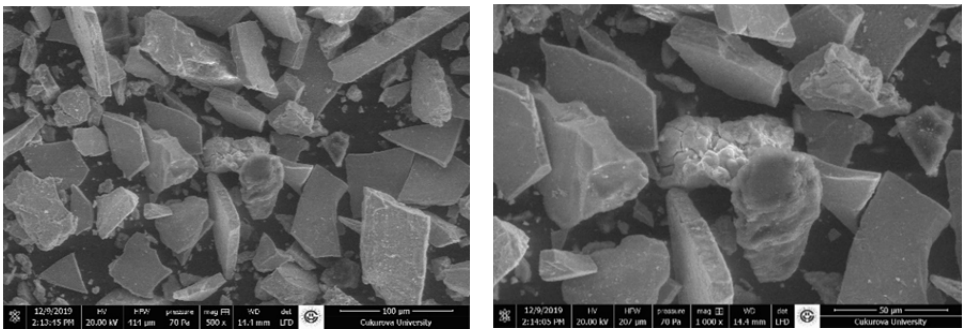

Figure 4.18. Iron ore concentrate (85.95 % recovery) at 650 °C temperature, 10 min exposure time, and 0.85 T in roasting tests.

4.2.3.2. Effect of Exposure Time on Iron Concentrate Grade and Recovery

The effect of the exposure time for 5 min, 10 min, and 15 min on the iron concentrate grade is shown in Fig. 4.19. Generally, exposure time had a significant effect on the iron concentrate grade and was positive especially at lower values of temperature and magnetic field intensity. Therefore, at 5 min exposure time, the grade of the iron concentrate was increased with decreasing temperature and magnetic field intensity. At exposure times of 10 min and 15 min, the general effect on the iron concentrate grades was the same as that at 5 min, therefore, grades of the iron concentrate were increased as temperature and magnetic field intensities were decreased. Compared to 5 min exposure time, the grades of the iron concentrate at 10 min exposure time were higher and reached their highest value (69.10 % Fe). At this point, temperature and magnetic field intensities were 450 °C and 0.25 T, respectively. SEM images of iron ore concentrate (69.10% Fe) at 450 °C temperature, 10 min exposure time and 0.25 T magnetic field intensity in the roasting test are shown in Fig. 4.20. As shown in fig. 4.20, the surface micrograph of iron ore concentrate at 69.10% Fe grade was irregular in size and shape.

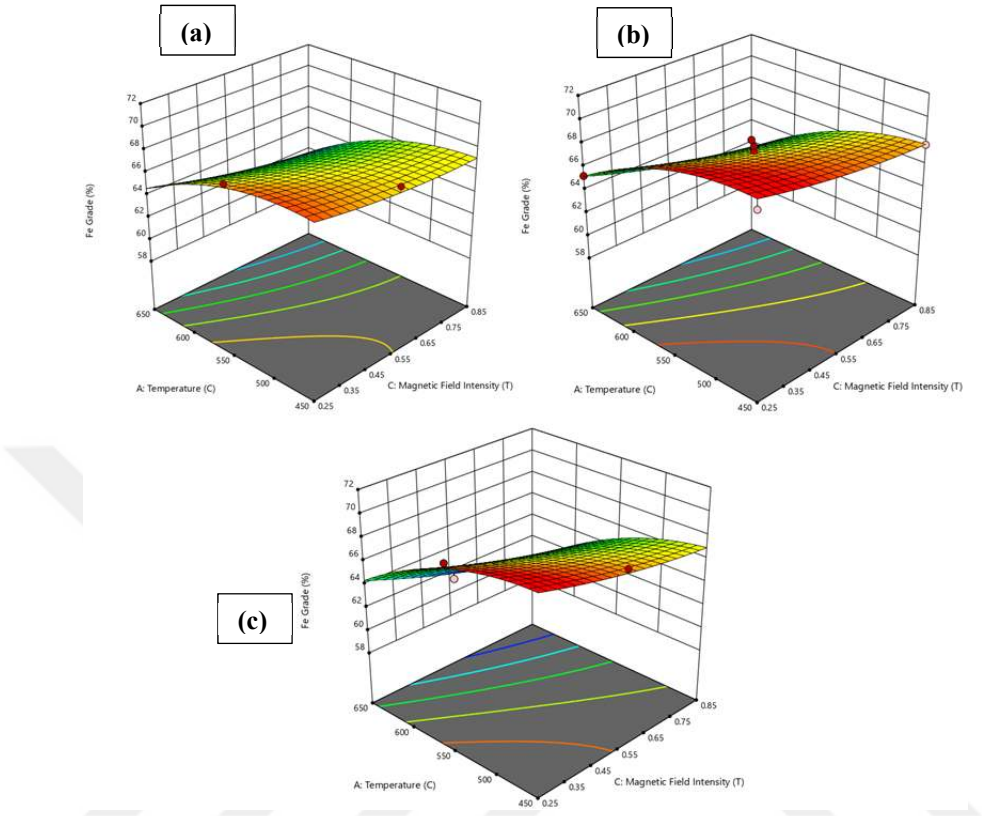

Figure 4.19. Effect of 5 min (a), 10 min (b), and 15 min (c) on iron grade.

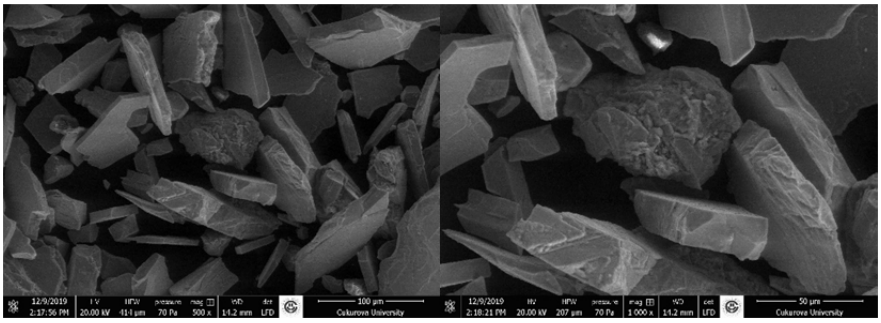

Figure 4.20. Iron ore concentrate (69.10% Fe grade) at 450 °C temperature, 10 min exposure time, and 0.25 T magnetic field intensity in roasting tests.

The effect of the exposure time for 5 min, 10 min, and 15 min on the iron concentrate recovery shown in Fig. 4.21. At 5, 10 min and 15 min exposure time, the iron concentrate recoveries were increased with increasing temperatures and magnetic field intensities. At 10 min exposure time, the iron concentrate recovery reached its highest value of 85.95 % at 650 °C temperature and 0.85 T magnetic field intensity.

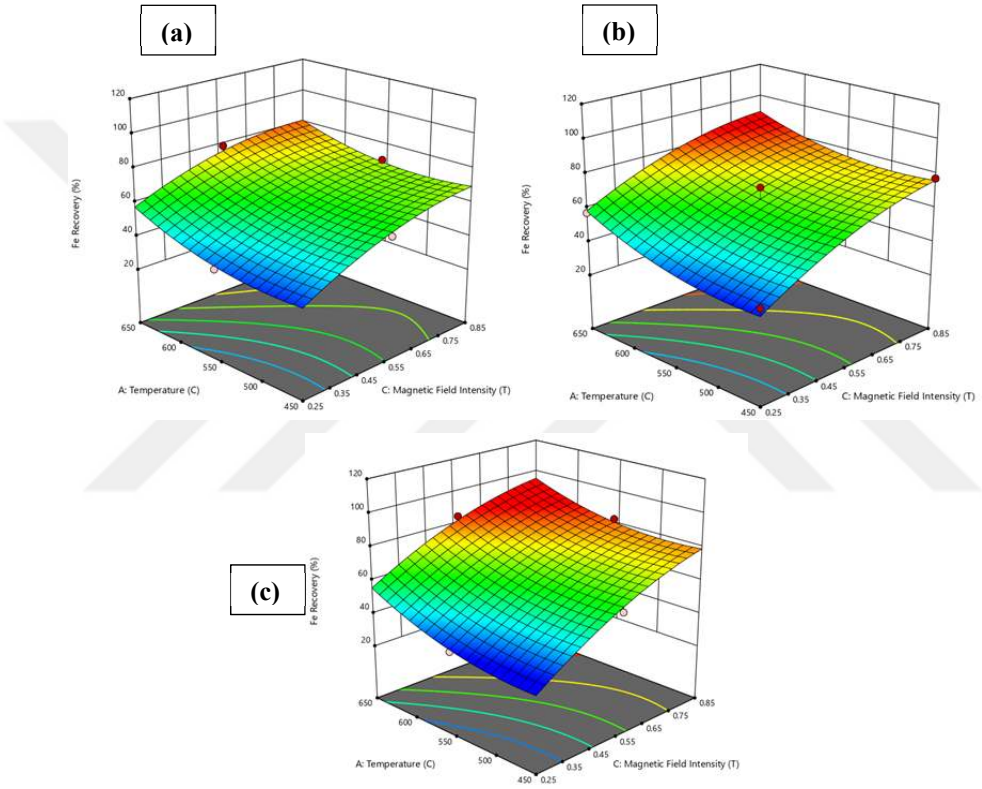

Figure 4.21. Effect of 5 min (a), 10 min (b), and 15 min (c) on iron concentrate recovery.

4.2.3.3. Effect of Magnetic Intensity on the Iron Grade and Recovery

Fig. 4.22. Showing the effect of 0.25 T, 0.55 T, and 0.85 T magnetic field intensities on the iron concentrate grade. The magnetic field intensities had a significant effect on the iron concentrate grade and compared to temperature and

exposure time, magnetic field intensities had the greatest effect on the grades of the iron concentrate. Generally, iron concentrate grades were significantly increased with decreasing magnetic field intensities. At different magnetic field intensities, iron concentrate grade was decreased with increasing temperature while the effect of exposure time on the grade of iron concentrate was not significant.

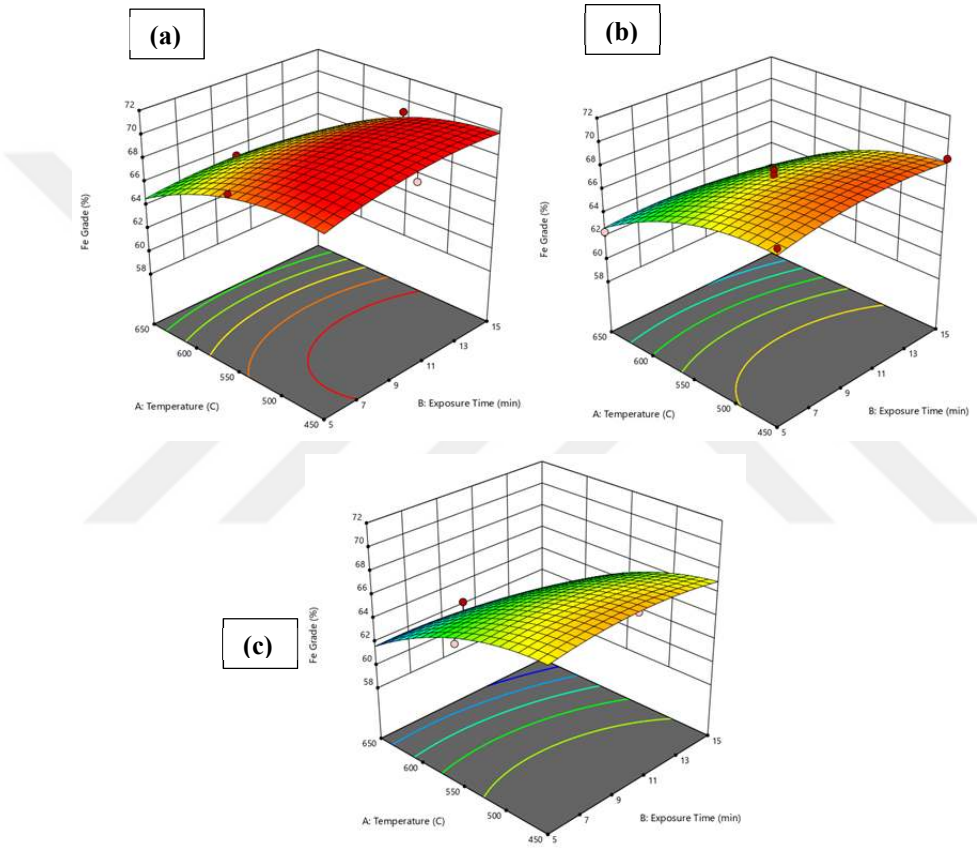

Figure 4.22. Effect of 0.25 T (a), 0.55 T (b), and 0.85 T (c) magnetic field intensity on iron concentrate grade.

