

INVESTIGATION OF THE PRE-CONCENTRATION POSSIBILITY OF
SCANDIUM ORES

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ABSTRACT

INVESTIGATION OF THE PRE-CONCENTRATION POSSIBILITY OF SCANDIUM ORES

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This study was aimed at investigating the possibility of obtaining a scandium (Sc) pre-concentrate from Eskişehir-Mihalıççık scandium bearing clayey ore through mineral processing methods namely attrition scrubbing, enhanced gravity separation by using falcon concentrator, magnetic separation and flotation concentration. In this context, the study began with the characterization of the run-of-mine Eskişehir-Mihalıççık scandium ore by performing mineralogical, XRD and chemical analyses. Mineralogical analysis indicated that the major Sc-bearing minerals clay minerals are montmorillonite (Al, Mg containing, smectite group clay mineral), nontronite (iron-rich, smectite group clay mineral), and also to lesser extent Sc-bearing diopside (Ca, Fe, Mg silicate mineral) and pyroxene (so-called pyroxenite, a rock-forming silicate mineral). Apart from that, calcite (CaCO_3), dolomite ($\text{CaMg}(\text{CO}_3)_2$), and quartz (SiO_2) were classified as the major impurity containing minerals. So in other words, obtaining a pre-concentrate enriched in scandium at the highest possible extent by the removal of gangue minerals, including calcite, quartz, and dolomite were also targeted. In the context of the characterization studies, performed elemental analysis indicated that the ore was relatively enriched in scandium content as 105 ppm by mainly concentrating in the clay minerals such as montmorillonite and nontronite. Also, the ore has relatively higher Fe content as 8.01 % and in terms of impurities, Ca, Mg, and Si grades of the ore were determined as 4.44 %, 5.16 %, and 24.45%, respectively. Based on

both mineralogical and chemical analysis, Eskişehir-Mihalıççık scandium ore was determined as a Sc-bearing clayey ore. At this point, the motivation of obtaining a pre-concentrate is preparation of a feed, relatively enriched in scandium content for increasing the efficiency and effectiveness of subsequent hydrometallurgical processes.

In the context of the pre-concentration studies of Eskişehir-Mihalıççık scandium ore, attrition scrubbing tests were performed initially in order to obtain a pre-concentrate enriched in scandium content by the removal of impurities such as Ca, Mg, and Si. Following the attrition scrubbing tests, operational parameters such as solid-liquid ratio, retention time and rotation speed were encountered using a statistical approach, through a full two level factorial experimental design. As the result of the optimization of the attrition scrubbing test parameters, feed that initially contains 105 ppm Sc., was enriched to 120 ppm at 85.06 % scandium recovery. Moreover, 42.93 % of Ca, 30.88 % of Mg, and 33.73 % of Si were removed in the tailings of the attrition scrubbing tests. Following the optimization of the attrition scrubbing tests, enhanced gravity separation by using falcon concentrator was performed on the previously obtained pre-concentrate in order to provide further pre-concentration and to enrich scandium content. Falcon concentrator tests were investigated in terms of the centrifugal force and fluidization water pressure. In this context, pre-concentrate of the attrition scrubbing experiments that initially contains 120 ppm Sc., was enriched to 138 ppm at 90.17 % scandium recovery. Also, 50.24 % of Ca, 26.65 % of Mg, and 20.14 % of Si were removed in the tailings of the falcon concentrator tests. By combining the results of the applied pre-concentration methods, namely attrition scrubbing and enhanced gravity separation by using falcon concentrator, a pre-concentrate that contains 138 ppm Sc was obtained at 76.70 % Sc recovery. In addition to these, 71.60% of Ca, 49.30 % of Mg, and 47.08 % of Si were removed by the present study.

As an alternative method to the enhanced gravity separation by using falcon concentrator, magnetic separation tests were performed for the further concentration of the pre-concentrate obtained from the attrition scrubbing tests. However, it was seen that the magnetic separation could not achieved its goal and thereby a pre-concentrate that was enriched in scandium could not be obtained as the result of the high intensity

wet magnetic separation. Apart from these, relative enrichment of previously obtained pre-concentrate in terms of scandium through flotation concentration was also attempted as an alternative method. In the flotation tests, two different flotation flowsheet was applied for the flotation of the major clayey components and the flotation of the iron-bearing minerals. As it was observed during the flotation tests, previously obtained pre-concentrate did not respond positively to the applied flotation concentration methods. So, a pre-concentrate that was enriched in scandium was could not be obtained by performing flotation method.

As the result of the present study, the conditions and the parameters of the selected pre-concentration methods namely attrition scrubbing and enhanced gravity separation by using falcon concentrator that would enable the most effective means of scandium pre-concentration was identified and discussed. In addition to these, a flowsheet for the pre-concentration of the Eskişehir-Mihalıççık Scandium ore was proposed.

Keywords: Attrition scrubbing, Falcon concentrator, Scandium, Clayey Ore, Pre-concentration

ÖZ

SKANDİYUM İÇERİKLİ CEVHERLERİN ÖN-ZENGİNLEŞTİRİLEBİLME OLANAKLARININ BELİRLENMESİ

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Bu çalışmada skandiyum içeren killi mineraller bakımından zengin Eskişehir-Mihalıççık cevherinin ön-zenginleştirme yöntemleri kullanılarak skandiyum (Sc) bakımından zenginleşmiş bir ön-konsantre ürünü elde edilme olanaklarının araştırılması amaçlanmıştır. Ön-zenginleştirme işlemleri kapsamında, aşındırmalı yıkama, santrifüjli gravite ayırma, manyetik separasyon ve flotasyon olmak üzere dört farklı metot denenmiştir. Başlangıçta, tüvenan Eskişehir-Mihalıççık skandiyum cevherinin karakterizasyon çalışmaları hem mineralojik (XRD) hem de kimyasal analiz teknikleri kullanılarak gerçekleştirilmiştir. Mineralojik analizler sonucunda başlıca skandiyum içeren minerallerin montmorillonite (Al, Mg içeren smektit gurubu kil minerali) ve nontronit (Fe açısından zengin smektit gurubu kil minerali) olduğu gözlemlenmiştir. Ayrıca, cevherdeki skandiyumun az da olsa diopside (Ca, Fe, Mg silikat) ve piroksen (kayaç oluşturan mineral gurubundan, piroksenit) gibi silikat minerallerinden kaynaklandığı görülmüştür. Cevherdeki majör gang mineralleri kalsit (CaCO_3), dolomit ($\text{CaMg}(\text{CO}_3)_2$) ve kuvars (SiO_2) olarak belirlenmiştir. Bir başka deyişle, mevcut çalışma ile kalsit, dolomit ve kuvars gibi cevherin içerdiği safsızlıkların atılarak, skandiyum bakımından zenginleşmiş bir ön konsantre elde edilmesi hedeflenmiştir. Karakterizasyon çalışmaları kapsamında yapılan kimyasal analiz sonuçlarına göre, cevherin 105 ppm Sc içeriğine sahip olduğu ve genellikle nontronit, montmorillonite gibi kil minerallerinde zenginleştiği saptanmıştır. Ayrıca,

cevherin % 8.01 Fe, % 4.44 Ca, % 5.16 Mg ve % 24.45 Si içeriğine sahip olduğu görülmüştür. Yapılan karakterizasyon çalışması sonucunda, cevherin Sc-içeren killi bir cevher türü olduğu saptanmıştır. Söz konusu ön-konsantre ürününün elde edilmesine yönelik en önemli motivasyon, takip eden hidrometalürjik işlemler için, bu işlemleri olumsuz etkileyebilecek gang minerallerinin mümkün olduğunca bertaraf edildiği ve aynı zamanda skandiyum içeriği bakımından görece zenginleşmiş bir besleme malzemesi elde edilmesidir.

Eskişehir-Mihalıççık Skandiyum cevheri üzerinde yapılan ön-zenginleştirme çalışmaları kapsamında, öncelikle aşındırmalı yıkama metodu kullanılarak Ca, Mg ve Si gibi impüritelerin uzaklaştırılması ile skandiyum açısından zengin bir ön-konsantre elde edilmesi amaçlanmıştır. Ön-testler sonrasında elde edilen veriler doğrultusunda, katı-sıvı oranı, rotasyon hızı ve aşındırmalı yıkama süresi gibi operasyonel parametreler faktöriyel dizayn yöntemi kullanılarak optimize edilmiştir. Optimum parametreler ile yapılan aşındırmalı yıkama testleri sonucunda, başlangıçta 105 ppm Sc içeriğine sahip besleme malzemesinden 120 ppm Sc içeren ön-konsantre % 85.06 Sc verimi ile elde edilmiştir. Bunlara ek olarak, besleme malzemesinin içerdiği Ca'nın % 42.93'ü, Mg'nin % 30.88'i ve Si'nin % 33.73'ü atık malzemede uzaklaştırılmıştır. Aşındırmalı yıkama testleri sonucunda elde edilen ön-konsantre, skandiyum açısından görece daha zengin bir ön-konsantre elde edebilmek amacıyla falcon santrifüjlü ayırıcıya beslenmiştir. Bu bağlamda, falcon konsantratör testleri, santrifüj kuvveti ve su basıncı parametreleri açısından değerlendirilerek optimize edilmiştir. Tespit edilen optimum parametreler ile yapılan testler sonucunda, başlangıçta 120 ppm Sc içeriğine sahip olan besleme malzemesinden 138 ppm Sc içeriğine sahip ön-konsantre % 90.17 Sc verimi ile elde edilmiştir. Bunlara ek olarak, besleme malzemesinin içerdiği Ca'nın % 50.24'ü, Mg'nin % 26.65'i ve Si'nin % 20.14'ü atık malzemede uzaklaştırılmıştır. Mevcut durumda, uygulanan ön-zenginleştirme yöntemleri sonucunda, 105 ppm Sc içeren cevherden 138 ppm Sc içeren ön-konsantre % 76.70 Sc verimi ile elde edilmiştir. Bunlara ek olarak, tüvenan cevherin içerdiği Ca'nın % 71.60'ı, Mg'nin %49.30'u ve Si'nin 47.08'i uzaklaştırılmıştır.