From Fig. 4.23., it was concluded that there was a significant effect of the magnetic field intensity on iron concentrate recovery. Compared to temperature and exposure time, the effect of the magnetic field intensity on iron concentrate recovery was the greatest. In general, iron concentrate recoveries increased as the

magnetic field intensities increased. When magnetic field intensities were 0.25 T, 0.55 T, and 0.85 T, the iron concentrate recoveries were increased with increasing temperatures while the effect of exposure time on the iron concentrate recoveries was not significant except at magnetic field intensity over at 0.55 T and temperature of 600 °C, the effect of the exposure time on the recoveries of the iron concentrate were positive, iron concentrate recoveries were increased with increasing of exposure times.

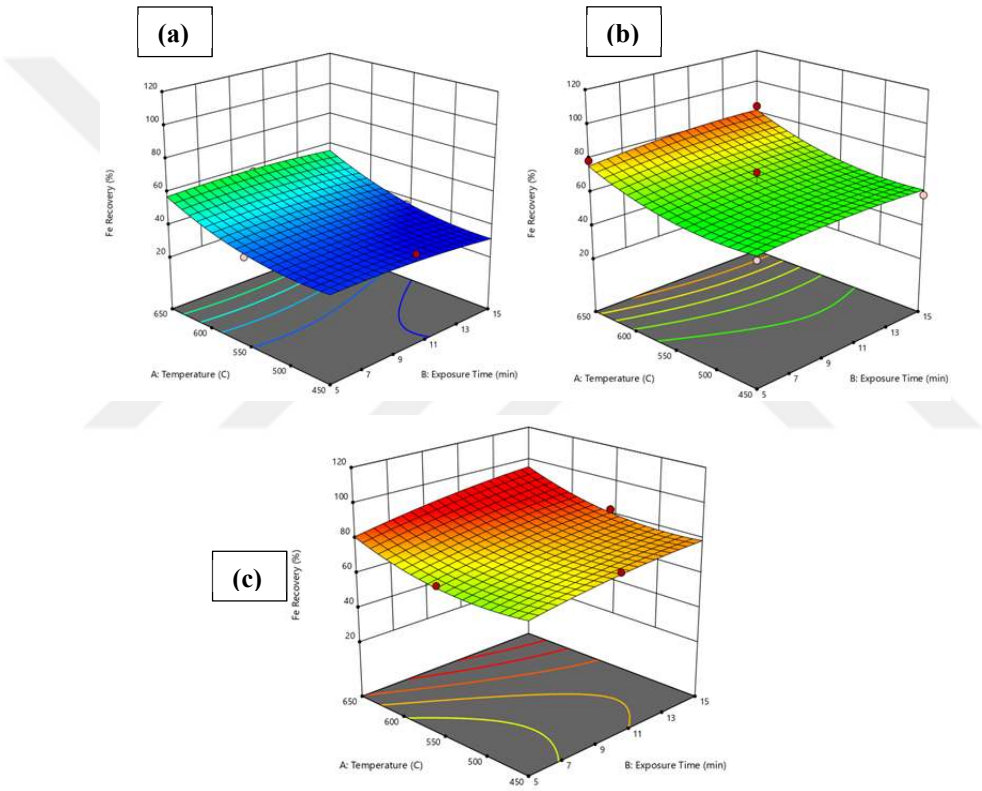

Figure 4.23. Effect of 0.25 T (a), 0.55 T (b), and 0.85 T (c) magnetic field intensity on iron concentrate recovery.

In the roasting test, according to both grade and recovery of iron concentrate, the optimum conditions were the flowed: 450 C temperatures, 10 min exposure time, 0.85 T magnetic field intensity, -150+38 μm, and 20 % solid/liquid

ratio. At these conditions, grade and recovery of the iron concentrate were 67.73 % Fe and 77.56 % respectively. The SEM images of iron concentrate at an optimum conditions 450 °C temperature, 10 min exposure time, 0.85 T magnetic field intensity, -150+38 μm, and 20 % solid/liquid ratio are shown in Fig. 4.24. As shown in fig. 4.24, the surface micrograph of iron ore concentrate at optimum conditions was irregular in size and shape.

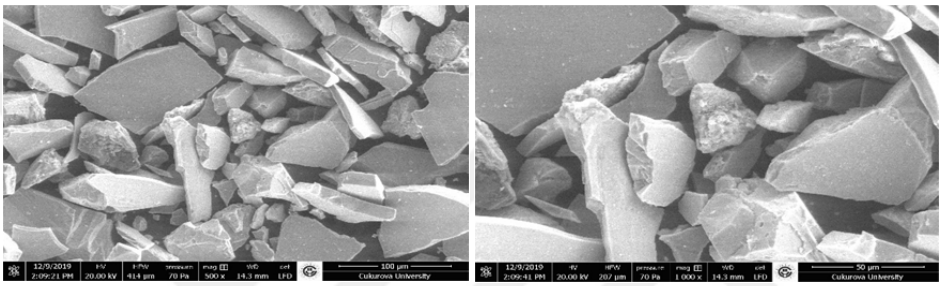

Figure 4.24. Iron concentrate at 450 °C temperature, 10 min exposure time, and 0.85 T magnetic field intensity in roasting tests.

According to the experimental results, both of the temperature, exposure time, and magnetic field intensity had a significant effect of the grade and recovery of the iron concentrate. The magnetic field intensity had the greatest effect on grade and recovery of the iron concentrate followed by the temperature while exposure time had the lowest effect on grade and recovery of the iron concentrate.

ANOVA for the Quadratic model was applied and statistically analyzed to iron concentrate grade and recovery recoveries respectively. The correlation coefficients (R^2) for grade and recovery of the iron concentrate were 0.963 (with 0.0047 P-value) (Tables 4.5 and 4.6) and 0.958 (with 0.006 P-value) respectively As is evident from the values of R^2 and P-value, there is a perfect correlation between the parameters used in the test .Equations 4 and 5 show the created models:

Fe grade= $67.08 - 2.75\ X_1 - 0.0925\ X_2 - 1.48\ X_3 - 0.4350\ X_1X_2 - 4125\ X_1X_3 - 0.405\ X_2X_3 - 1.36\ X_1^2 - 0.621\ X_2^2 + 0.3804\ X_3^2$. (4)

Fe recovery = $65.65 + 8.97\ X_1 + 1.83\ X_2 + 17.85\ X_3 + 1.32\ X_1X_2 - 1.95\ X_1X_3 + 4\ X_2X_3 + 6.37\ X_1^2 - 2.09\ X_2^2 - 6.51\ X_3^2$. (5)

Where; X_1: temperature(C), X_2: exposure time (min) and X_3: magnetic intensity (T)

Table 4.5. ANOVA for Quadratic model (Grade).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	89.52	9	9.95	14.09	0.0047	significant
X_1-Temperature	60.56	1	60.56	85.80	0.0002	
X_2-Exposure time	0.0684	1	0.0684	0.0970	0.7680	
X_3-Magnetic strength field	17.61	1	17.61	24.96	0.0041	
$X_1\ X_2$	0.7569	1	0.7569	1.07	0.3478	
$X_1\ X_3$	0.6806	1	0.6806	0.9644	0.3712	
$X_1\ X_3$	0.6561	1	0.6561	0.9297	0.3792	
X_1^2	6.88	1	6.88	9.74	0.0262	
X_2^3	1.77	1	1.77	2.51	0.1743	
X_3^3	0.5343	1	0.5343	0.7571	0.4240	
Residual	3.53	5	0.7057			
Lack of Fit	2.71	3	0.9021	2.19	0.3284	not significant
Pure Error	0.8225	2	0.4112			
Cor Total	93.05	14				

Table 4.6. Fit Statistics (Grade)

Std. Dev.	0.8401	**R^2**	0.9621
Mean	66.18	**Adjusted R^2**	0.8938
C.V. %	1.27	**Predicted R^2**	0.5148
		Adeq Precision	12.3484

4.3. Falcon Concentrator Experiments

The upgrading of specularite ore with Falcon concentrator was carried out and the effect of effective parameters on grade and recovery of the iron concentrate was investigated. Effect each parameter on grade and recovery of iron concentrate was discussed in the following subtitles. Table 4.7. results of Falcon concentrator experiments, which were analyzed in the Box-Behnken test design.

Table 4.7. Results of Box-Behnken test design of Falcon concentrator experiments

	A: Pressure Psi (X_1)	**B: Frequency Hz (X_2)**	**C: Particle Size µm (X_3)**	**Fe Grade (%)**	**Fe Recovery (%)**
1	30	45.165	250	58.77	99.91
2	40	35	250	59.10	91.81
3	30	35	75	63.93	68.06
4	30	55.33	425	59.54	99.69
5	30	45.165	250	58.70	99.75
6	20	55.33	250	58.35	99.93
7	20	35	250	58.57	99.49
8	20	45.165	75	60.30	82.29
9	40	45.165	75	62.56	78.95
10	40	55.33	250	58.35	99.16
11	30	55.33	75	62.28	81.34
12	20	45.165	425	59.39	99.69
13	40	45.165	425	59.34	99.92
14	30	45.165	250	58.37	99.14
15	30	35	425	60.21	99.76

4.3.1. Effect of Pressure on Grade and Recovery of the Iron Concentrate

Falcon concentrator experiments were carried out to investigate the effect of pressure on the specularite upgrading in the Falcon concentrator separation method. The effect of the pressure on the grade and recovery iron concentrate at 20, 30, and 40 psi, respectively are shown in Fig. 4.25. The results showed that the pressure had a significant effect on grade iron concentrate. At pressure 20 psi, when the particle size of the specularite sample and centrifugal force was increased, iron concentrate grade gradually decreased to reach to particle size and the centrifugal force of 250 μm and 45.165 Hz respectively after when iron concentrate grade began to increase venially with an increase in particle size and centrifugal force.

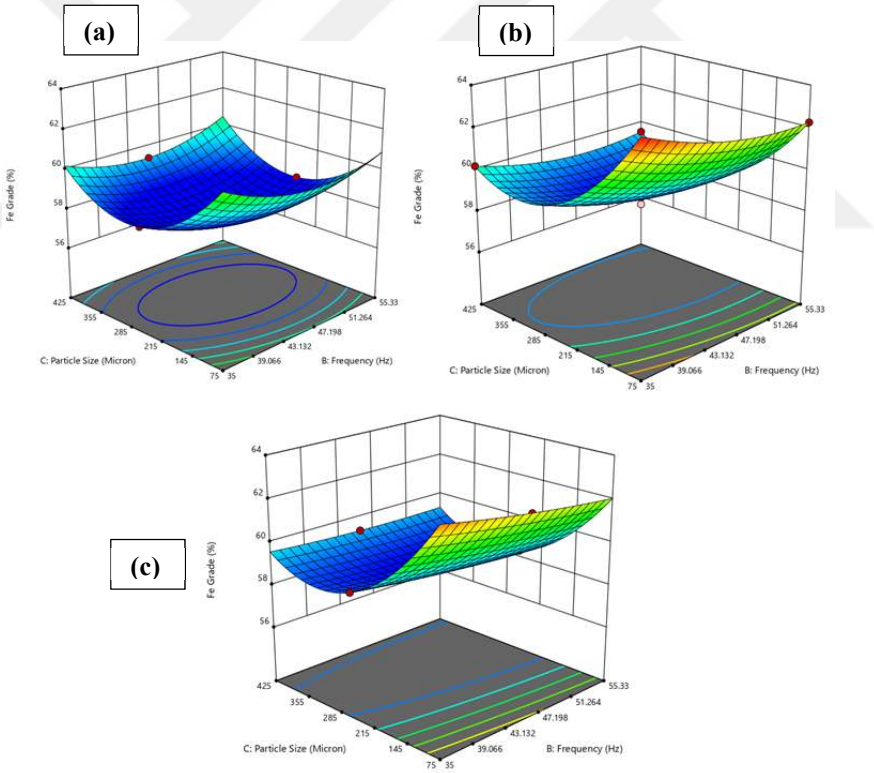

Figure 4.25. Effect of 20 psi (a), 30 psi (b), and 40 psi (c) pressure on iron grade.

At pressure 30 psi, the grade of iron decreased with increase in particle size and centrifugal force and the iron concentrate grade was at highest value (63.93% Fe) when particle size and centrifugal force were at the lowest value of 75 μm and 35 Hz respectively. The SEM images of iron concentrate (63.93% Fe grade) at 30 psi pressure, 35 Hz centrifugal force, and 75 μm particle size is shown in Fig. 4.26. The surface micrograph of iron ore concentrate at 63.93% Fe grade was irregular in size and shape as shown in fig 4.26.

At pressure 40 psi, effect of the particle size and centrifugal force were negative. Therefore, grade of the iron concentrate was decreased with increasing particle size and centrifugal force.

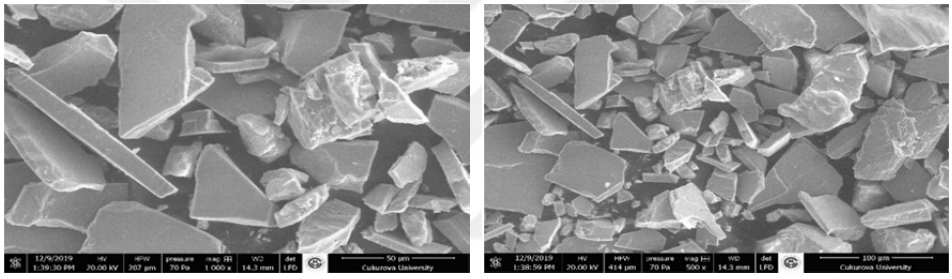

Figure 4.26. Iron concentrate (63.93% Fe grade) at 30 psi pressure, 35 Hz centrifugal force, and 75 μm particle size.