Falcon konsantratör kullanılarak uygulanan santrifüjlü ayırma yöntemine alternative olarak daha önce aşındırmalı yıkamadan elde edilen ön-konsantreye manyetik

separasyon uygulanmıştır. Ön-konsantrenin yüksek alan şiddetinde yaş manyetik separasyona tabi tutulması sonrasında, manyetik separasyonun amacına ulaşamadığı ve bu bağlamda skandiyum açısından zenginleşmiş bir ön-konsantre elde edilemediği görülmüştür. Bunlara ek olarak, aşındırmalı yıkama testlerinden elde edilen ön-konsantreye majör kil minerallerinin ve demir içeren minerallerin yüzdürülmesi hedeflenerek iki farklı flotasyon akım şeması uygulanmıştır. Flotasyon deneyleri esnasında da görüldüğü üzere, cevher flotasyon metoduna olumlu tepki vermemiştir ve bu sebeple skandiyum açısından zenginleşmiş bir ön-konsantrenin flotasyon metodu kullanılarak elde edilemeyeceği görülmüştür.

Mevcut çalışma ile Eskişehir-Mihalıççık skandiyum cevherinden skandiyum açısından zengin bir ön-konsantre elde edilmesi amacıyla uygulanan ön-zenginleştirme metotlarından aşındırmalı yıkama ve falcon konsantratörün en etkin metotlar olduğu görülmüştür. Optimum operasyon parametrelerinin belirlenmesi sonucunda cevherden skandiyum açısından zenginleştirilmiş bir ön-konsantre elde edilebilmesi için uygun akım şeması önerilmiştir.

Anahtar Kelimeler: Aşındırmalı yıkama, Falcon konsantratör, Skandiyum, Killi cevher, Ön-zenginleştirme

To My Beloved Family

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CHAPTER 1

INTRODUCTION

1.1 General

To comprehend the discovery and importance of metallic elements, it is necessary to take a glance at the past, to era of the creation of mankind. Creation of mankind and necessity of maintaining their life, brought along with number of needs. Many of these mentioned needs were fulfilled by means of metallic elements. If the importance of metallic elements needs to be examined closely, the crucial importance of them could be understood from the names of Ages, such as Copper Age and Iron Age. Apart from iron and copper, by the discovery of other metallic elements, mankind satisfied their needs and developed. Metallic elements, which played an essential role in the emergence and development of many industries, also laid the foundations of modern society.

In this day and age, science and technology develops relevantly and rapidly at modern societies. With these developments, metallic elements become more and more important. Not only metallic elements but also rare earth elements are increasing in their importance by their key roles in various industries. They are called as ‘Seeds of Technology’ by Japanese and ‘Technology Metals’ by the US Department of Energy. Also, rare earth elements were identified as both strategic and critical elements by United States Geological Survey (USGS), because they are currently used for the production of more than 200 technological products regarding various industries. They are commonly used in the production of cellular phones, computer hard drives, electric and hybrid vehicles, flat screen monitors and televisions as key components. In addition to these, they are also essential in military electronics industries for the production of electronic displays, guidance systems, lasers, radar and sonar systems

(Ishee *et al.*, 2013). Concordantly, by taking a glance at the rare earth elements, they can be defined as a set of seventeen chemical elements in the Periodic Table, which includes fifteen lanthanides plus scandium (Sc) and yttrium (Table 1.1).

Table 1.1 Periodic Table of the elements (Bigler, 2017)

The periodic table displays elements from Hydrogen (H) to Oganesson (Og). Key features include:

- Periods:** 1 to 7, with Lanthanides and Actinides shown below the main table.
- Groups:** I A to VIII A, with III A to VII A also labeled.
- Color Coding:**
 - Gases:** Yellow background (e.g., H, He, Ne, Ar, Kr, Xe, Rn, Og).
 - Liquids:** Blue background (e.g., Hg, Br).
 - Metalloids:** Pink background (e.g., B, Si, As, Sb, Te, Po, At).
 - Rare Earth Metals:** Yellow background (e.g., Sc, Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu).
- Legend:**
 - atomic # →
 - atomic symbol →
 - English element name →
 - ions commonly formed
 - atomic mass (rounded)

Scandium, which is known as the lightest rare earth element, was first predicted in 1869 by Mendeleev as an unknown element called eka-boron with an estimated atomic weight of 45 (Horovitz *et al.*, 1975). It was discovered in 1879 by Lars Frederik Nilson, who is a professor of analytical chemistry, during an extraction work of rare earths from gadolinite. Minerals containing this new element were originally found in Scandinavia; therefore it was named as scandium after its discovery (Borisenko, 1961). In early days of its discovery, physical and chemical properties of scandium could not be defined due to the rare occurrence of this element. As an early investigation, scandium mineral was distinguished from other rare earths by its weak basicity, molecular weight, which is less than 131 and its spark spectrum. Although scandium has common properties with rare earth elements, it has many properties similar to beryllium. This situation at that time brought a controversy among scientists whether scandium is rare earth or not. In progress of time, developments were made in investigations about physical and chemical properties of scandium. Based on these

developments, some scientists stated that scandium is a rare earth element by supporting the following issues (Horovitz *et al.*, 1975);

- Its cyanoplatinate complexes are similar to those of cerium and yttrium earths.
- It is found with rare earths and obtained from rare earth minerals.
- Its sparingly soluble double alkali sulphate is similar to those of cerium earth elements.

On the contrary, there were also scientists who were in opposition to the ideas mentioned above;

- Unlike other rare earth elements, scandium has light atomic weight.
- Scandium has a hexahydrated sulphate compound which is very soluble in water, like beryllium sulphate.
- Scandium and beryllium have many common properties.

While these discussions were taking place among scientists, the scandium minerals thortveitite, bazzite, kolbeckite, sterretite and schetelig were found. Although most of the features of this mysterious element had not been known for a certain period of time, its features have been discovered recently. As the 50th most abundant element in the Earth's crust, scandium has a critical importance both in terms of occurrence with rare earth elements and its industrial usage.

The above mentioned industrial significance of scandium emerges at many points in daily life, for example, scandium products and alloys are used in aircrafts, baseball bats, bicycle frames, solid oxide fuel cells, and stadium lightning. Examining the utilization areas of scandium in more detail indicated that it has many utilization areas owing to its physical and chemical properties. It is mainly used in metallurgical, ceramic, catalytic, electronic, lightning, and nuclear areas (Youngblood and Gschneidner, 1975). In addition to these, scandium alloys can be used in both aircraft and military industries. In metallurgical applications, scandium is an effective alloying

element and used for refining the grain size of aluminum, spheroidizing graphite, and lowering the oxidation rate of light lanthanides. As well as its metallurgical usage, scandium is used as an alloying agent in ceramic applications. As a catalyst, scandium is used in dehydrogenation of isopropyl alcohol, cyclohexane, and dehydration of isopropyl alcohol. In electronics, scandium can be used for adjusting magnetic properties of materials. Also, chloride, nitrate, and oxide complexes of scandium are used in lightning applications. Nuclear usage of scandium is mostly related with its radioactive isotope ^{46}Sc , which is used as a common tracing agent. Because of its alloy with aluminum is strong and light, scandium is used in aircraft industry. Also, it is used in solid oxide fuel cells owing to its electrical conductivity and heat stabilization qualities.

Although scandium has such important utilization areas, its production rate and number of producers are unfortunately limited. The reasons for this situation are the absence of easily recoverable deposits and low concentration of this element in the Earth's Crust (Baroch, 1960). On the other hand, scandium is a dispersed element in the lithosphere and generally produced as a by-product or co-product of primary metal processes and reprocessing tailings. Therefore, there is not any identified single deposit for scandium. Previously identified scandium resources are located in Australia, Canada, China, Kazakhstan, Russia, Ukraine, Madagascar, Norway, the Philippines, and the United States (U.S. Geological Survey, 2017). Annually, 10 to 15 tons of scandium is supplied and consumed worldwide. Majority of scandium is extracted in China and the most important products of scandium are scandium oxide, scandium fluoride, and scandium metal. Due to the scarcity and low production rate of scandium products, there is not any known market for scandium today. Most of scandium products are sold between private parties at undisclosed prices. Prices of scandium products can vary according to the quality, volume, availability, source of product, and current demand. Prices for scandium compounds and metal scandium can be found in reports of U.S. Geological Survey (USGS) (Table 1-2).

Table 1.2 Scandium product prices with respect to years (U.S. Geological Survey, 2018)

<i>Year</i>	<i>2013</i>	<i>2014</i>	<i>2015</i>	<i>2016</i>	<i>2017</i>
<i>Scandium (Metal) (\$/gr)</i>	213	221	221	228	226
<i>Scandium Acetate (\$/gr)</i>	51.90	43	43	44	44
<i>Scandium Chloride (\$/gr)</i>	148	123	123	126	124
<i>Scandium Flouride (\$/gr)</i>	253	263	263	270	277
<i>Scandium Iodide (\$/gr)</i>	228	187	187	149	183
<i>Scandium Oxide (\$/kg)</i>	5000	5000	5100	4600	4600
<i>Scandium-Aluminum Alloy (\$/kg)</i>	155	386	220	340	350

Current utilization areas and importance of scandium reveal the truth that scandium has an even more important place in the near future. Then, the production rate of scandium, which cannot fully meet the current demand, needs to be increased rapidly. With the improvement of the production rate, it can be thought that the scandium product prices will be reduced to normal levels and thus the demand is expected to increase. Associated with the increased usage of scandium, the technological developments will be accelerated and occurrence of important scientific studies will be noticed. In addition to these, it will contribute significantly in terms of economics.

1.2 Objectives and Scope of the Study

Dispersed state of scandium in the Earth's crust and scarcity of high grade scandium deposits restricts scandium production. In addition to these, high processing costs of processing methods have also a deterrent effect on scandium production. In the present case, the need for a lower cost production is obvious. It is believed that the way, for lowering the current higher costs, passed through the mineral processing operations rather than costly metallurgical methods. Also, it is considered that the mineral processing operations performed before the metallurgical processes can reduce the processing costs and prepare a relatively enriched scandium pre-concentrate.