Due to the inverse relationship between grade and recovery, the grade increases with a decrease in recovery and vice versa, for this reason, it is noted from Fig. 4.27. the effect of pressure on recovery of the iron concentrate at different pressures (20 psi, 30 psi, and 40 psi) increases recovery by increasing particle size and centrifugal force. At different pressures, at a particle size of more than 250 μm, and different central forces, recoveries were at their highest values. At all the different pressures and a particle size of fewer than 250 μm, the yield was reduced by decreasing both particle size and pressure. The highest value of the grade of iron concentrate was (99.93%) at pressure, centrifugal force, and particle size of 20 psi, 55.33 Hz, and 250 μm respectively.

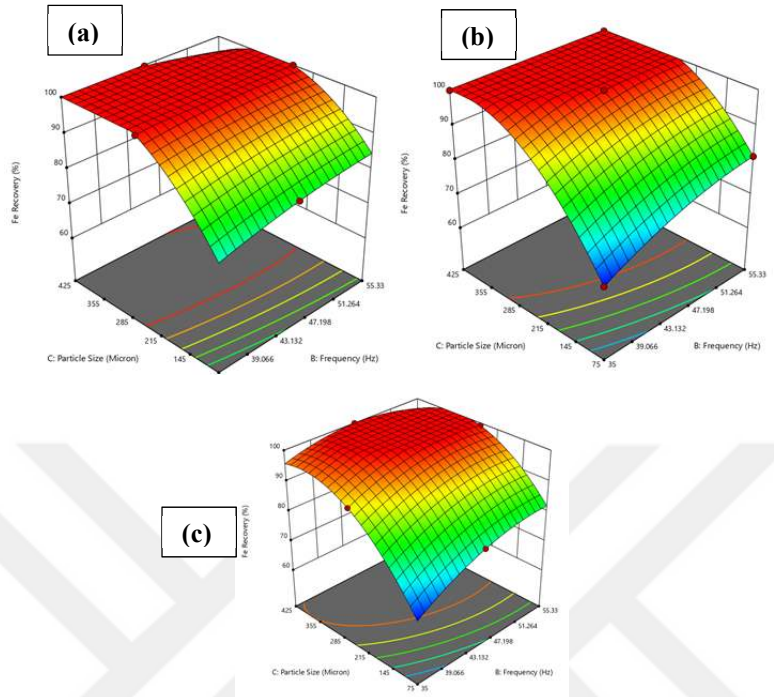

Figure 4.27. Effect of 20 psi (a), 30 psi (b), and 40 psi (c) pressure on iron concentrate recovery.

4.3.2. Effect of Centrifugal Force on Grade and Recovery of the Iron Concentrate

The effect of the 35 Hz, 45.165 Hz, and 55.33 Hz centrifugal forces on iron concentrate grade is shown in Fig. 4.28. The centrifugal force has a negative effect on iron concentrate grade. However, the iron concentrate grade decreases with an increase in centrifugal force. At 35 Hz centrifugal force, iron concentrate grade decreases with an increase in the particle size, and then iron concentrate grade begins to increase slightly with an increase in the particle size. While the iron concentrate grade is increased with an increase in the pressure to certain pressure and then iron concentrate grade is decreased with the increase of the pressure except at the particle size of 425 μm, the iron concentrate grade decreases slightly with the increase in the pressure. At centrifugal force 45.165 Hz, the effect of the particle size and pressure on the iron concentrate grade remain similar as that at the

centrifugal force of 35 Hz, therefore, iron concentrate grade decreases with increase in the particle size to the certain value of particle size and then iron concentrate grade begin to increase when particle size increase while the effect of pressure on iron concentrate grade is varying up and down except when the particle size is 425 µm, the effect of pressure on iron concentrate grade is negative as iron concentrate grade decreases with increasing pressure. At 55.33Hz centrifugal force, the effect of the particle size and pressure is similar to that at other values of the centrifugal force and comparing with the centrifugal force of 35 Hz, 45.165 Hz, the iron concentrate grades are at the lowest values.

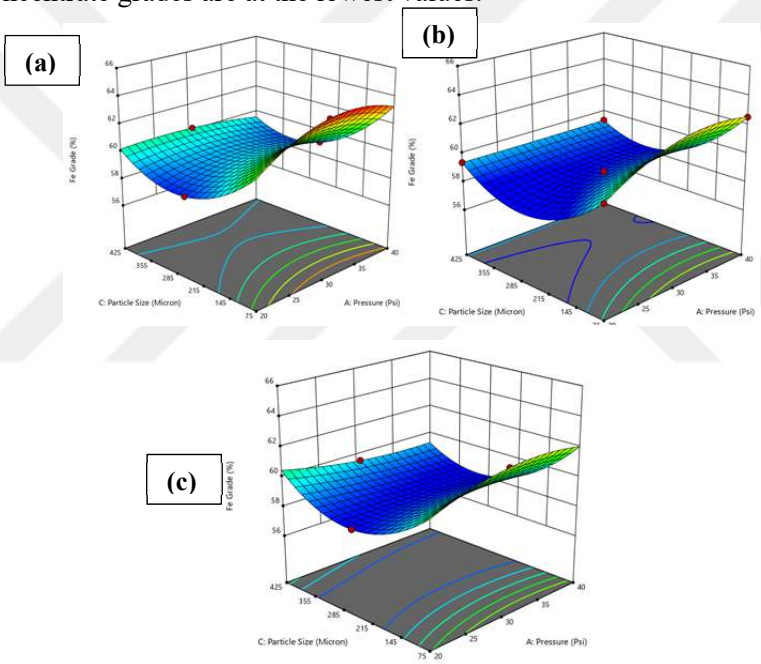

Figure 4.28. Effect of 35 Hz (a), 45.165 Hz (b), and 55.33 Hz (c) centrifugal force on iron concentrate grade.

4.3.2.1. Effect of Centrifugal Force on the Iron Concentrate Recovery

Fig. 4.29. shows the effect of the centrifugal force on iron concentrate grade at 35 Hz, 45.165 Hz, and 55.33 Hz respectively. The results obtained show that, generally, the grade of the iron concentrate was slightly increased with

increasing centrifugal force. Therefore, at the different centrifugal forces, the grade of the iron concentrate was increased with increasing particle size and decreasing pressure. At the centrifugal force of 35 Hz, the recovery was at it's the lowest value of 68.06% at pressure and particle size of 30 psi and 75 µm respectively. At the centrifugal force of both 45.165 Hz and 55.33 Hz, the effect of the pressure and particle size on the iron concentrate recovery was similar to its effect at a centrifugal force of 35 Hz, where iron concentrate recovery was increased with increasing particle size and decreasing pressure.

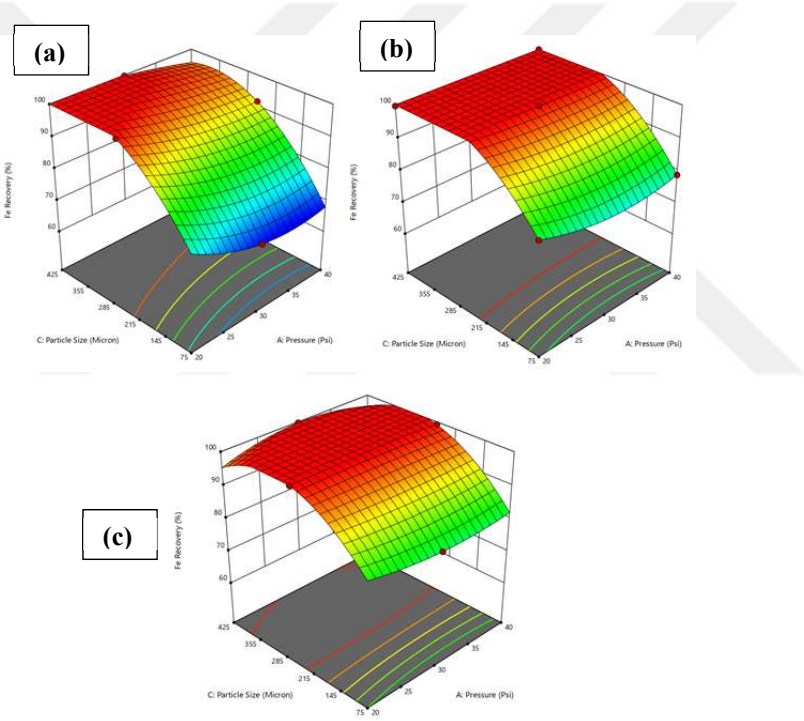

Figure 4.29. Effect of 35 Hz (a), 45.16 Hz (b), and 55.33 Hz (c) centrifugal force on iron concentrate recovery.

4.3.3. Effect of Particle Size on Grade and Recovery of the Iron Concentrate

Particle size has the greatest effect on the iron concentrate grade as shown in Fig. 4.30. When particle size was 75 µm, where 30 psi pressure and 35

centrifugal force were used, iron concentrate grade was at the highest value and reached 63.93% Fe. At particle size of 250 µm, the grade of iron concentrate was increased to a certain extent and then decreased slightly by increasing pressure, while the iron concentrate grade was reduced and then increased slightly by increasing the centrifugal force. At particle size of 425 µm, the effect of the pressure and centrifugal force on the grade of iron concentrate was uneven, slightly decreasing, and increasing with increasing and decreasing both pressure and centrifugal force. At 450 µm particle size, the highest value of iron concentrate grade was 60.21 % Fe at 30 psi pressure and 35 Hz centrifugal force.

The SEM images of iron concentrate (60.21 % Fe grade) at 30 psi pressure,35 Hz centrifugal force, and 425 µm particle size are shown in Fig. 4.31.

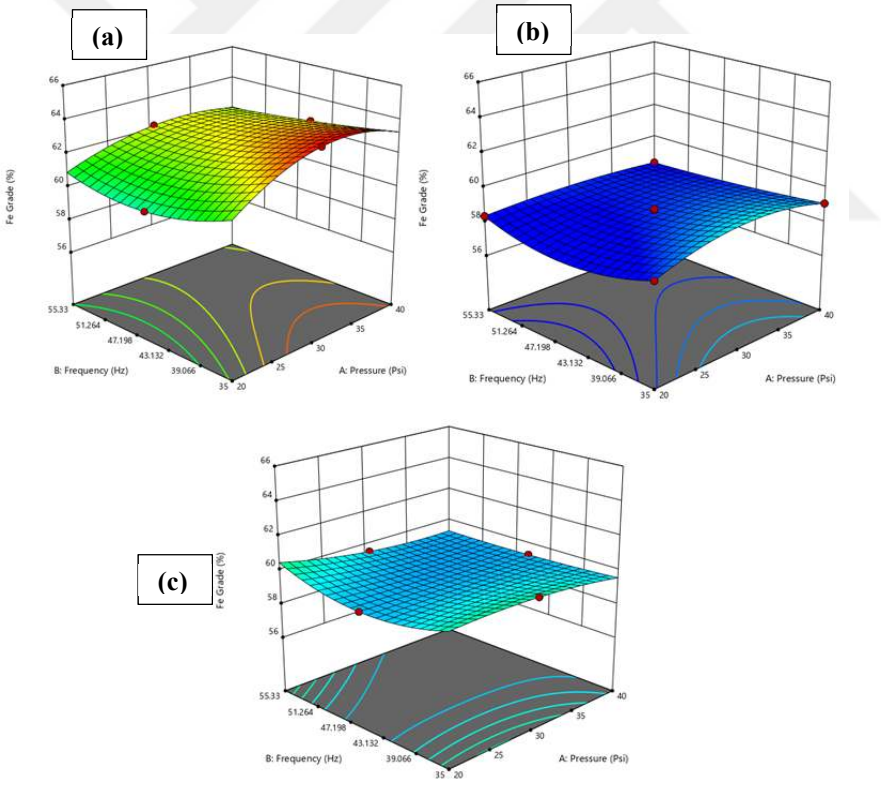

Figure 4.30. Effect of 75 µm (a), 250 µm (b), and 425 µm (c) particle size on iron concentrate grade.