The objective of this study is determining the possibility of obtaining a scandium (Sc) pre-concentrate. The motivation of obtaining a pre-concentrate is the preparation of a feed which is relatively enriched in scandium content for increasing the efficiency and effectiveness of subsequent hydrometallurgical processes. Through pre-concentration, the rejection of gangue minerals that would cause negative impacts during hydrometallurgical treatment, at the highest possible extent, is also targeted. Pre-concentration work will focus on the application of vigorous attrition-agitation as well as use of various concentration procedures such as gravity concentration techniques, magnetic separation, etc. Relative enrichment of scandium through flotation of the major clayey components will also be attempted. For each technique, the conditions and parameters that would enable the most effective means of scandium pre-concentration will be determined and discussed.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 Scandium

2.1.1 Discovery and History

In the early years of the discovery of the Periodic Table, there were missing gaps related with atomic weights 45, 68 and 70. Respectively, these gaps were defined as eka-boron, eka-aluminum and eka-silicon by Mendeleev. At that time, these gaps gained the attention of many scientists and during a period of ten years eight elements were discovered. In 1879, after ten years from the discovery of the Periodic Table, Lars Frederik Nilson, who is an analytical chemistry professor, discovered the element scandium when working on the mineral gadolinite (Horovitz *et al.*, 1975). Nilson remarked different properties of this new mineral from lanthanons such as its weak basicity, molecular weight which is less than 131, and its spark spectrum.

Initially, Nilson was unable to define the complete chemical properties of this new element due to the small amount obtained from this new element. As a starting point, he assumed that it was a tetravalent element but then as a result of extensive chemical analysis, he found that it is a white oxide which is hardly soluble in nitric acid, more soluble in hydrochloric acid and different from other rare earth elements, also its nitrate complexes decompose at relatively low temperatures unlike other rare earth elements. Based on these properties, it was understood that this new element fitted well to the empty square of the Periodic Table and its properties are compatible with properties previously identified by Mendeleev. The name of the element, scandium, comes from Scandinavia where the minerals containing this new element were originally found (Horovitz C. T., 1999).

By means of discoveries about scandium mentioned above, interest on the scandium element had increased for a certain period of time. While Nilson had been studying the element, on the other hand, French chemist Per Theodor Cleve started to deal with scandium and extracted 0.8 g. of it from 4 kg. gadolinite, 1.2 g of it from 3 kg of kielhaute. Both Nilson and Cleve analyzed scandium in greater details and Nilson found that it is trivalent. With this finding, Nilson and Cleve were sure that eka-boron, which was identified by Mendeleev, was the element scandium (Table 2.1).

Table 2.1 Properties of eka-boron predicted by Mendeleev and properties identified for scandium by Nilson and Cleve (Horovitz *et al.*, 1975)

Characteristics of Eka-Boron (Eb) predicted by Mendeleev	Characteristics of Scandium observed by Nilson and Cleve
Atomic weight is 45.	Atomic Weight is 44.
Oxide of Eka-Boron (Eb_2O_3) has a specific gravity of 3.5 and it is more basic than alumina, less basic than yttria or magnesia. Eka-Boron Oxide is insoluble in alkalies and may not decompose NH_4Cl	Oxide of Scandium (Sc_2O_3) has a specific gravity of 3.86 and it is more basic than alumina, less basic than yttria or magnesia. Scandium Oxide is insoluble in alkalies and does not decompose NH_4Cl .
Crystallization of its salt compounds is difficult and appears colorless. Its precipitates with alkalies are gelatinous.	Crystallization of its salt compounds is difficult and appears colorless. Its precipitates with alkalies are gelatinous. Sulphate compound of it crystallizes hardly.
Carbonate compound of it would probably be a basic salt and insoluble in water.	Scandium Carbonate [$\text{Sc}_2(\text{CO}_3)_3$] is insoluble in water and loses CO_2 easily.

Table 2.1 Cont'd

Double sulphate compounds of it would not be alums.	Double sulphate compounds of it are not alums.
Anhydrous EbCl_3 compound would be less volatile than AlCl_3 . Aqueous solution of it could be more easily hydrolyzed than MgCl_2 .	Sublimation of ScCl_3 starts at 850°C , but it is 100°C for AlCl_3 . Also, hydrolysis of aqueous solutions are higher.
It would not be discovered spectroscopically.	It was not discovered spectroscopically.

After the discovery of scandium and its limited properties, it did not draw attention by chemists and other scientists for more than twenty years. At the beginning of the twentieth century, many of rare earth metals including scandium started to be examined closely. In the context of these studies, in 1908, Eberhard found that scandium was present in more than eight hundred different mineral species. He also mentioned that scandium is concentrated in euxenite, columbite, micas, ytrotitanite, and some other minerals with higher amounts. Based upon the studies made by Eberhard, Vernadskii stated that scandium, which concentrated in rare earth crust, is transferred with outflows of acidic magmas and dispersed in bodies formed in pneumatolytic stage of their solidification. Therefore, scandium was classified as typically dispersed element such as gallium, indium, rubidium, and germanium (Borisenko, 1961).

In the light of these developments, in 1911, the first true scandium mineral thortveitite was found by Schetelig and after four years from this discovery, Artini found another scandium mineral, namely bazzite, which is scandium beryl. By the discovery of scandium minerals, studies about scandium accelerated and these studies were followed by the studies about the geochemistry of scandium at the beginning of 1930's. In 1930, W. and J. Noddack stated that the abundance of scandium in the earth crust is 6 ppm. Following that, Goldschmidt and Peters found the isomorphous replacement of scandium ion by ferrous and magnesium ions. In 1937, Fischer et. al. obtained the

metallic scandium for the first time by electrolyzing the melt of lithium, potassium and scandium chlorides. Because of the obtained metallic scandium contained a little amount of impurities, Petru et. al. produced high purity scandium in 1956 for the first time. Also, scandium was used as a geological thermometer by Oftedal, due to the increasing scandium content of biotites through basic to acidic rocks (Horovitz *et al.*, 1975).

Discoveries and developments about scandium continued until the beginning of the twenty first century and continue with regular intervals at the present time. Full historical review on the discovery and developments about scandium is given in the following table (Table 2.2).

Table 2.2 Historical review on the discovery and developments about scandium (Horovitz C. T., 1999)

Year	Event	Reference
1869	Prediction of an unknown element eka-boron	Mendeleev
1879	Discovery of scandium in gadolinite and euxenite	Nilson
1879	Preparation of 1.2 grams of scandium oxide	Cleve
1898	Estimation of lithosphere abundance of scandium	Vogt
1908	Patent for separation of scandium from minerals	Meyer
1909	Determination of scandium in the solar system	Fowler
1911	First true mineral of scandium Thortveitite discovered	Schetelig
1914	Scandium β -diketonate [Sc(acac) ₃] prepared	Morgan
1923	Atomic mass of scandium measured definitely	Aston
1924	Determination of magnetic resonance lines of scandium	Goudsmit and Zeeman
1925	Determination of scandium in plant material	Von Lippman
1932	Calculation of diamagnetic susceptibility of scandium	Van Vleck and Angus

Table 2.2 Cont'd

Year	Event	Reference
1935	Identification of scandium isotopes	De Hevesy
1937	Preparation of metallic scandium	Fischer et al.
1938	Determination of scandium in animals	Lux; Noddack
1939	Bone micrographs with scandium X-rays	Dershem
1946	⁴⁶ Sc become publicly available	Ames Laboratory
1947	Inhibition of thromboplastic effect	Chargaff and Green
1948	Using ⁴⁶ Sc as environmental tracer	Arrol
1959	Synthetically Thortveitite produced	Petru et al.
1961	High purity scandium metal produced	Daane
1962	Binary alloys of scandium studied (with Be, Bi, Cd, Ce, Mn, Po, Tc, Ti, Tl, U)	Gschneidner Jr.
1963	Preparation of single scandium crystals	Savitsky et al.
1966	Identification of ⁵¹ Sc isotope	Erskine et al.
1972	Scandium ion is used in ion microprobe mass analysis	Guthrie and Blewer
1973	Preparation of 99.9 purity atomic scandium	Spedding and Croat
1982	⁴⁶ Sc labelled for MAb tumor imaging	Scheinberg et al.
1996	Sc chelate conjugated MAb injected to a patient	Scheinberg

2.1.2 Utilization and Production

Scandium is a widely dispersed element in nature; therefore it has no identified single source. It is mainly produced by processing of different ores, tailings and residues as a by-product. Most of scandium is produced from thortveitite, which is a scandium-yttrium silicate mineral and from uranium processing as a by-product. Also, scandium can be produced from different resources such as tantalum, molybdenum, titanium-tungsten, aluminum, kolbeckite, and variscite process residues. In addition to these, lower grade domestic sources for scandium production are aluminum, cobalt, iron,

nickel, phosphate, tin, zinc, and zirconium deposits (U.S. Geological Survey, 2010). China, which is known for its interest in rare earth elements and scandium, has many ore deposits and produces scandium mainly from ionic clays or titanium oxide processing (Ricketts and Duyvesteyn, 2017). Main scandium sources are located in Australia, China, Kazakhstan, Madagascar, Norway, Russia, Ukraine and the USA (U.S. Geological Survey, 2010).

For investigating the importance of scandium, it is necessary to examine the usage areas of scandium. Industrial usage of this element dates back to old times, when Soviet Union existed. The first nation, which figured out the importance of scandium and used in industry, is the Soviet Union. Discovery of the usage of scandium in Soviet aircraft industry, created reactions in the United States and led to the exploration of scandium resources (Ross and Rosenbaum, 1962). Due to the superior chemical and physical properties of scandium, by means of its light and durable nature, its alloys were used by the Soviet Union and U.S. Air Force in aircraft and ballistic missiles in the Cold War of 1960's, but low abundance and high processing cost restricted its use at those years (Usuda *et al.*, 2011). Scandium, with many features that have been explored, has started to play a key role at many areas recently. Nowadays, scandium has three main utilization areas, these are aerospace industry, solid oxide fuel cells, and electronics industry. But also it is used in various applications in terms of metallurgical, ceramic, electronic, lightning and nuclear areas.