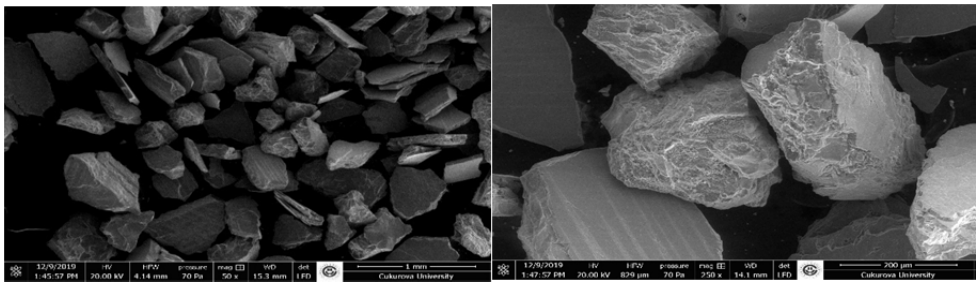

Figure 4.31. SEM images of iron concentrate (60.21 % Fe grade) at 30 psi pressure, 35 Hz centrifugal force and 425 µm particle size

The effect of the particle size on the iron concentrate recovery at 75 µm, 250 µm, and 425 µm is shown in Fig. 4.32. As seen from the Fig., at a particle size of 75 µm, the recovery of the iron concentrate was increased with an increase in the pressure and decrease of the centrifugal force to a certain extent, and then iron concentrate recovery began to increase slightly with increasing pressure. At this particle size, the recovery of the iron concentrate was at the lowest value. On the other hand, the value of grade of the iron concentrate was at its highest, which explains the inverse relationship between grade and recovery. At 250 µm particle size, the iron concentrate recovery was at the highest value at all the values of the pressure and centrifugal except at pressure and centrifugal were 40 psi and 35 Hz respectively, and iron concentrate recovery was 91.81%.

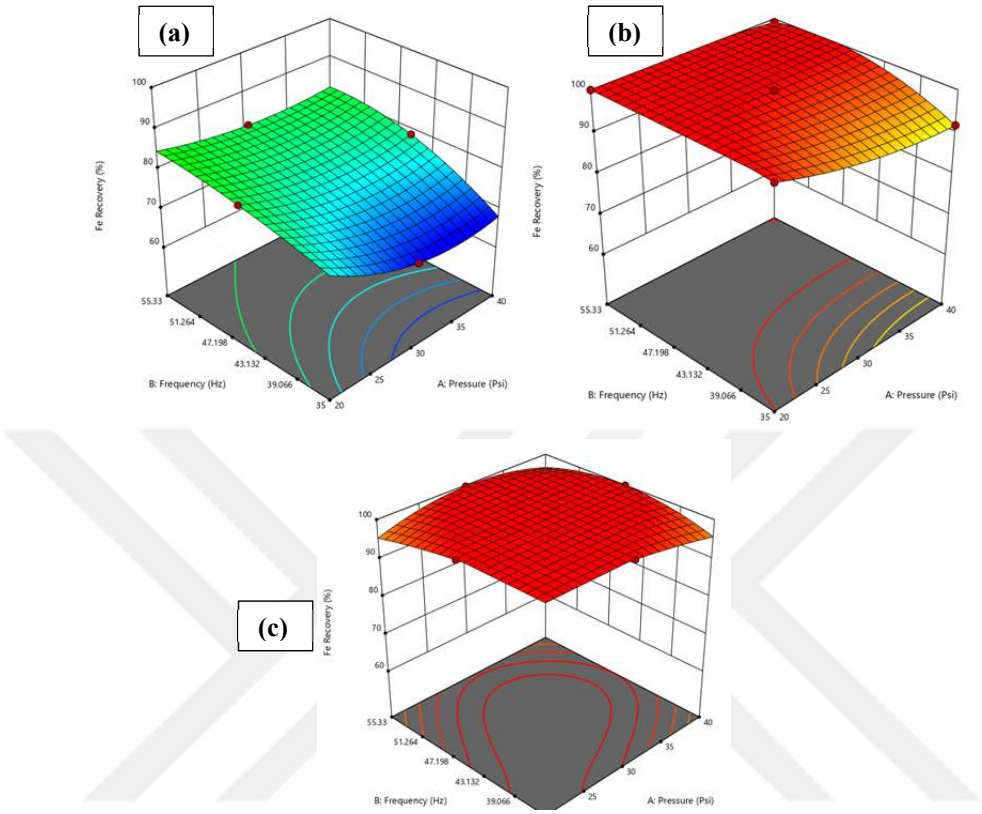

Figure 4.32. Effect of 75 µm (a), 250 µm (b), and 425 µm (c) particle size on iron concentrate recovery.

In Falcon concentrator test, 30 psi pressure, 55.33 Hz centrifugal force, -75 µm particle size, and 20 % solid/liquid ratio were the optimum conditions while grade and recovery of the iron concentrate at these conditions were 62.28% Fe and 81.34 % respectively.

According to the experimental results, both of the frequency and particle size had a significant effect of grade and recovery of the iron concentrate while the effect of the pressure on the grade and recovery of the iron concentrate was not significant. In Falcon concentrator test, the particle size had the greatest effect on grade and recovery of the iron concentrate.

ANOVA tests revealed that iron concentrate grade and recovery recoveries could be explained by cubic models. The correlation coefficients (R^2) for iron concentrate grade and recovery were calculated as 0.998 (0.013P-value) and 0.999(0.0013P-value) respectively. As can be seen from the high R^2 values, there is an excellent correlation between parameters. Equations 6 and 7 show the created models:

$$\text{Fe grade (\%)} = 58.61 + 0.5525\ X_1 - 58\ X_2 - 1.62\ X_3 - 1325\ X_1X_2 - 0.5775\ X_1X_3 + 0.245\ X_2X_3 - 0.5567\ X_1^2 + 0.5358\ X_2^2 + 2.34\ X_3^2 + 0.3375\ X_1^2X_2 + 0.5825\ X_1^2X_3 - 0.42\ X_1X_2^2 \quad (6)$$

$$\text{Fe recovery (\%)} = 99.6 - 0.7775\ X_1 + 3.3\ X_2 + 12.51\ X_3 + 1.73\ X_1X_2 + 0.8925\ X_1X_3 + 03.34\ X_2X_3 + 0.4988\ X_1^2 - 2.50\ X_2^2 - 9.89\ X_3^2 - 1.36\ X_1^2X_2 - 2.92\ X_1^2X_3 - 1.34\ X_1X_2^2 \quad (7)$$

Where; X_1: pressure (psi), X_2: centrifugal force (Hz) and X_3: particle size (μm).

4.3.4. Reverse Anionic Flotation Experiments

Reverse anionic flotation experiments were carried out to reveal the role of depressant dosage, collector dosage, and froth collection time in the flotation of quartz from specularite. Table 4.8. shows results of Box Behnken test design of reverse anionic flotation experiments,

The effect of each parameter on the grade and recovery of iron concentrate was discussed in the following headings.

Table 4.8. Box Behnken test design of reverse anionic flotation experiments.

Run	Collector g/ton (x_1)	Depressant g/ton (x_2)	Froth Collection Time Min(x_3)	Fe Grade %	Fe Recovery %
1	1500	10000	2	62.79	95.74
2	1000	5250	2	63.04	96.85
3	1500	5250	1	62.26	98.96
4	1000	500	1	62.02	98.65
5	1000	10000	3	62.93	93.8
6	1500	500	2	60.99	96.24
7	1000	10000	1	61.04	96.57
8	1500	5250	3	62.98	92.93
9	1000	500	3	61.57	93.15
10	1000	5250	2	62.31	95.05
11	1000	5250	2	62.29	95.04
12	500	5250	1	62.55	94.76
13	500	500	2	61.59	91.09
14	500	10000	2	61.93	93.23
15	500	5250	3	62.41	92.4

4.3.5. Effect of Depressant Dosage on Grade and Recovery of the Iron Concentrate

The dosage of the corn starch had a significant and positive effect on iron concentrate grade. Generally, the grade of the iron concentrate was slightly increased with increasing depressant dosages. Fig. 4.33. shows the effect of the depressant on iron concentrate grade at 500 g/ton, 5250 g/ton, and 10000 g/ton conditions respectively. Using 500 g/ton depressant, iron concentrate grade was decreased at increasing collector and froth collection time. At a depressant dosage

of 5250 g/ton, iron concentrate grade was slightly increased by increasing collector and froth collection time. The highest grade of iron concentrate (63.04% Fe) was obtained from the medium values of collector and froth collection time. At the depressant dosage of 10000 g/ton, iron concentrate grade was continuing to increase with increase in both collector and froth collection time and the general effect of the depressant dosage on the iron concentrate grade at this dosage was more positive compared to lower dosages of depressant. High pH values have an effect that increases the effect of corn starch (Yang et al., 2017). Experiments in this study were conducted at pH 12.5 also resulted in >63% iron concentration grades (Fig. 4.45.).

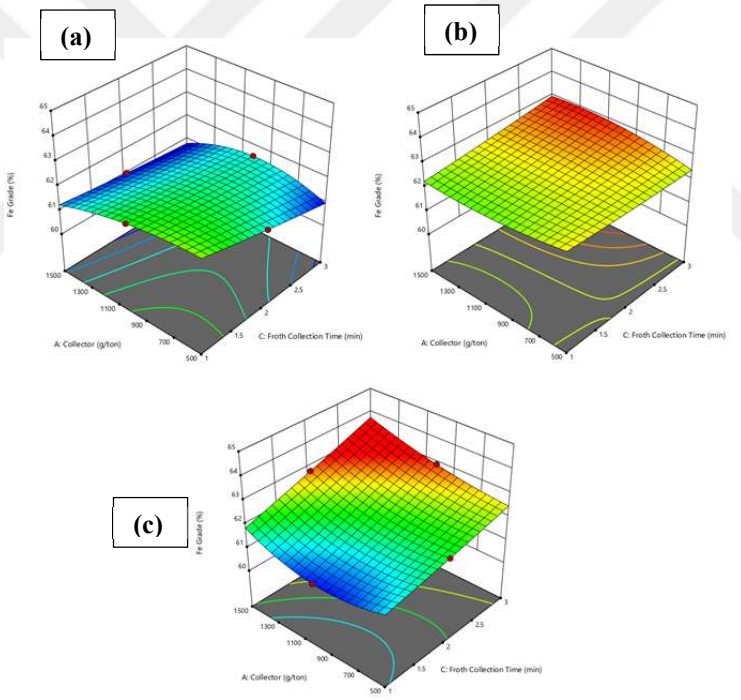

Figure 4.33. Effect of 500 g/ton (a), 5250 g/ton (b), and 10000 g/ton (c) depressant dosages on iron concentrate grade.

Effect of the depressant dosages on specularite at 500 g/ton, 5250 g/ton, and 10000 g/ton were shown in Fig. 4.34. As shown, the effect of the depressant on

the iron concentrate recovery was negative and the effect was almost the same for all experiments. At different dosages of depressant, the iron concentrate recovery was increased with increasing collector dosage and decreasing froth collection time. The highest value of iron concentrate recovery (98.96%) was achieved by 5252 g/ton of depressant usage at 1500 g/ton collector and 1 min froth collection time.

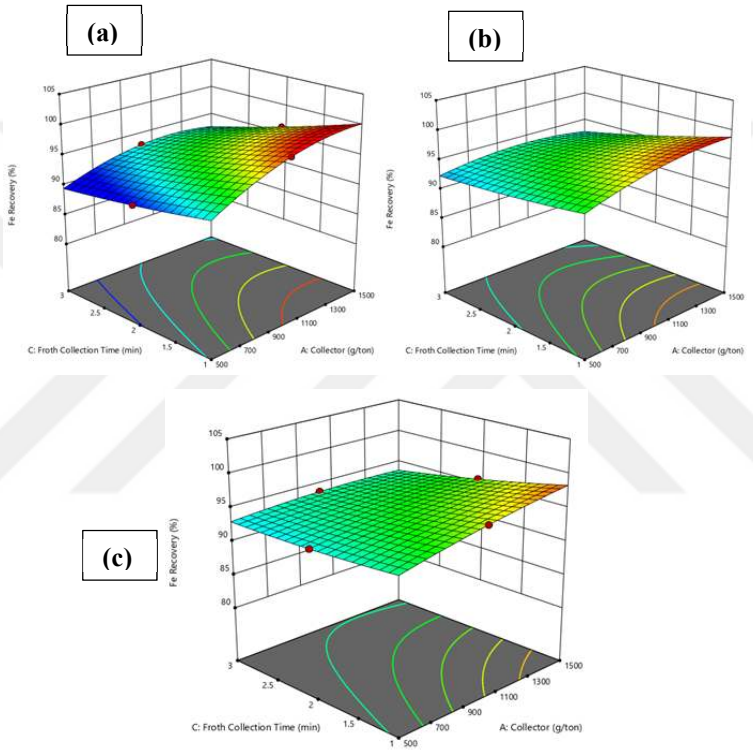

Figure 4.34. Effect of 500 g/ton (a), 5250 g/ton (b) and 10000 g/ton (c) depressant dosage on iron concentrate recovery.