One of the most important application areas of scandium is metallurgy. It is known as an effective alloying agent. For strengthening magnesium, scandium is used with other alloying elements. Also, scandium is used to strengthen aluminum and straighten the grain size of aluminum. Aluminum, which is alloyed with small amounts of scandium, approximately 0.2 %, has excellent properties in terms of grain refining without making any heat treatment (Ricketts and Duyvesteyn, 2017). In other words, scandium addition to aluminum provides high corrosion resistance, heat tolerance, superior mechanical and welding properties (Duyvesteyn and Putnam, 2014). Based on these beneficial effects of scandium, its aluminum alloys naturally attracts aircraft

manufacturers. They believe that scandium aluminum alloy both reduce the product weight and also it has a potential to reduce the processing cost. Therefore, scandium aluminum alloys are generally used in the area of aerospace industry and in non-conventional designed sport equipment such as bike frames, baseball bats and hand guns (Venkateswarlu *et al.*, 2008). Though it is newer than other application areas, scandium aluminum alloys are started to be used in 3D printing with respect to its high strength, moldability, and weldability. By considering the above mentioned effects of aluminum scandium alloys, following benefits for the industrial usage and technological developments can be mentioned (Duyvesteyn and Putnam, 2014);

- Reduced processing costs
- Potential of reducing material weights
- Longer product life and sustainability
- Creating a market for scandium production and trade at trustworthy prices
- Creating innovative technological improvements.

Spheroidizing effect of scandium on graphite is another usage of scandium in metallurgical applications; it makes the graphite particles larger than those formed by magnesium and rare earth additions (Khudokormov and Komarov, 1963). Also, due to the corrosive resistance of scandium, Sc-M (where M = Sb, As, Bi, Cr, Pb, Mn, Mo, Ni, Nb, Ta, Th, Sn, W or V) alloys increases the resistance of zirconium. Corrosion resistance of zirconium scandium alloy was observed at 350 °C for water and 540 °C, 40 atm. for steam (Misch and Van Drunen, 1961). In addition to these, scandium additions lower the rate of oxidation of light lanthanides.

Another utilization area of scandium is electronics, which includes solid oxide fuel cells, computer memory cores, high voltage tension wires and computer monitors. In this context, scandium is more popularly used in solid oxide fuel cells. Before giving any information about the usage of scandium in solid oxide fuel cells, it would be more accurate to define solid oxide fuel cell and its advantages briefly. Fuel cells can be thought as the converter of a fuel source and oxygen source into an electric current by

conducting reactions between the fuel and oxidant (reactants) via the help of a high temperature environment. Also, water, carbon dioxide and heat can be obtained as co-products. By briefly stating the structure of solid oxide fuel cells, it consists of a solid electrolyte, which is made from hard ceramic material, squeezed between an anode and a cathode for the separation of reactants. Benefits and advantages, which are provided by solid oxide fuel cells in our daily life, can be summarized as (Duyvesteyn and Putnam, 2014);

- Providing higher efficiencies than conventional production methods by reaching 85 %
- Cleaner production method than conventional methods by lower emissions
- Providing operating flexibility and mobility
- Thanks to its design, economic fuel consumption of solid oxide fuel cell provides economic savings.

In conventional solid oxide fuel cells, yttrium-stabilized zirconium (yttria) is used as the material of anode and cathode parts. Due to the high operating temperatures of these cells, there exists thermal fatigue, oxidation problem of metal components and also safety measures at that much of high temperatures have high operating costs (Duyvesteyn and Putnam, 2014). Therefore, reactants in solid oxide fuel cells play an important role both in terms of efficiency and economics of the process. The use of scandium instead of yttria, which has a better ionic electrical conductivity and higher heat resistance, provides higher power density at lower temperatures, by this way it reduces the cost of materials for thermal shielding and increases the service life of the cell (Duyvesteyn and Putnam, 2014). In other words, usage of scandium in solid oxide fuel cells, which have zirconium oxide electrolyte already reduces the operating temperature to approximately 150 °C and increasing both the operating efficiency and safety as well (Ricketts and Duyvesteyn, 2017).

In the past, scandium contained ferrites were used in rapid switching computer memory cores. Addition of scandium to ferrites of magnesium-manganese lowers the magnetic induction (Youngblood and Gschneidner, 1975). Also, addition of trace amount of scandium into yttrium iron garnet causes the substitution of iron and scandium by which magnetic moment and permeability increase as well the Curie temperature decreases (Cunningham, 1965). Another utilization of scandium in electronics industry is the usage of scandium oxide in combination with garnet ferrites, which increases the magnetic moment and therefore they are commonly used in microwave applications. As the last utilization of scandium in electronics industry, its compound with gadolinium, gallium, and garnet are used in the production of laser material (Duyvesteyn and Putnam, 2014).

As the dispersed state of scandium, its utilization areas also show dispersion through different branches. In ceramic applications, scandium is used for two main reasons. Firstly, addition of scandium to ceramic material directly affects the strength of the material. Secondly, due to the higher heat resistance and thermal shock properties, it is contained in glass compositions for flame spray coatings (Youngblood and Gschneidner, 1975). In addition to these, the multiline spectra of scandium iodide compound in the vapor phase enable its usage in high intensity lights. At the same time, these lights simulate the natural sunlight directly related with its scandium content. In our daily life, many of these lights can be seen in stadiums and studios. As an activator and host for phosphor, scandium is used in color television receivers (United States of America Patent No. US 3010909 A, 1961). Lastly, isotope of scandium ^{46}Sc is used in nuclear applications as a tracing agent. Because of its high affinity to silica-alumina catalysts, it is commonly used in refinery cracks. Additionally, by adsorbing ^{46}Sc on the clay suspensions, their movement has been already studied in the past.

Scandium, with the above mentioned such important utilization areas, has just started to attract attention and studied by industrial companies. Also, another reason for the ongoing scandium projects is the insufficient production rate with respect to current

demand (Ricketts and Duyvesteyn, 2017). For these reasons, it was thought that providing information about companies and projects working on scandium will be a useful literature review for future researchers. Industrially, scandium was first produced by the Russians during the Cold War period. The first production of scandium was made from Nova Mine, which mainly contains iron ore and located in the Zhovti Vody Town of Ukraine (Duyvesteyn and Putnam, 2014). Scandium, which had attracted interest in America after the production of Russia, is now being studied by many countries, including China, Australia, Canada and European. Current scandium production and studies are more focused on titanium, tungsten, uranium residues, red mud, phosphogypsum, lateritic nickel-cobalt ores, and ion-adsorbed type clay deposits. In this context, general information about scandium projects and contractor companies of these projects will be given below.

- **Scandium International Mining Corp. & Scandium Investments LLC**

Scandium International Mining Corp. and Scandium Investments LLC have made an association agreement recently. They have two projects called the Nyngan and Honeybugle Scandium Projects. As the first and the foremost project, deposit of Nyngan Scandium project is located approximately 450 kilometers of northwest of Sydney. According to feasibility studies, mine life was calculated as 20 years and there are 5,690,000 tonnes of measured resource associated with 11,230,000 tonnes of indicated resource based on 100 ppm scandium cut-off grade. Nyngan Scandium Project is exclusively focused on scandium, which makes this project different from the previous and current projects. In addition to these, the head grade for scandium was determined as 409 ppm (Lycopodium, 2016). The proposed production process generally consists of metallurgical processes, including high pressure acid leaching and solvent extraction. Production flowsheet issued by the company is given in Figure 2.1.

As can be seen from the production flowsheet of Nyngan Scandium Project, it consists of scrubbing, screening, milling, cycloning and thickening in terms of mineral processing operations. With the completed feasibility studies, the pilot work of the Nyngan Scandium Project is still continuing (Kaya, 2016). The second project, which is called Honeybugle Scandium Project, is located near the Nyngan Deposit. As the result of the first stage of the conducted drilling studies, the head assay was determined as 270 ppm scandium. Also, drilling studies at the area are still continuing.

- **Clean TeQ Holdings Ltd.**

Syerston Nickel Cobalt Scandium Project is owned by Clean TeQ Holdings Ltd. Deposit and is located at 350 kilometers northwest of Sydney. According to the feasibility and pre-studies, the mine life was calculated as 20 years and there are 5,800,000 tonnes of measured resource associated with 15,900,000 tonnes of indicated resource based on 300 ppm scandium cut-off grade (Clean TeQ Holdings Ltd., 2016). The Head grade of scandium was determined as 583 ppm, which makes this mine the deposit with the highest scandium grade (Ricketts and Duyvesteyn, 2017). The proposed production process generally consist of hydrometallurgical processes, including high pressure acid leaching and resin in pulp ion exchange chromatography. Production flowsheet issued by the company is given in Figure 2.2.