4.3.6. Effect of Collector Dosage on Grade and Recovery of the Iron Concentrate

As seen in Fig. 4.35., under the highest depressant and froth collection time conditions, Fe grade increased according to the increase in the Aero 343 collector dosage. When 500 g/ton collector was used, iron concentrate grade was slightly

increased with the increase of the depressant. Then, iron concentrate grade was slightly decreased with increased depressant values but iron concentrate grade continued to increase by increasing the depressant at froth collection time of 3 minutes. When the collector was used more than 1000 g/ton and both of the depressant and froth collection time were at the highest values, the effect of Aero 343 on iron concentrate grade was positive. While the collector dosage was the lowest, more flotation time and highest values of depressant dosages were needed to obtain high-grade concentrate. No significant increase was observed in the iron grades after 5000 g/ton of depressant and 2 min of froth collection time. At collector dosage of 1500 g/ton, the effect of the froth collection time on iron concentrate grade remained the same at the lower froth collection times (1-2 min). The maximum iron content was obtained with the depressant dosage (5252 g/ton) for 2 min (Fig. 4.35.).

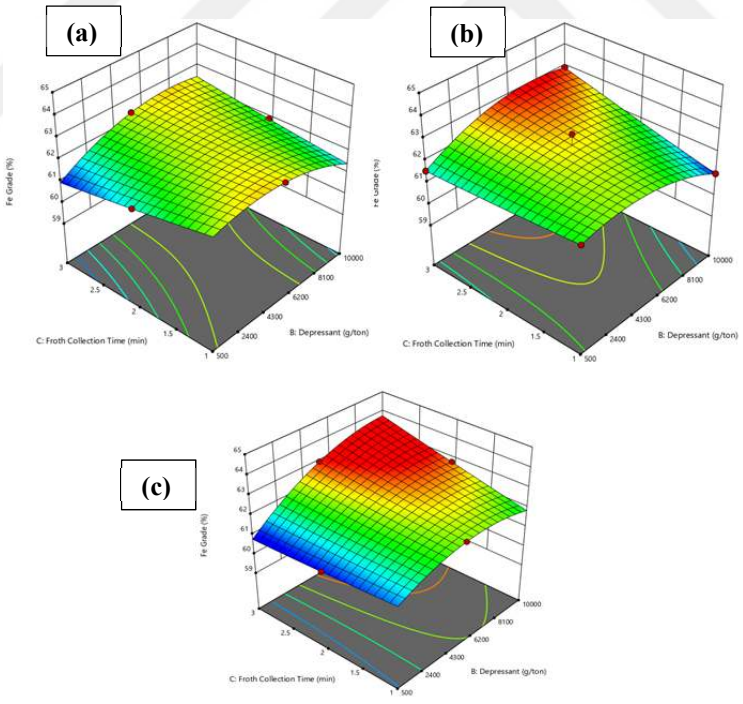

Figure 4.35. Effect of 500 g/ton (a), 1000 g/ton (b) and 1500 g/ton (c) collector dosage on iron concentrate grade.

The effect of the 500 g/ton, 1000g/ton, and 1500 g/ton collector dosages on iron concentrate yield were shown in Fig. 4.36. The collector dosage had a positive effect on iron concentrate recovery. However, the iron concentrate recovery increased according to the increase in the collector dosage and the decrease in both depressant dosage and froth collection time. Iron concentrate recovery reached its highest value at 1500 g/ton collector dosage. At this test, depressant and froth collection time were 500 g/ton and 1 min respectively.

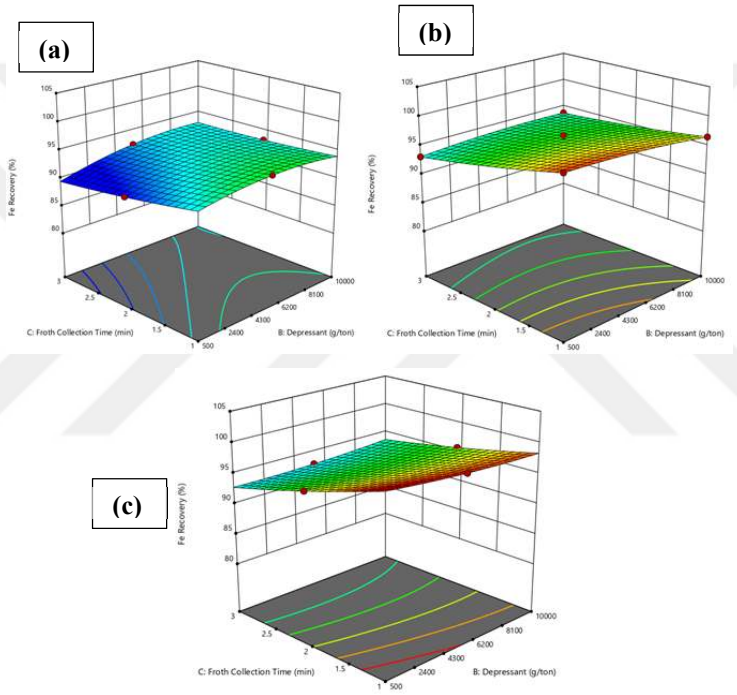

Figure 4.36. Effect of 500 g/ton (a), 1000 g/ton (b) and 1500 g/ton (c) collector dosage on iron concentrate recovery.

4.3.7. Effect of Collection Time on Grade and Recovery of the Iron Concentrate

The effect of froth collection time on iron concentrate grade was observed and the results obtained as shown in Fig. 4.37. at froth collection times of 1, 2, and 3 min respectively. Generally, the effect of froth collection time on the iron

concentrate grade was not stable. However, the effect was positive at 3 min and the highest values of depressant and collector. For 1 min collection time, iron concentrate grade was slightly increased with the increase in the depressant and then iron concentrate grade began to decrease slightly with the increase in the depressant dosage. When 3350 g/ton depressant and 500 g/ton collector were used, the iron concentrate grade began to decrease slightly. Under the 1500 g/ton collector usage condition, a significant increase in Fe grade seems to be obtained according to the increase in depressant dosage. While the other variables were at the highest value, the iron grade was obtained at the highest value for 3 min froth collection time.

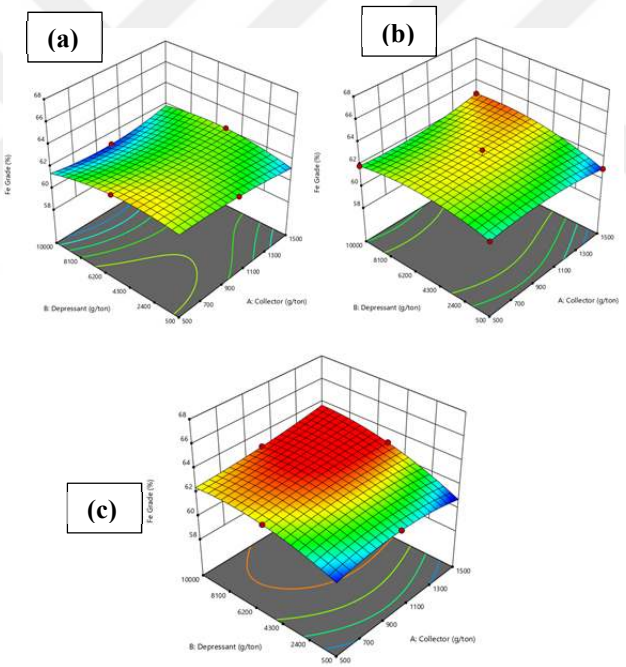

Figure 4.37. Effect of (%) at 1 (a), 2 (b) and 3 (c). collection time (min.) on iron grade

Effect the froth collection time on iron concentrate recovery was remarkable. Froth collection time had the greatest effect on the iron concentrate

recovery as shown in Fig. 4.38. When froth collection time was 1 min, where 5250 g/ton depressant and 1500 g/ton collector were used, iron concentrate recovery reached 98.96%. This is because of the reverse anionic flotation method. In standard flotation tests, recovery recoveries increase with increasing froth collection times. Nevertheless, this phenomenon is vice versa in the reverse anionic flotation method which floats the gangue minerals. When the froth collection times were increased, the iron concentrate recoveries were decreasing gradually.

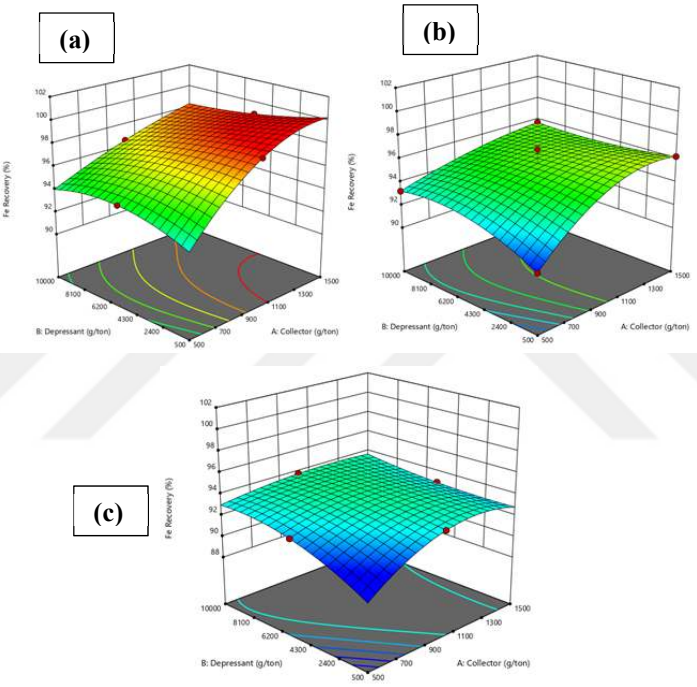

Figure 4.38. Effect of time on iron concentrate recovery (%) at 1 (a), 2 (b), and 3 min (c).

In the anionic flotation test, depressant dosage had the greatest influence on the iron concentrate grade while the effect of the collector dosage and froth collection time was less. Froth collection time had the greatest effect on iron concentrate yield.

ANOVA tests revealed that iron concentrate grade and recovery recoveries could be explained by cubic models. The correlation coefficients (R^2) for iron concentrate grade and recovery were calculated as 0.94 and 0.97, respectively. It was shown a good correlation for parameters. Equations 8 and 9 show the created models:

$$\text{Fe grade (\%)} = 62.55 + 0.07X_1 + 0.095\ X_2 + 0.36\ X_3 + 0.365\ X_1X_2 + 0.215X_1X_3 + 0.585\ X_2X_3 - 0.0308\ X_1^2 - 0.6908\ X_2^2 + 0.0342\ X_3^2 + 0.44\ X_1^2X_2 - 0.215\ X_1^2X_3 - 0.005X_1X_2^2 \quad (8)$$

$$\text{Fe recovery (\%)} = 95.65 + 1.18\ X_1 - 0.358\ X_2 - 2.07\ X_3 - 0.66\ X_1X_2 - 0.9175\ X_1X_3 + 0.6825\ X_2X_3 - 1.18\ X_1^2 - 0.3958\ X_2^2 + 0.2917\ X_3^2 + 0.7675\ X_1^2X_2 - 0.03\ X_1^2X_3 - 0.7325\ X_1X_2^2 \quad (9)$$

Where; X_1: collector dosage (g/ton), X_2: depressant dosage (g/ton) and X_3: froth collection time (min).

4.3.8. Reverse Cationic Flotation Experiments

Reverse cationic flotation test carried out to investigate the effect of the dosages of the depressant, collector, and frother as well as to investigate the effect of the froth collection time on the grade and recovery of the iron concentrate and increase it as possible. Table 4.9. shows the results of Box Behnken test design of reverse cationic flotation experiments.

Table 4.9. Results of Box Behnken test design of reverse cationic flotation experiments.

	Depressant g/ton (X_1)	**Collector g/ton (X_2)**	**Frother g/ton (X_3)**	**Time (Min) (X_4)**	**Fe Grade %**	**Fe Recovery %**
1	500	400	100	2	67.42	68.08
2	500	300	150	2	61.86	85.88
3	500	400	150	3	64.69	90.57
4	500	200	50	3	60.19	82.94
5	400	200	100	3	63.37	84.52
6	500	400	50	3	66.77	65.49
7	500	300	50	2	63.26	78.50
8	500	200	150	3	64.85	80.81
9	500	300	100	4	61.07	67.89
10	500	300	100	4	65.62	67.09
11	600	300	100	2	64.80	66.37
12	600	400	100	3	65.16	59.57
13	500	300	50	4	67.41	86.01
14	500	300	150	4	66.60	77.31
15	500	400	100	4	69.17	56.64
16	500	200	100	4	65.03	84.68
17	400	400	100	3	68.05	77.11
18	400	300	50	3	64.34	76.68
19	600	200	100	3	63.62	80.18
20	600	300	150	3	63.19	76.29
21	500	200	100	2	60.62	79.79
22	400	300	100	2	68.95	81.90
23	600	300	50	3	68.11	75.95
24	400	300	150	3	68.28	70.22

The effect of these parameters was as follows:

4.3.9. Effect of Depressant Dosage on Grade and Recovery

Effect of depressant dosages at 2,3 and 4 min collection time were investigated and the results showed that when the froth collection time of 2 min, at depressant dosages of 400 g/ton, the effect of frother and collector dosages were positive, therefore, iron concentrate grade was increased by increasing both frother and collector dosages (Fig 4.39). At this time collection and depressant dosage, the highest value of iron concentrate was 98.58% at frother dosage and collector dosage of 100 g/ton and 300 g/ton respectively. At depressant dosages of 500 g/ton, the iron concentrate was slightly increasing with increased frother and collector dosages until the reach of frother and collector dosages of 80 g/ton and 320 g/ton respectively, after this point, the iron concentrate grade was decreased slightly with frother and collector dosages were increased. When the depressant dosage was 600 g/ton, iron concentrate grade was slightly increased by increasing both frother and collector dosages until reaching to frother dosage of 100 g/ton after this frother dosage the iron concentrations began to decrease slightly with increasing frother dosage.

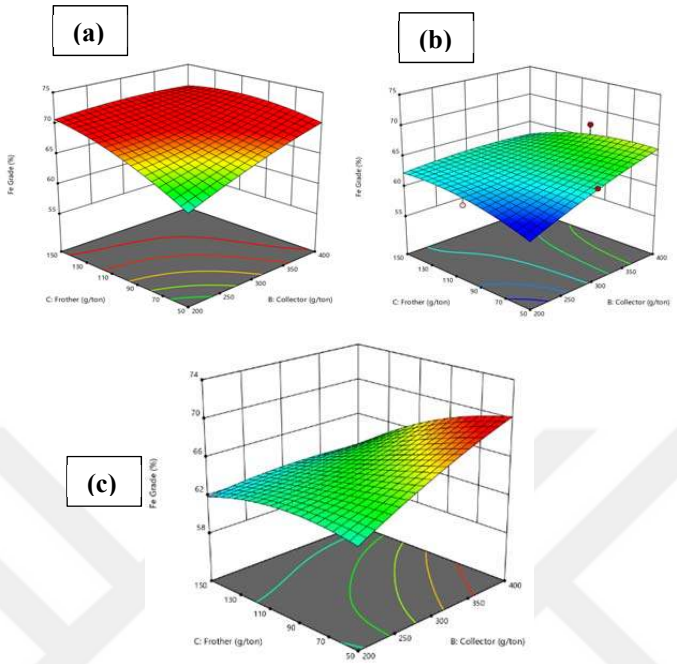

Figure 4.39. Effect of 400 g/ton (a). 500g/ton (b) and 600 g/ton (c) depressant dosages at 2 min froth collection time on iron concentrate grade,

At 3 min collection time, when the depressant dosage was 400 g/ton, the effect of frother and collector dosages was positive. Therefore, iron concentrate grade was increased by increasing both frother and collector dosages. Generally, at 400 g/ton depressant dosage, good depressing abilities for iron concentrate during the flotation test was observed. At 500 g / ton of the depressant dosage, iron concentrate grade was increased with increasing doses of frother and collector except at the highest frother value of 150 g/ton, the iron concentrate grade was slightly increasing with increasing collector dosage and then began to decrease slightly. At 600 g/ton depressant dosage, the general effect of the depressant dosage on iron concentrate dosage was approximately the same at 500 g/ton. At 600 g/ton depressant dosage (Fig. 4.40.).

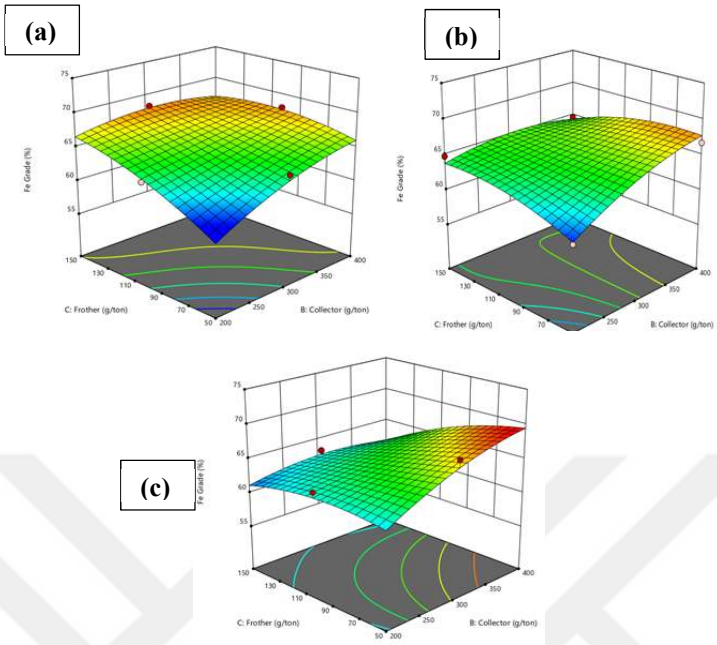

Figure 4.40. Effect of 400 g/ton (a). 500g/ton (b) and 600 g/ton (c) depressant dosages at 3 min froth collection time on the iron concentrate grade.

At 4 min of froth collection time, as shown in Fig. 4.43., the general effect of depressant dosages on the grade of iron concentrate remained similar to that at 2 and 3 mins. at this time of froth collection grade of iron, concentrate reach to it is the highest value of 69.17% Fe due to collection more amount of silica and calcium oxide comparing with another froth collection time. At this point, depressant, frother, and collector dosages were 500 g/ton, 100 g/ton, and 400 g/ton respectively **(**Fig. 4.41).

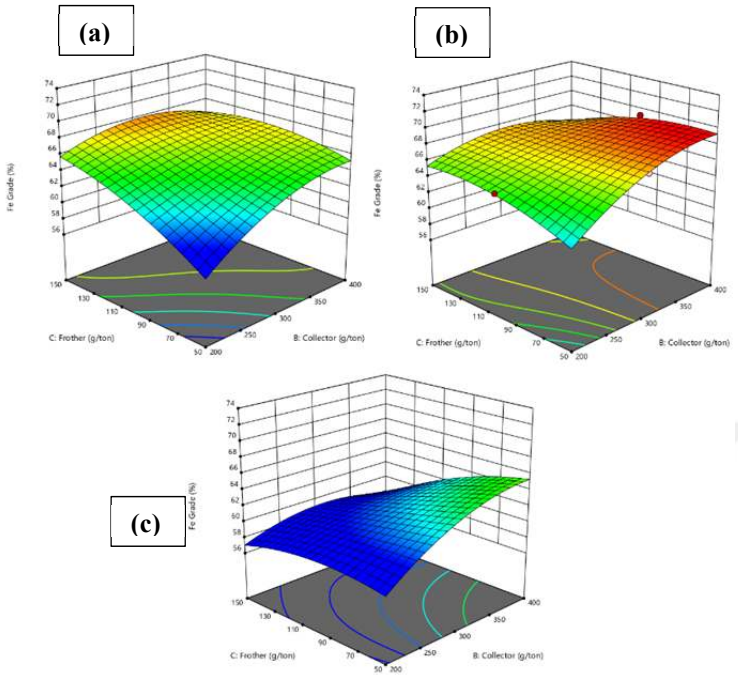

Figure 4.41. Effect of 400 g/ton (a). 500g/ton (b) and 600 g/ton (c) depressant dosages at 4 min froth collection time on iron concentrate grade.

Fig. 4.42. shows the effect of depressant dosages on the iron concentrate recovery. The result showed that generally at 400 g/ton depressant dosages iron concentrate recovery was slightly increased and then decreased by increasing frother dosage when the collector dosage was less than 250 g/ton. When the depressant dosage was 500 g/ton, where 400 g/ton collector dosage and 150 g/ton frother dosage were used, iron concentrate recovery was at its highest value and reached 90.57%. At 500 g/ton and 600 g/ton depressant dosages, the iron concentrate recovery was decreased and then began to increase with increasing frother dosage and recovery was increased and then decreased with increasing collector dosage but at a high frother dosage (more than 130 g/ton) the recovery was continue increased with increasing collector dosage. At the point of 120 g/ton frother dosage and 300 g/ton collector dosage, the negative effect of dosages after this point on the iron concentrate recovery was clear. The effect of depressant

dosages over 500 g/ton on the iron concentrate recovery was negative compared with depressant dosages less than 500 g/ton (Fig. 4.42).

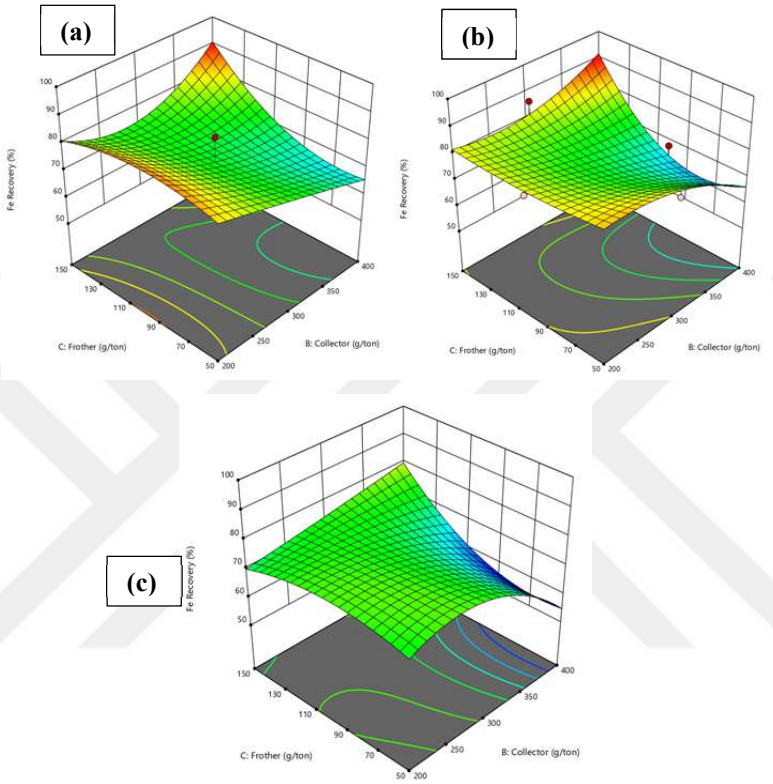

Figure 4.42. Effect of 400 g/ton (a). 500g/ton (b) and 600 g/ton (c) depressant dosages on the iron concentrate recovery.

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4.3.10. Effect of Collector Dosage on Grade and Recovery the Iron Concentrate

Fig. 4.43. shows the effect of collector dosages on the grade of iron concentrate at 2, 3, and 4 min of froth collection time. From the Fig., it is clear that at 2 min of froth collection time and different collector doses, the general effect of collector dosages on iron concentrate time was noticeably different whereas the grade of iron concentrate was increased with increasing frother dosages while the iron concentrate grade was decreased and then began to increase again with

increasing depressant dosages. At 200 g/ton collector dosage, the iron concentrate grade was lower than that at 300 and 400 g/ton. Compared with collector dosages of 200 g/ton and 300 g/ton, the iron concentrate grade values were higher due to the positive effect of collector dosages.

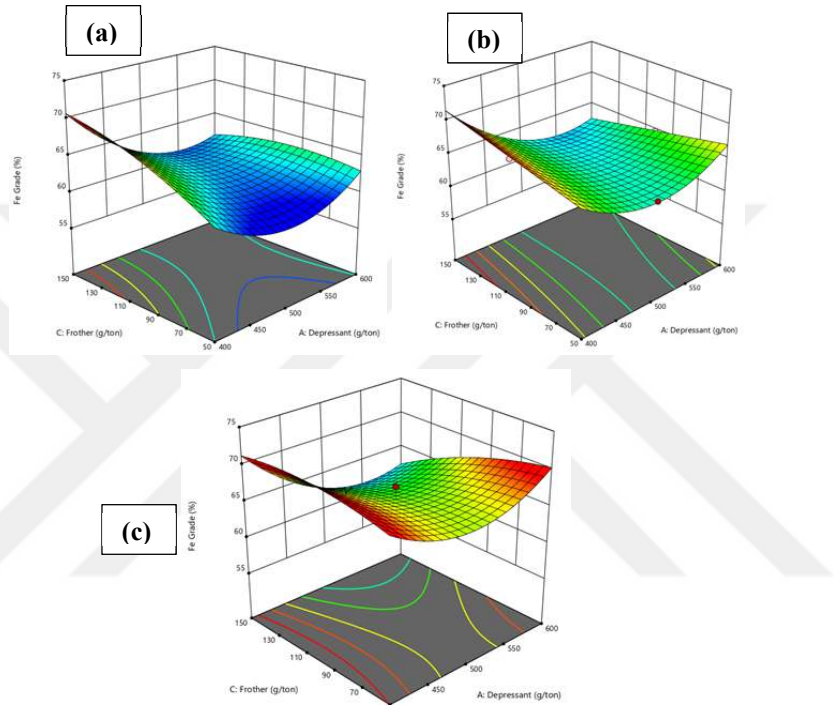

Figure 4.43. Effect of 200 g/ton (a). 300g/ton (b) and 400 g/ton (c) collector dosages at 2 min froth collection time on iron concentrate grade.