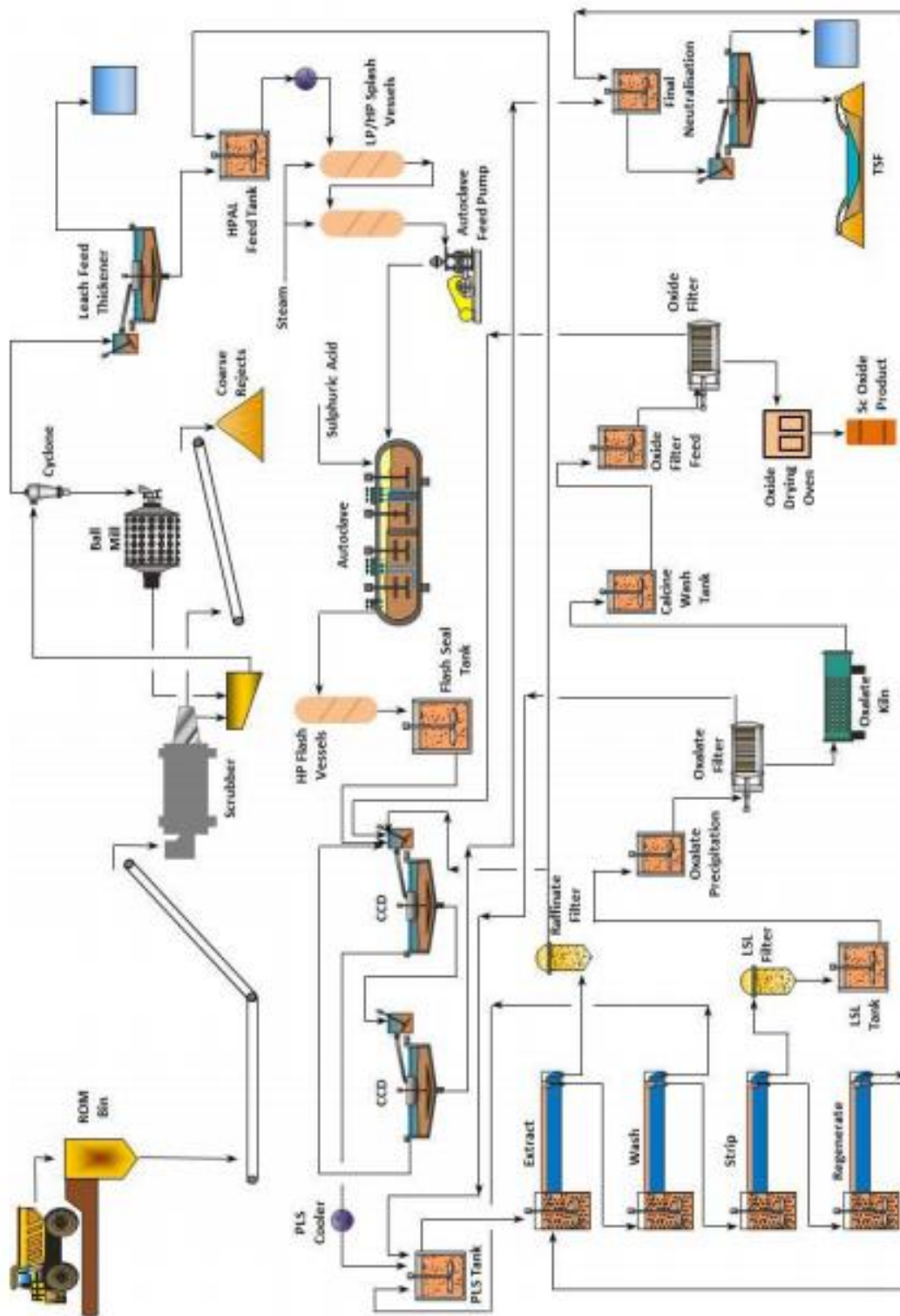


Figure 2.1. Production flowsheet of Nyngan Scandium Project (Lycopodium, 2016)

Within the scope of the project, nickel and cobalt, which are originally contained in the run-of-mine ore, will be extracted as by-products. Also, the prominent part of the project is that ion exchange chromatography is being planned to be used instead of conventional solvent extraction process and the project showed that ion exchange chromatography could be an alternative way to solvent extraction process. In the light of these developments, pilot studies of the project are still continuing.

- **Platina Resources Ltd.**

Owendale Scandium-Platinum Project is owned by Platina Resources Ltd. The deposit is located 80 kilometers northwest of Parkes and 350 kilometers west of Sydney. In terms of geology, the deposit type is lateritic and contains nickel, cobalt and platinum apart from scandium. The Owendale Project is at an earlier stage compared to the other projects mentioned above and mineral resource estimation of the site has recently been completed. According to the mineral resource estimation studies, there are 4,400,000 tonnes of measured resource associated with 6,400,000 tonnes of indicated resource based on 300 ppm scandium cut-off grade (Platina Resources Limited, 2017).

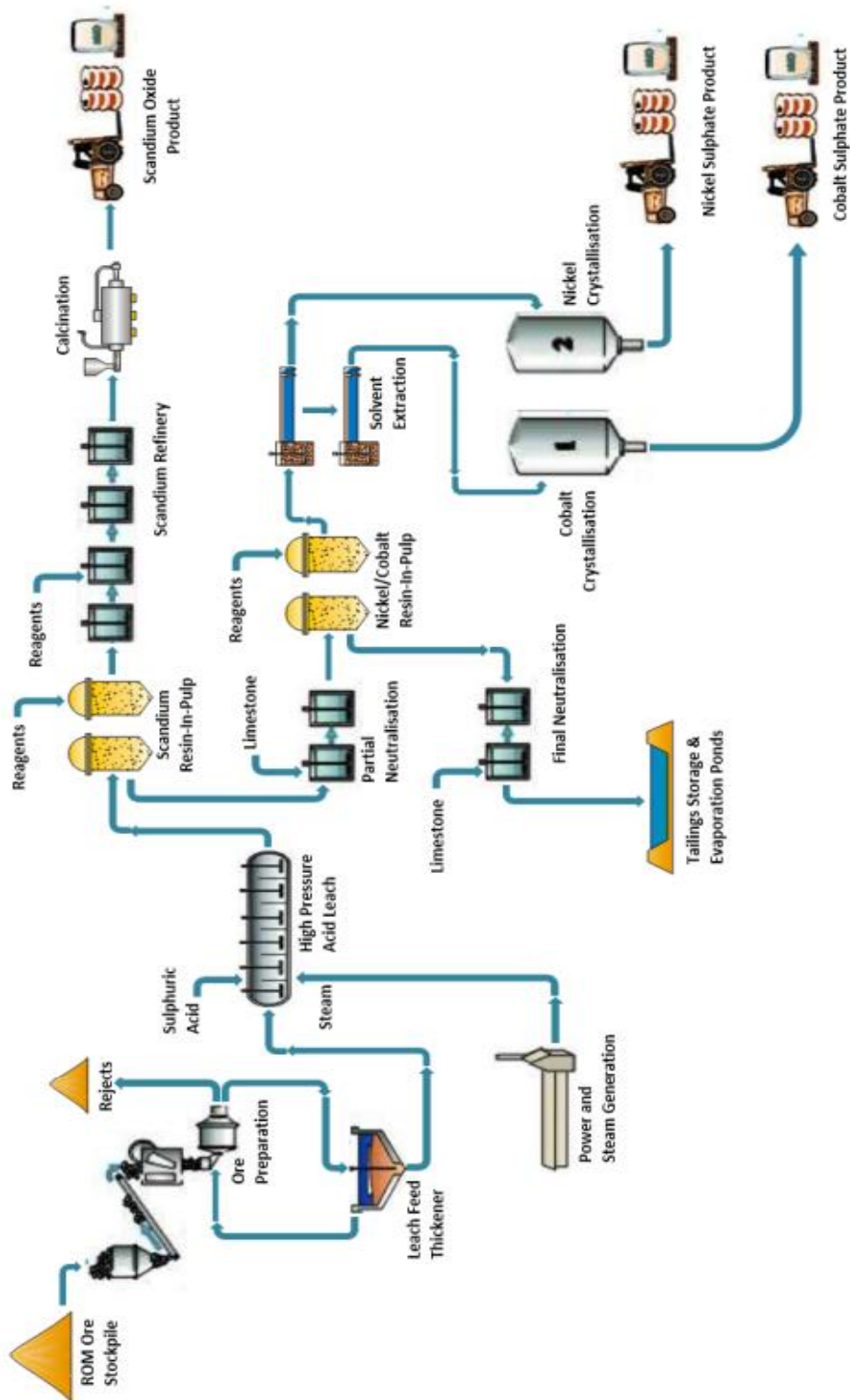


Figure 2.2. Production flowsheet of Syerston Nickel-Cobalt-Scandium Project (Clean TeQ Holdings Ltd., 2016)

The head grade of scandium was determined as 384 ppm (Ricketts and Duyvesteyn, 2017). Cobalt, nickel and platinum is being planned to extract as by products. According to the Company's statement, drilling studies were continuing in 2017 and also the feasibility study will be continued until the beginning of 2018.

- **Metallica Minerals Ltd. & Australian Mines Ltd.**

ScONi Scandium-Cobalt Project is carried out by partnership of Metallica Minerals Ltd. & Australian Mines Ltd. Project mainly contains five different deposits which are named as Greenvale, Lucknow, Bell Creek, Minnamoolka, and Kokomo. These deposits are located 250 kilometers northwest of Queensland. Pre-feasibility study for the deposits has been completed recently (Australian Mines Limited, 2016). According to the pre-feasibility study, the head grade of scandium was determined as 208 ppm at 120 ppm scandium cut-off grade. (Ricketts and Duyvesteyn, 2017). Companies are willing to extract cobalt and nickel as by-products and also three of the deposits are relatively enriched in terms of nickel and cobalt, the other two deposits are relatively enriched in terms of scandium and cobalt. At the present moment, the final feasibility study is being carried out by the Australian Mines and the drilling studies are still in progress (Australian Mines Limited, 2016).

- **Jervois Mining Ltd. & Australian Mines Ltd.**

Flemington Scandium-Cobalt-Nickel-Platinum Project is carried out by the joint venture of Jervois Mining Ltd. & Australian Mines Ltd. The deposit is located 450 kilometers west of Sydney, near Syerston Project of Clean TeQ Holdings Ltd. (Australian Mines Limited, 2016). Scoping study of the project has been completed by Australian Mines Ltd. According to this scoping study, head grade of scandium was determined as 450 ppm at 300 ppm scandium cut-off grade. Also, the companies are willing to extract cobalt, nickel, and

platinum as by products (Ricketts and Duyvesteyn, 2017). Information about the progress of the Flemington Project is kept confidential at the moment.

- **Sumitomo Metals Mining Company Ltd.**

Sumitomo Metals Mining Company Ltd. has two nickel-cobalt plants, Coral Bay Nickel Plant and Taganito Nickel Plant, in Philippines. Due to the occurrence of scandium in nickel-cobalt mixed sulfide ores, the company decided to establish a pilot plant in Taganito Nickel Plant for the extraction of scandium (Sumitomo Mining Company Limited, 2016). It is known that the main process will include high pressure acid leaching (HPAL), ion exchange and solvent extraction, but detailed information about the project has not been disclosed yet (Kaya, 2016).