Fig. 4.44. shows the effect of 200g/ton, 300 g/ton, and 400 g/ton collector dosages on the grade of iron concentrate. In general, in the reverse anionic flotation test, the collector selectivity to float the quartz was increased by increasing the collector dosages. From the Fig., it is clear that the collector doses have a significant and positive effect on the iron concentrate grades. Therefore, the iron concentrate grades were increased with increasing collector dosages due to increasing the selectivity of flotation. At collector dosages of 200 g/ton,300 g/ton and 400 g/ton, generally, the iron concentrate grades were increased with an

increasing frother and depressant dosages except when both the depressant and frother dosages were over 450 g/ton and 100 g/ton respectively, the general effect of collector dosages on iron concentrate was negative and iron concentrate grades were decreased with increasing depressant and frother dosages. Also, the negative effect of depressant dosages over 600 g/ton and frother dosages over 150 g/ton was observed during preliminary experiments. Compared with 200 g/ton collector dosage, when the collector dosage was 300 g/ton the iron concentrate grade was higher while the grades of iron concentrate were at its highest values at 400 g/ton collector dosage.

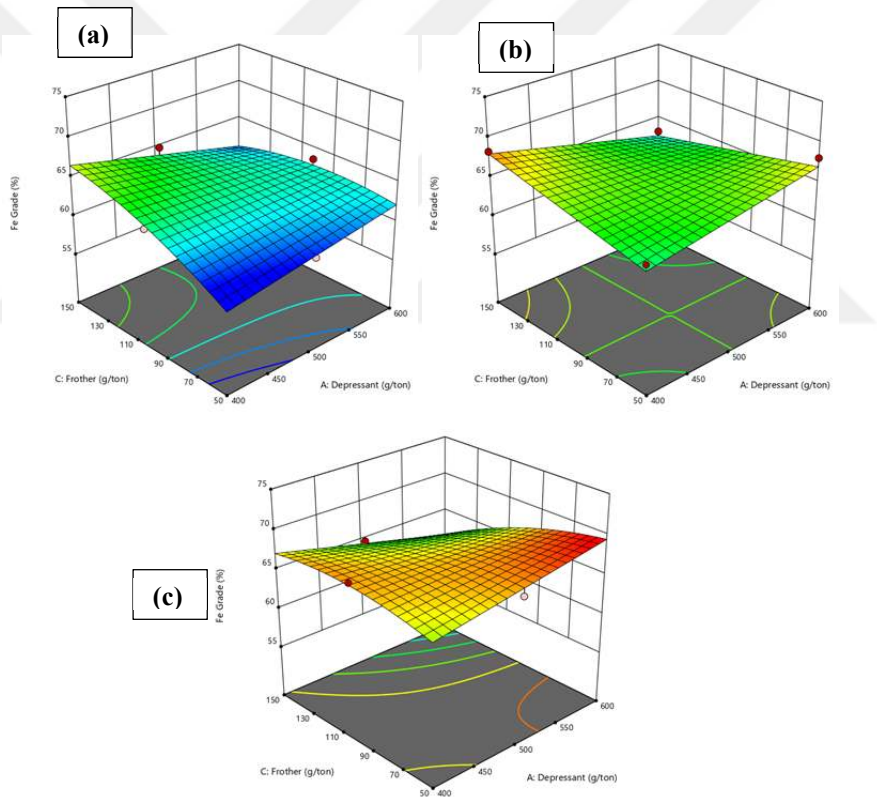

Figure 4.44. Effect of 200 g/ton (a). 300g/ton (b) and 400 g/ton (c) collector dosages at 3 min froth collection time on iron concentrate grade.

At 4 min of froth collection time and all the dosages of the collector, grades of iron concentrate were increased with increasing frother dosage whereas the grades were increased and then began to decrease with increasing depressant dosages. The negative effect of depressant dosage at 600 g/ton remained the same and at this dosage, grades were decreased as frother dosage was increasing. At this time of collection time, the grade of iron concentrate was at its highest value of 69.17% Fe at collector, depressant, and frother dosages of 400 g/ton, 500 g/ton, and 100 g/ton respectively. This was because of reverse flotation (Fig. 4.45).

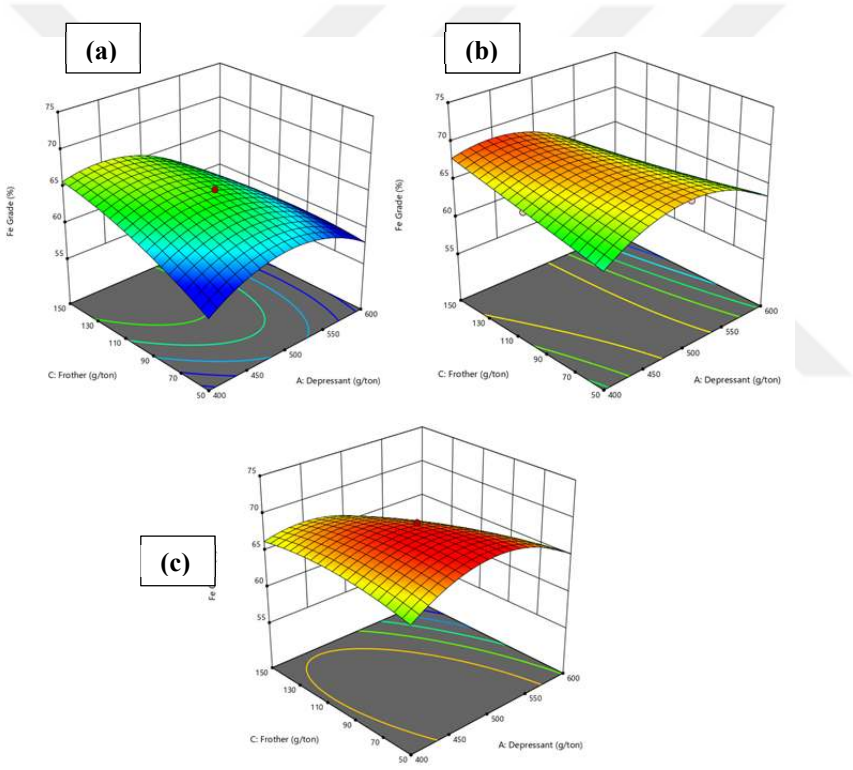

Figure 4.45. Effect of 200 g/ton (a). 300g/ton (b) and 400 g/ton (c) collector dosages at 4 min froth collection time on iron concentrate grade.

Effect of the collector dosages on the iron concentrate recovery shown in Fig. 4.46. At 200 g/ton collector dosage, generally, iron concentrate recovery was slightly increased and then began to decrease with an increase in both frother and

depressant dosages. When the collector dosage was 300 g/ton, iron concentrate recovery was decreased and then slightly increased by frother dosage increase while the iron concentrate recovery was increased and then slightly decreased with increasing depressant dosage. At 400 g/ton collector dosage, iron concentrate grade was largely increased with increasing frother dosage while the iron concentrate grade was slightly increased and then decreased with increasing depressant dosage. At this collector dosage, it is clear that there is a negative effect of the frother dosages on iron concentrate recoveries when were at dosages less than 100 g/ton . Iron concentrate recovery was at its lowest value of 54.64% at 400 g/ton collector dosage. At this point, both depressant and frother dosages were 500 g/ton and 100 g/ton respectively for 4 min froth collection time. But on the other hand, the iron concentrate grade was at its highest value and reached 69.17% Fe and this was because of reverse flotation.

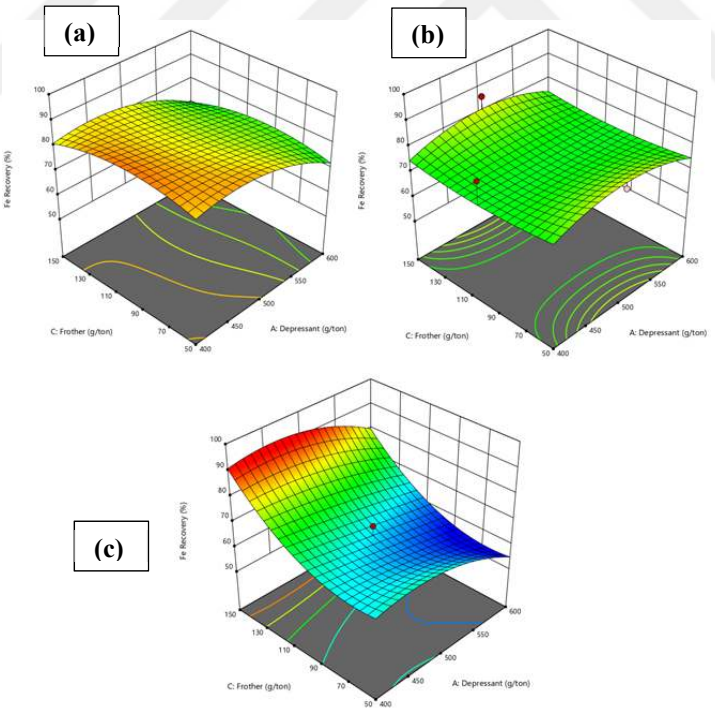

Figure 4.46. Effect of 200 g/ton (a). 300g/ton (b) and 400 g/ton (c) collector dosages on iron concentrate recovery.

4.3.11. Effect of Frother Dosage on Grade and Recovery of the Iron Concentrate

The frother affects the particle bubble adhesion kinetics, rendering the relaxation time shorter than the contact time. Under these conditions, the collision time is longer than the time required for thinning and rupture of the lamella surrounding the bubble (Araujo, 2005). For this reason, when using these doses on the experiments, we note that it has a significant impact on grades and recoveries of the iron concentrate compared to those experiments which did not use these doses. The presence of the frother works to raise the efficiency of the bubble, maintain and increase the collision time, which in turn works to collecting and taking as many undesirable materials (silica and calcium oxide) from the specularite ore and thus increase the grades and recoveries of iron concentrate.

At 2 min collection time, when frother dosage was 50 g /ton, iron concentration grade was decreased to a certain extent and then increased with increasing dosage of depressant while the effect of collector dosage on the grade of iron concentrate was more positive as grade increased with increasing collector dosage. At doses over 100 g / ton, the overall effect was almost similar to that at 50 g / ton. In contrast, the highest grades of iron concentrate were when the depressant dosages were at their lowest doses (400 g/ton) (Fig. 4.47).

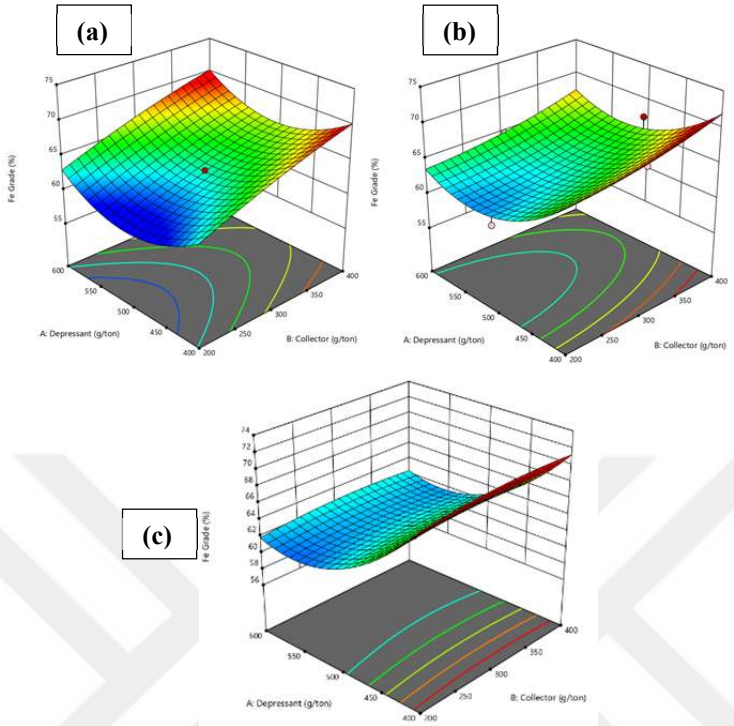

Figure 4.47. Effect of 50 g/ton (a). 100 g/ton (b) and 150 g/ton (c) frother dosages at 2 min froth collection time on iron concentrate grade.

At 3 min collection time, as seen in Fig. 4.48. when the frother dosage was 50 g/ton, iron concentrate grade was increased with an increase in both depressant and collector doses. At 100 g/ton frother dosage, iron concentrate grade increased when the collector dosage was increased while there was no significant effect for depressant dosages on iron concentrate grades observed at this frother dosage. At 150 g/ton frother dosage, depressant dosages have been a negative effect on iron concentrate grades while collector dosages had a slight and varying effect on the recovery of iron concentrate, therefore, iron concentrate grades were increased with decreasing depressant dosages while iron concentrate grades were slightly increased and then decreased with increasing collector dosages.

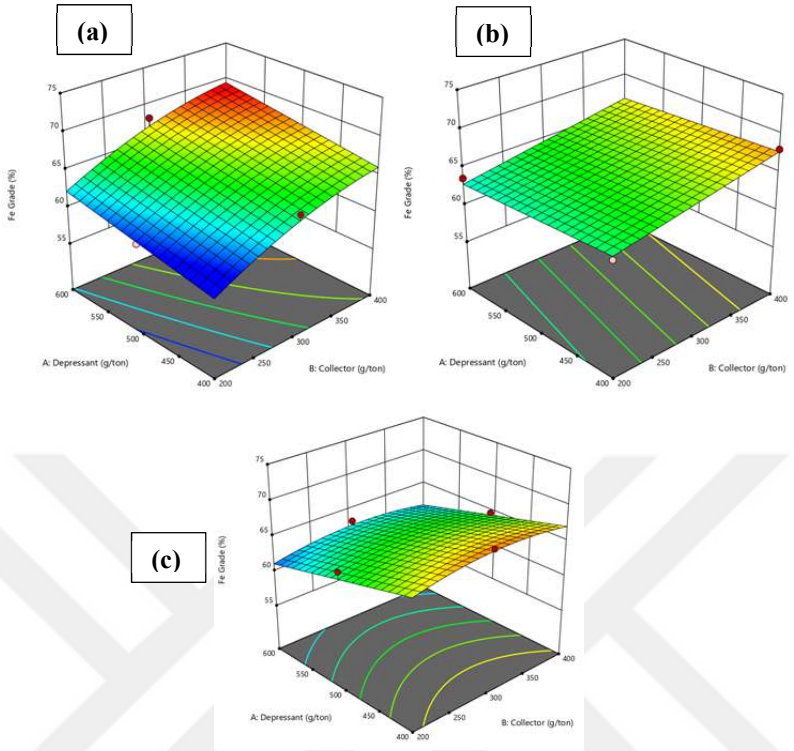

Figure 4.48. Effect of 50 g/ton (a). 100 g/ton (b) and 150 g/ton (c) frother dosages at 3 min froth collection time on iron concentrate grade.

As shown in Fig. 4.49, the effect of 4 min of froth collection time at 50 g/ton, 100 g/ton, and 150 g / ton doses of iron concentrate grade. At 50 g/ton and 100 g / ton doses, the effect of depressant doses on the grade of iron concentrate varied as it increases to medium dosages of depressant and then began to decrease with an increasing depressant, while the iron concentrate grade was increased with the increase of collector doses. At doses over 100 g / ton, the negative effect of depressant doses on the grade of iron concentrate at doses of more than 500 g / ton was noted (Fig. 4.49).

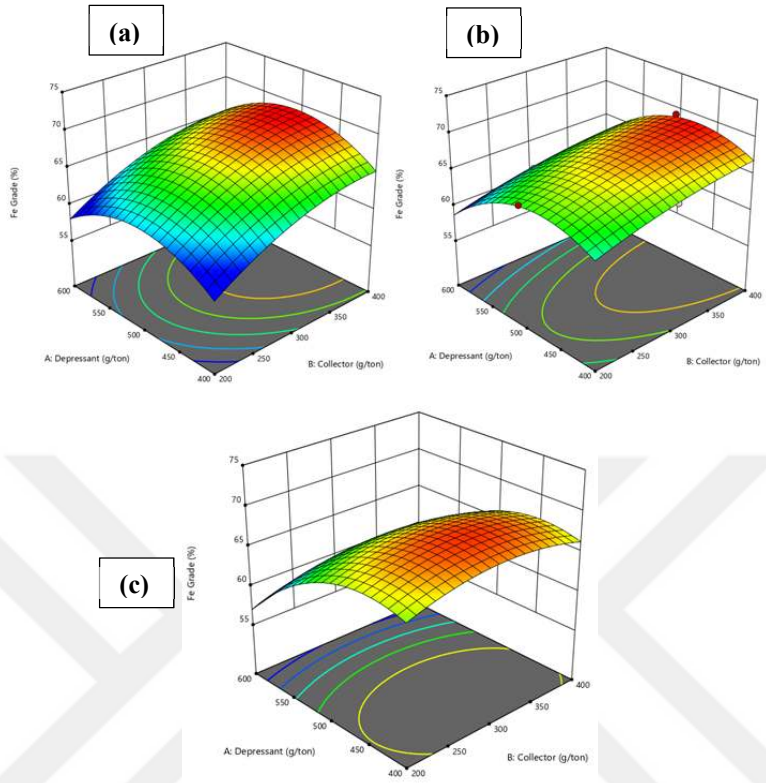

Figure 4.49. Effect of 50 g/ton (a). 100 g/ton (b) and 150 g/ton (c) frother dosages at 4 min froth collection time on iron concentrate grade.

Fig. 4.50. showed the effect of 50 g/ton, 100 g/ton and 150 g/ton frother dosages on the iron concentrate recovery. As shown at the several doses of frother, in general, there is a remarkable varying effect on recoveries of iron concentrate. At 100 g/ton frother dosage, the negative effect of the collector dosages on the iron concentrate recovery remained the same, therefore, the iron concentrate recovery was decreased with increasing collector dosage while the effect of the depressant dosages on the iron concentrate recovery at this frother dosage was not significant. At 150 g/ton frother dosage, iron concentrate recovery reached its highest value of 90.57 % at 500 g/ton depressant dosage, 400 g/ton collector dosage, and 3 min froth collection time.

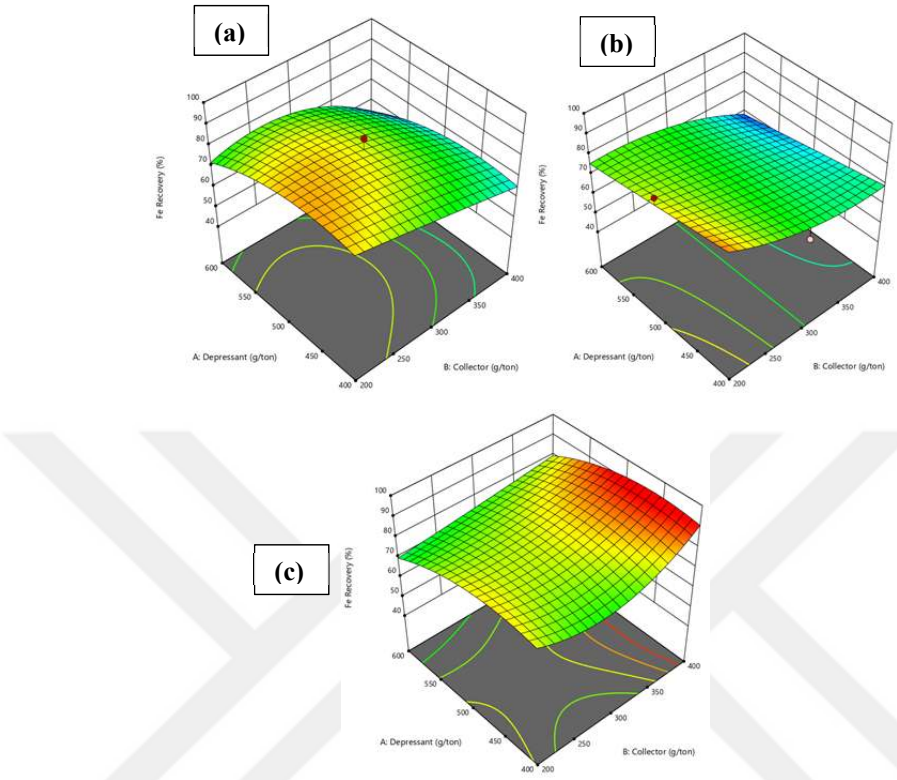

Figure 4.50. Effect of 50 g/ton (a), 100g/ton (b) and 150 g/ton (c) frother dosages on iron concentrate recovery.

In the cationic flotation test, collector dosage had the greatest effect on grade and recovery of the iron concentrate while the effect of the collector dosage and froth collection time was less. According to ANOVA for Reduced Quartic model, the correlation coefficients (R^2) for iron concentrate grade and recovery were calculated as 0.907 (<0.0001 P-value) and 0.75 (0.0007 P-value) respectively. It was showed a good agreement of the generated model with the obtained experimental results for the grade of iron concentrate grade while R^2 for iron concentrate recovery was showed not as good correlation for parameters. The equations obtained from models were as follows:

$$Fe\ (\%) = 65.30 - 0.495\ X_1 + 1.96\ X_2 + 2.22\ X_4 - 2.21\ X_1X_3 - 1.68\ X_2X_3 - 3.99\ X_1^2X_4 - 1.68\ X_1X_4^2 - 0.68\ X_2^2\ X_4 - 1.17\ X_2^2X_3^2 \quad (10)$$

$$Fe\ recovery\ (\%) = 72.82 - 8.47\ X_2 + 6.8\ X_2X_3 + 8.12\ X_3^2 - 5.47\ X_1X_2^2 + 5.74X_2^2X_3 + 6.55\ X_2X_3^2 - 6.15\ X_1^2X_3^2 \quad (11)$$

Where; X_1: depressant dosage (g/ton), X_2: collector dosage (g/ton), X_3: frother dosage and X_4: froth collection time (min).

5. CONCLUSION AND RECOMMENDATIONS

The specularite ore sample was investigated applying shaking tables, Wet Magnetic Separation, Microwave, and Conventional Roasting Followed by Wet Magnetic Separation, Separation of Falcon Concentrator, and Reverse Anionic and Reverse Cationic Flotation methods. The result of the chemical analysis (XRF) of specularite ore showed a high ratio of Fe_2O_3 as well as the high content of silica and calcium oxide. According to experimental results, it was possible to obtain Fe_2O_3 concentrates in the grades that can be used by the industry which can be saleable (>62% Fe) by separating the specularite mineral from the gangues using physical (shaking table, wet magnetic and Falcon concentrator), roasting (microwave and conventional roasting) and physicochemical (reverse anionic and reverse cationic flotation) separation techniques. These results were as follows:

- In shaking table separation tests, iron concentrate grade of 63.47% Fe (with 92.17% recovery) was obtained at the optimum conditions of +74-150 μm particle size, 3° slope angle, 12 L/min water flow rate and 175 strokes/min shaking speed.
- In wet magnetic separation tests, the maximum value of iron concentrate grade was 69.07% Fe (with 22.23% recovery) at 0.25 T, -150 μm particle size and 20% solid/liquid ratio while the highest value of iron concentrate recovery was 67% (with 66.79% Fe grade) at 0.75 T and particle size of -74 μm and 20% solid/liquid ratio.
- In microwave heating following by wet magnetic separation, maximum iron concentrate grade was 68.90 % Fe (with 27.28 % recovery) at conditions of 0.9 KW microwave power, 2 min exposure time, 0.25 T magnetic field intensity, -150+38 μm particle size and 20 % solid/liquid ratio while the maximum iron concentrate recovery was 87.37% (with

66.06% Fe grade) at conditions of 0.72 KW microwave power, 3 min exposure time, 0.85 T magnetic field intensity, -150+38 μm particle size and 20 % solid/liquid ratio.

- In conventional roasting following by wet magnetic separation, the highest value of iron concentrate grade was 69.10 % Fe (with 41.16 % recovery) at conditions of 450 C temperature, 10 min exposure time, 0.25 T magnetic field intensity, -150+38 μm particle size and 20 % solid/liquid ratio while the maximum iron concentrate recovery was 85.95% (with 62.26% Fe grade) at conditions of 650 °C temperature, 10 min exposure time, 0.85 T magnetic field intensity, -150+38 μm particle size and 20 % solid/liquid ratio.
- In Falcon concentrator, 63.93 % Fe (with 68.06% recovery) iron concentrate grade was obtained from conditions of 30 psi pressure, 35 Hz centrifugal force, - 75 μm particle size, and 20 % solid/liquid.
- In reverse anionic flotation test, optimum conditions for the best iron concentrate grade was 63.04% Fe (with 96.85% recovery) and that was at conditions of 5250 g/ton corn starch, 1000 g/ton Aero 343 collector, 600 g/ton MIBC, 2 min froth collection time at pH 12.5 and -106+38 μm particle size while the highest iron concentrate recovery (98.96% with 62.26% Fe grade) was obtained at optimum conditions: 5250 g/ton corn starch, 1500 g/ton Aero 343, 600 g/ton MIBC, 1 min froth collection time and -106+38 μm particle size.
- In reverse cationic flotation method, the maximum iron concentrate grade was 68.95 % Fe (with 81.9% recovery) at conditions of 400 g/ton depressant dosage, 300 g/ton collector dosage, 100 g/ton frother dosage, 2 min froth collection time, 3 pH and -106+38 particle size while the maximum iron concentrate recovery was 90.57 % (with 64.69 % Fe grade) and that was obtained at optimum conditions: 500 g/ton depressant dosage,

400 g/ton collector dosage, 150 g/ton frother dosage, 3 min froth collection time, 3 pH and -150+38 μm particle size.

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- The possibility to obtain a high grade and recoveries of the iron concentrate by applying combination from two methods together (magnetic separation and shaking table separation methods followed by reverse flotation method).
- The possibility to obtain a high grade and recovery of iron concentrate by using combination of collectors in different proportions of dosages in the reverse flotation method.

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