- **Meta Nikel Kobalt A.Ş.**

Meta Nikel Kobalt A.Ş. has two nickel-cobalt deposits in Manisa and Eskişehir, Gördes Deposit and Yunusemre Deposit. Gördes Deposit is located approximately 115 kilometers from Manisa and Yunusemre Deposit is located approximately 100 kilometers from Eskişehir. Also, the company has a nickel-cobalt plant in Gördes, which includes high pressure acid leaching process. Due to the occurrence of scandium in lateritic nickel-cobalt deposits, the company is planning to extract scandium as a co-product by taking advantage of the available nickel-cobalt plant. According to the stated information, the production route of the company includes sulfuric acid re-leaching of the second neutralization underflow material (second iron precipitation) precipitated at pH equals to 4.75 (Ricketts and Duyvesteyn, 2017). Within the scope of this project, Meta Nickel Cobalt Company is aiming to produce both scandium fluoride and scandium oxide end products and also pilot studies are continuing. On the other hand, one of the main objectives of the company is to be the first scandium producer from lateritic type deposits using MHP route for

nickel and cobalt production (Kaya, 2016). However, detailed information about the ongoing studies has not been announced yet.

- **Russian Aluminum Company (RUSAL)**

Russian Aluminum Company has bauxite mines and industrial tailings of these mines, which are also known as red mud containing scandium as well. The company has been working on scandium extraction at its aluminum smelter located at Ural Mountains since 2013. According to the press releases, the company has produced 99 % purity scandium oxide as the product of the installed pilot plant. Also, the company is planning to use this product at its aluminum production process as an alloying agent (Russian Aluminum Company, 2017).

- **NioCorp Developments Ltd.**

NioCorp Developments Ltd. has a niobium project, which is called Elk Creek Niobium Project, located in Nebraska. The deposit is located approximately 75 kilometers southeast of Lincoln and 100 km south of Omaha. The deposit also contains scandium and titanium together with niobium (Srk Consulting, Roche Ltd., 2015). According to the feasibility study, the head grade for scandium was determined as approximately 73 ppm. Although the studies about scandium production are still continuing, the company stated that they managed to complete a small scale production of scandium oxide in July 2016, in terms of small scale (Ricketts and Duyvesteyn, 2017). There is no further information available about scandium production of the company for now.

Due to the fact that scandium is a critical metal, many projects about scandium production have been made. Also, it can be seen that companies have directed their way to such deposits, which contain scandium. As a result, the main objective of all of the above mentioned companies and their projects is, producing a sellable scandium

product and taking a place in the global market. It is thought that the demand for scandium is directly related with the technological developments and by the technological improvements in future, the demand for scandium will increase rapidly. For this reason, the possible increase in the number of projects on scandium production will not be surprising.

2.1.3 Physical and Chemical Properties

Scandium is located at the third group and the fourth period of the Periodic Table, contiguous to the lanthanides and the actinides, as the first and lightest transition metal. Therefore, it has many similar physical and chemical properties with lanthanides and actinides. The physical and chemical properties of scandium are given in Table 2-3.

Table 2.3 Physical and chemical properties of scandium

Property	Value
Atomic Number	21
Atomic Weight (g)	44.9559
Density (g/cm³)	2.989
Ionic Radius (Å)	0.745
Metallic Radius (Å)	1.6406
Melting Point (°C)	1541
Boiling Point (°C)	2836
Electronegativity (eV)	1.30

As a soft and silvery metal, scandium is readily tarnished in air. By the way, its location in the Periodic Table and electronic configuration provides some extraordinary properties for this metal. Similar to other rare earth elements, three outer valence electrons of scandium beyond the argon rare gas core make chemical and physical properties of this element similar to rare earth metals. Also, the atomic volume curve shows that scandium is located between two peaks which belong to potassium and cobalt. The most important feature of the elements like K, Ca, Ti, V, Cr, and Mn, which are located in the same region, is their lithophilic structure. For this reason, scandium is also a lithophilic element. In addition to these, such elements such as yttrium,

zirconium, rare earth elements, thorium, and uranium have lithophilic structure (Borisenko, 1961). The atomic volume curve and the structures of elements are given in Figure 2.3.

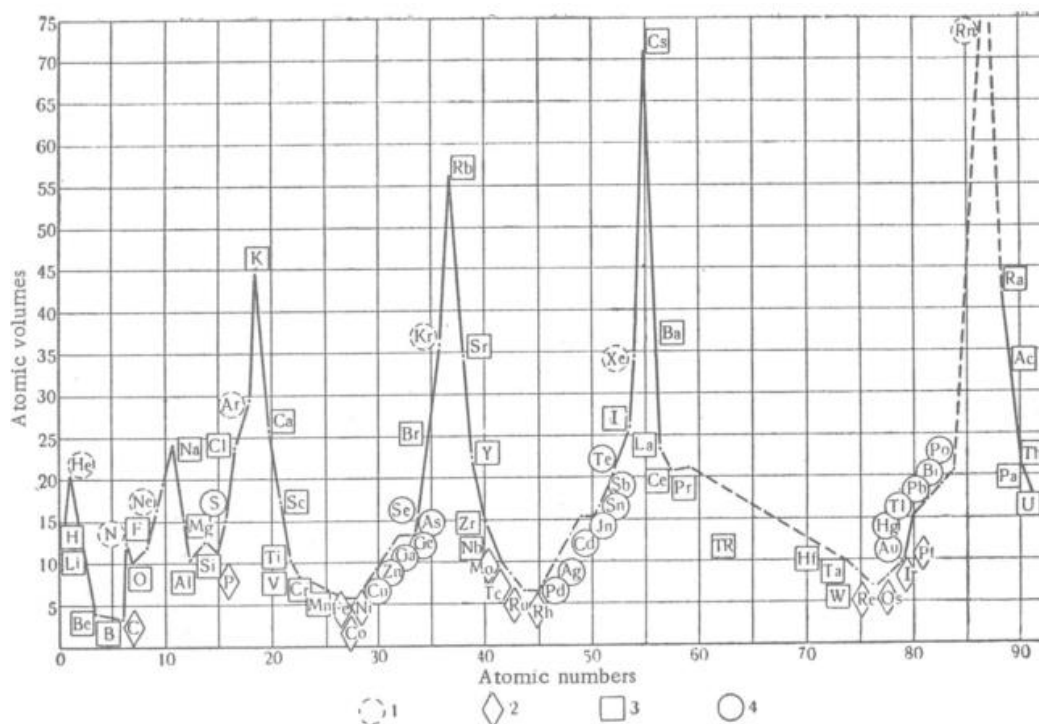


Figure 2.3 Atomic volume curve of the elements: 1: Atmophilic 2: Siderophilic 3: Lithophilic 4: Chalcophilic (Borisenko, 1961)

However, there are also substantial differences between the properties of scandium and other rare earth metals. For instance, scandium has higher melting and boiling points than other rare earth metals (Gschneidner, 1975). Also, its electronegativity and ionic radius are the other distinctive properties of scandium. It has a relatively smaller ionic radius, which resembles to ferrous ions and higher electronegativity than other rare earth metals (Teatum *et al.*, 1959). The ionic radius and electronegativity value for scandium, iron, magnesium, and manganese are given in Table 2.4.

Table 2.4 Ionic radius and electronegativity values for scandium, magnesium, iron and manganese (Borisenko, 1961)

Ion	Ionic Radius (Å)	Electronegativity (eV)
Mn ²⁺	0.80	1.40
Fe ²⁺	0.74	1.65
Mg ²⁺	0.66	1.20
Sc ³⁺	0.745	1.30

Based on these ionic radius and electronegativity values, it was found that dispersion of scandium in ferromagnesian minerals at the early stage of formation of magmatic rocks, is directly related to the similarities between ionic radiuses of scandium and ferrous ions. Due to the difference between ionic radius and electronegativity of scandium and magnesium, it was expected that scandium has much more tendency to get in to lattice structure of ferrous ions (Ringwood, 1955). At this stage, scandium and iron make ion exchange (substitution) and scandium enters to the structure of the rock as in the dispersed state.

Besides the physical properties of scandium, it can be thought as a chemically active element by means of its smaller ionic radius. Therefore, it forms strong complexes with both anions and natural ligands, despite its extensive hydrolysis in solution (Melson and Stotz, 1971). In addition to these, scandium dissolves as the result of the reaction with dilute mineral acids and also it reacts with many typical metalloids (Borisenko, 1961). Based on the contained anions, scandium compounds can be examined in three categories, these are; (Melson, 1975)

- Oxygen containing compounds
- Halides
- Nitrogen containing compounds

Some of the oxygen containing compounds of scandium are, scandium oxide [Sc₂O₃], scandium hydroxide [Sc(OH)₃], scandium carbonate [Sc(CO₃)₃], scandium nitrate [Sc(NO₃)₃], scandium pyrophosphate [ScHP₂O₇.3H₂O], scandium sulfate [Sc₂(SO₄)₃], scandium selenide [Sc₂Se₃], and scandium halite [Sc(ClO₄)₃]. As the first and foremost

scandium compound, scandium oxide is a refractory white powder and is insoluble in water. Its basicity, which is lesser than the rare earth compounds, is higher than aluminum oxide. Scandium hydroxide is known for its amphoteric properties and weakly basic character with solubility of $5 \cdot 10^{-7}$ mole/liter in water (Borisenko, 1961). Also, scandium carbonate was managed to be produced by addition of ammonium or sodium carbonate to scandium solution, but its properties have not been studied properly yet. Scandium nitrate compounds are highly hygroscopic and are prepared from aqueous solution of scandium chloride and dinitrogen pentoxide (Melson, 1975). Also, phosphor is a strong chelating agent for scandium, thus scandium pyrophosphate has high stability and low solubility both in water and dilute mineral acids. As the last well known oxygen containing compound, scandium sulfate can be produced in anhydrous form and it is a hygroscopic white crystalline substance.

Second category for scandium compounds are scandium halides, which are scandium fluoride [ScF_3], scandium chloride [ScCl_3], scandium bromide [ScBr_3], and scandium iodide [ScI_3]. First of all, scandium fluoride, which is still playing an important role in the industry, is known as a stable white crystalline compound. It may form complex acids such as H_3ScF_6 by reacting with solutions of hydrofluoric acid, potassium, sodium and ammonium fluorides. Another halide compound of scandium is scandium chloride, which is a hygroscopic white crystalline compound and has a melting point of 939°C , sublimates between 800°C and 850°C . The dissolution of scandium chloride in water and hydrochloric acid solutions is easy and part of hydrochloric acid discards for the formation of basic chlorides, such as ScOCl by the result heating. These basic chlorides of scandium are insoluble in water and resistant to alkalis and acids (Borisenko, 1961). Typically, both scandium bromide and iodide have analogous properties with scandium chloride.

Nitrogen containing compounds of scandium are explained as the last category of scandium compounds. These are scandium with ammonia compounds and scandium with thiocyanate compounds. As a result of the adsorption of ammonia by scandium oxalate tri-hydrate or hexahydrate at 25°C , scandium ammonia compound

[Sc(NH₃)₆]₂(C₂O₄)₃] is formed (Vickery, 1955). According to the studies about scandium thiocyanate complexes, [Sc(NCS)]²⁺ was found as the most stable scandium thiocyanate compound. Also, scandium can be extracted into diethyleter from the aqueous solution of thiocyanate (Melson, 1975). Table 2.5 is given for the purpose of summarizing the variety of scandium compounds.

Table 2.5 Scandium compounds (Horovitz C. T., 1999)

Compound Name	Formula
Scandium Boride	ScB ₂
Scandium Bromide	ScBr ₃
Scandium Carbonate	Sc ₂ (CO ₃) ₃
Scandium Chloride	ScCl ₃
Scandium Fluoride	ScF ₃
Scandium Hydride	ScH ₃
Scandium Hydroxide	Sc(OH) ₃
Scandium Iodide	ScI ₃
Scandium Nitrate	Sc(NO ₃) ₃
Scandium Nitride	ScN
Scandium Oxide	Sc ₂ O ₃
Scandium Selenide	Sc ₂ Se ₃
Scandium Sulfate	Sc ₂ (SO ₄) ₃
Scandium Sulfide	Sc ₂ S ₃
Scandium Telluride	Sc ₂ Te ₃

2.1.4 Geology, Geochemistry and Mineralogy

Geology, geochemistry, and mineralogy of scandium, which has such important utilization areas, chemical and physical properties as a rare earth element, had been intensively investigated by many researchers. By these investigations, it was found that scandium is a typically dispersed element in nature as the 50th most abundant element with 20 to 30 ppm average concentration in the Earth's crust. As mentioned earlier, scandium is a lithophilic element, which concentrates close to the surface and readily combine with oxygen, due to the trivalent form of the element at its compounds (Schock, 1975). Therefore, it is not surprising that scandium mainly occurs in

meteorites, the sun, the moon earth atmosphere, waters, soils, oils, coals and fertilizers and also as a result of the wide distribution of this element, it may be present in many minerals at trace amounts. Geology of this valuable element had been investigated even on the moon and samples taken from the moon surface had been analyzed for examining the scandium content. During these investigations, great tendency of scandium to igneous rocks rather than basalts, breccias, and fines was explored (Annell and Helz, 1970). In addition to these, scandium, which was even encountered in surface air, shows a correlation with iron, aluminum, chromium and airborne dust by the following calculated correlation coefficients of 0.94 for iron, 0.91 for aluminum, 0.86 for chromium and 0.87 for airborne dust (Brar *et al.*, 1970). Also, researches about the occurrence of scandium in waters shows that scandium was concentrated in deep waters and this discovery proves the relation of scandium with silicates. The occurrence state of scandium in surface and near surface waters, which have low acidity or low basicity, is generally thought to be taken place as the result of the transportation of the element by adsorption or complexes (Horovitz C. , 1975). According to the studies made about scandium amounts of soils, concentration of scandium in soils, which is covering the basic rocks, is higher than soils covering the acidic rocks. This finding has been proved with respect to the higher content of scandium in basic and ultrabasic rocks rather than acidic rocks (Viinogradov, 1959). For the scandium content of soil samples, the correlation of scandium with iron, cobalt, europium, and samarium was observed. As the result of this observed correlation, the following correlation coefficients were calculated, these are 0.96 for cobalt, 0.80 for iron and 0.89 for europium (Horovitz C. , 1975). Based on these values, it can be said that scandium directly follows cobalt, iron, europium, and samarium during the deposition of soils. On the other hand, the relationship between scandium and clay was observed, as the result of the studies on soil samples, with a correlation coefficient of 0.79 and also it was presumed that this correlation was based on the scandium iron correlation in the slime. Further to these, it was determined that scandium followed the clay fraction of soils during the leaching trials by using hydrochloric acid (Kline *et al.*, 1969). Finally, the occurrence of scandium in coal deposits was identified and the concentration of scandium varies between 3 to 300 ppm for ash samples taken from

bituminous and brown coals (Horovitz C. , 1975). Later, the chemical analysis made on low ranking coals of USA showed that, scandium content was varying between 1 and 1000 ppm (Deul and Anell, 1956).

Scandium, as a dispersed element, is present in many different formations and concentrated in many minerals. Its average concentration is more than gold, silver, and bismuth, however scandium does not form any independent single deposit (Borisenko, 1961). Because of this situation, scandium bearing minerals can be present in deposits with different genetic types. In the light of the researches, it was mentioned that scandium presents in rocks, which are related with the early stages of magmatic differentiation and after the magmatic differentiation stage, scandium can also be concentrated in pegmatites, pneumatolytic-hydrothermal deposits (Schock, 1975). Prior to explaining the occurrence of scandium in these deposits, it is necessary to mention the genetic classifications of them. Pegmatite and pneumatolytic-hydrothermal type deposits can be grouped under endogenous processes which were formed by forces within the Earth. Sedimentary type deposits can be grouped under exogenous processes which were formed by forces on or above the Earth's surface. The genetic classification of scandium-bearing deposits is given in Table 2.6.

Table 2.6 Genetic classification of scandium-bearing deposits (Borisenko, 1961)

Endogenic Deposits	Exogenic Deposits
Pegmatite deposits	Sedimentary deposits
Pneumatolytic Hydrothermal deposits	- Placer deposits
- Albitized granite deposits	- Sorptive deposits
- Greisens & Quartz Veins	• Sandstones
- Skarn deposits	• Clays
- Carbonatite deposits	• Variegated sandstones
- Davidite mineralization in old complexes	

In the case of pegmatite type scandium bearing deposits, they generally have relation with granites (biotite-microcline granites) excluding carbonatite type deposits. This relation comes from the intrusion of granitic magma during diversified periods. Scandium bearing pegmatites can be found in two forms, these are pegmatites with rare elements and natrolithium pegmatites (Fersman, 1939). For pegmatites with rare elements, typical scandium bearing minerals are Thortveitite, Euxenite, Khlopinite, Obruchevite, Ampangabeite, Samarskite, Fergusonite, Xenotime, Orthite, Gadolinite, and Crytolite. Also, Cassiterite and Columbite are typical scandium bearing elements for natrolithium pegmatite deposits. Enrichment of scandium in such ores is directly related with the digestion of scandium via the mother melt of enclosed gabbro and amphibolite (Borisenko, 1961). Despite all, small dimensions of pegmatite deposits and lower content of scandium bearing minerals in them make these deposits industrially challenging for the extraction of scandium.

Pneumatolytic hydrothermal scandium bearing deposits can be divided into five different groups. Scandium bearing albitized granite deposits are directly related with acidic rocks, which generally contain columbite and zircon as main scandium bearing minerals. And also the presence of niobium and zirconium in such deposits, makes these deposits more important (Borisenko, 1961). Occurrence of scandium in greisen and quartz vein deposits, which have wolframite, cassiterite, beryl, and mica as the main scandium bearing minerals, is directly related with the intrusions of acidic magma. The higher amount and uniform distribution of scandium in wolframite and cassiterite, make these types of deposits industrially important. Scandium bearing skarn deposits have rarely seen and scandium is generally enriched in muscovite and ferrimuscovite minerals. Even though these deposits have not been industrially operated yet, the large scale of these deposits will make them promising resources of scandium. Appearing as the only different deposits, carbonatite deposits are associated with ultrabasic alkaline rocks. They contain calcite, ankerite and dolomite, as the main minerals with scandium bearing pyrochlore, baddeleyite, pyroxene, and olivine. It was seen that scandium is relatively enriched in pyroxenite minerals of carbonatite deposits (Borisenko, 1961). As the last type of scandium bearing pneumatolytic-hydrothermal

deposits, davidite mineralization in old complexes can be given. Scandiums' are present in davidite mineral however together with the occurrence of uranium and thorium it can be seen as the main challenge of these deposits during the processing and extraction of scandium.

As the scandium bearing exogenic deposits, sedimentary type deposits can be grouped into two main types, these are placer and sorptive sedimentary deposits. In case of placer deposits, accumulation of scandium, which is already presented in cassiterite, xenotime, zircon, garnet, wolframite, baddeleyite, pyrochlore, and etc., in placers are related with the effect of hypergenic conditions. The sorptive sedimentary deposits can be considered fewer than under three subgroups and the first of them is called sedimentary deposits of sandstones. For these deposits, it is known that scandium is present in rhabdophanite as an isomorphous impurity (Borisenko, 1961). Second type of sedimentary deposits is the clay deposits, by which scandium is mostly related with fish bone residues. It was predicted that scandium enriched in these residues originated from the phosphor content. Except those, it was known that scandium may be present in various types of sandstones within minor amounts. Also the relationship of scandium with iron and manganese is another remarkable point for sedimentary deposits. By considering these above mentioned specifications for scandium bearing sedimentary deposits, they may be economically important both in terms of scandium content and other rare earth metal content.

Recently, the depletion of nickel-cobalt sulfide deposits has made lateritic nickel-cobalt deposits more important. Briefly, lateritisation is a geological process of continuous physical, chemical and biological weathering occurring during millions of years (Kaya, 2016). At first, these deposits had drawn attention because of their nickel and cobalt contents but now they are more noticeable due to their scandium content. Lateritic deposits have a layered profile and can occur in three different formations as clay silicate based, hydrous magnesium silicate based, and oxide based. The overlying clayey material is followed by the relatively enriched goethitic limonite. Saprolite, which is found in the lower layer, is known as the scandium bearing layer. Pyroxenite

layer under those mentioned, is followed by the bed rock layer, which is relatively enriched due to the weathering processes (Ricketts and Duyvesteyn, 2017). The formation of laterites begins from serpentinite rocks and especially for the deposits, which have not completed their serpentinization stage, serpentinite minerals are the first products of weathering process (Butt and Cluzel, 2013). Such laterite deposits have also been observed in Turkey and are known as sub-lateritic deposits. Scandium grade of such deposits in Turkey varies between 30 and 130 ppm. It has been observed that scandium concentrated in laterites is generally found together with elements such as Fe, Cr, Al and Co. This situation comes from the integration of scandium with siderophile elements. From a geochemical point of view to the occurrence of scandium in laterite deposits, it was observed that the majority of scandium was adsorbed on aluminum-goethite, while the other part of scandium was entering the crystal structure of hematite by making substitution with Fe^{3+} (Chasse *et al.*, 2016). Also, the concentration of scandium in iron-rich pyroxenes is another case. For that case, enrichment of scandium in clinopyroxenite group, which covers a group of minerals such as diopside, aegirine, augite, and hedenbergite, was observed. As a summary, the presence of scandium in lateritic deposits may be associated with three different cases as the concentration of parent rock, long-term alterations in tectonic environment and lateritic conditions that provide the necessary medium during weathering process for the substitution of scandium with iron (Chasse *et al.*, 2016). Recent discovery of lateritic deposits in Australia, which contains high amounts of scandium, reaching up to 700 ppm Sc grade, such as Syerston, Owendale, Nyngan, and Flemington deposits, is expected to change the scale of scandium production by making these types of deposits as promising resources of scandium.

Ion adsorbed clay deposits, which are more commonly discovered than existing Sc-bearing deposits, are easier to operate both in terms of processing operations and operating costs despite their low content of scandium and rare earth elements. These deposits are generally found in China, but also regions with subtropical climate conditions possibly have them. Ion adsorbed clay deposits, namely weathered crust elution deposits, are formed as the consequence of physical, chemical, and biological

weathering of rare earth enriched granitic and volcanic rocks of subtropical regions under warm, humid and diluted acidic conditions (Bao and Zhao, 2008). Formation process of rare earth element and scandium bearing ion adsorption clay deposits are given in Figure 2.4. During the weathering of such deposits, corresponding to a long geological process, aluminosilicate minerals, which are promising in terms of their high ion exchange capacity, are formed. Such examples of these aluminosilicate minerals are Kaolinite, Illite, Halloysite, and Smectite (Papangelakis and Moldoveanu, 2014). Following the formation of aluminosilicate minerals, as the positive hydrated ions, scandium and other rare earth elements are adsorbed on the clay minerals. On the other hand, as the result of weathering at sedimentary deposits, scandium may be transported, redeposited or sorbed on clay minerals (Schock, 1975). This process occurs due to the negative charging of clay surfaces during isomorphous substitution of cations such as Al, Si, or Mg (Meunier, 2005). In addition to all these, ion adsorbed clay deposits can be thought as an important resource of scandium due to the low radioactive element content and high amount of reserves.

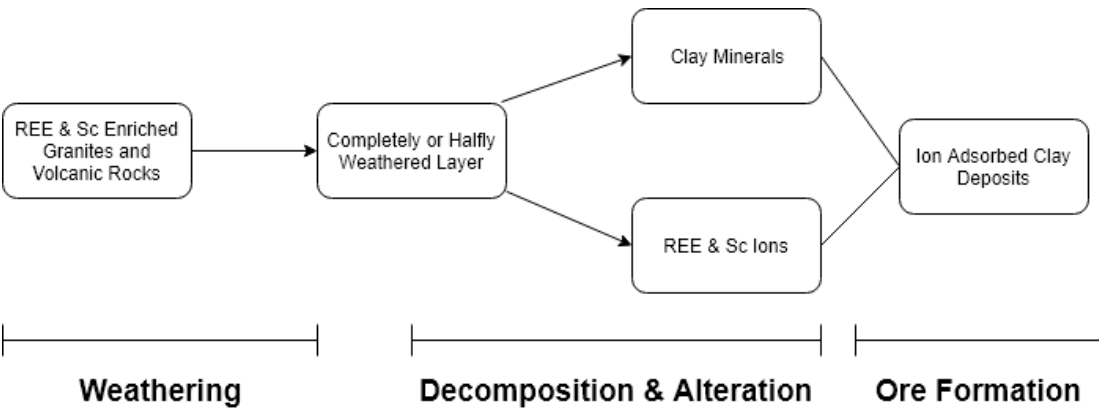


Figure 2.4 Formation mechanism of ion-adsorbed clay deposits modified from (Papangelakis and Moldoveanu, 2014)

From a geological and mineralogical point of view, although the crustal abundance of scandium is more than elements such as gold, silver, and bismuth, its minerals are formed as the result of formation processes occurred under exceptional geological conditions. Therefore, these minerals, namely true scandium minerals that are

relatively enriched in terms of their scandium content, are rarely found in the earth's crust. Major minerals containing scandium are given in Table 2-7. As an element with higher crustal abundance than lead, scandium is widely dispersed through the earth's crust in more than 100 minerals (U.S. Geological Survey, 2017). By making a general classification to occurrence forms of scandium bearing minerals, they are mainly found in the forms of oxides, tungstates, molybdates, carbonates, phosphates, and silicates (Borisenko, 1961). Among these, scandium is mostly found in oxide and silicate formations, which contains ferromagnesian and dark colored minerals such as ferrous iron, magnesium, manganese, calcium, zirconium and other rare earth elements. It was shown that the scandium content of dunite and olivine group minerals is lower with respect to pyroxenites which are their counterpart dark colored mineral group (Goldschmidt and Peters, 1931). Diopside, as one of the most common member of pyroxene group, contains relatively higher amounts of scandium (Devore, 1955). On the other hand, relatively higher scandium contents in lateritic deposits directly are related to the accumulation of scandium, as the result of substitutions with elements like Al^{3+} and Fe^{2+} , in kaolinite, takovite and gibbsite (Fe containing) during the weathering of bed rocks, namely peridotites and serpentinites (Maksimovic, 1958).

Table 2.7 Major minerals of scandium

<i>Mineral Name</i>	<i>Formula</i>
Thortveitite	$Sc_2Si_2O_7$
Bazzite	$Be_3Sc_2Si_6O_{18}$
Kolbeckite	$(Sc, Be, Ca)(Si, P)O_4 \cdot 2H_2O$
Sc-Ixiolite	$(Ta, Nb, Sn, Mn, Fe, Sc, \dots)_2O_4$
Sc-Perrierite	$(Ce, La, Ca)_4(Fe^{2+}, Sc)(Ti, Fe^{3+})_2Ti_2[Si_2O_7]_2O_8$
Magbasite	$KBa(Al, Sc)Fe^{2+}Mg_5F_2Si_6O_{20}$
Sterretite	$Sc(PO_4) \cdot 2H_2O$
Pretulite	$ScPO_4$
Allendeite	$Sc_4Zr_3O_{12}$
Jervisite	$(Na, Ca, Fe^{2+})(Sc, Mg, Fe^{2+})Si_2O_6$

As previously mentioned, both enrichment of scandium in iron containing minerals, due to the siderophilic origin of scandium, and presence of it in more than 100 minerals are directly related with its geochemical properties. The introduction mechanism of scandium to scandium-bearing minerals is chemical substitutions, including isomorphous replacement, isovalent replacement, and heterovalent replacement. Heterovalent substitution of scandium with iron and magnesium in parent rocks, such as pyroxene, olivine, and amphibole, can also be observed in zircons, as the substitution of scandium with tetravalent zirconium (Borisenko, 1961). Similar to these, it was observed that scandium can make heterovalent substitutions with Mn, Nb, Ta, and Sn in many different minerals (Raade *et al.*, 2004). As another substitution mechanism, so called isovalent substitution, is generally related to ionic radius, electronegativity, and coordination numbers. Ionic radius has the foremost property that provides the substitution, thus scandium commonly makes isovalent substitution with ferric iron (Fe^{3+}) that has nearly same ionic radius with scandium. Also, identical coordination numbers of scandium and rare earth elements make the substitution process convenient. In addition to these, scandium can also make substitutions with aluminum (Al^{3+}) and yttrium (Y^{3+}). Presence of scandium in lateritic and clayey structured deposits is directly associated with its geochemical properties and also strong correlation with iron. Scandium iron correlation can be seen in clayey minerals, including illite, chlorite and montmorillonite, however it cannot be seen in kaolinite type clay minerals. This correlation and the presence of scandium in their structure can be explained as follows; iron contained in clay minerals like illite, chlorite, and montmorillonite, is entered into the octahedral structure of these minerals by making isomorphic substitutions, but this is not the case for kaolinite type clay minerals, where iron does not make any substitution and occur in the form of ferric oxide or hydroxide, in that case scandium cannot enter into the crystal structure of minerals and found as adsorbed on minerals. Therefore, by considering the factors affecting the mechanism of adsorption, including grain size of adsorbent, presence of other ions and their solubility, it is reasonable that no correlation between scandium and iron is observed (Birgöl, 1981). Although the adsorbed state of scandium on kaolinite type clay minerals seems to be beneficial in terms of processing of scandium, such low

concentrations of scandium in this kind of clay minerals prevents the economical extraction of scandium from these ores. In the case of occurrence of iron in crystal structure of illite, montmorillonite and chlorite, namely layer silicates, scandium also entered into the crystal structure as the result of diadochic substitutions by taking the advantage of its ionic size. According to the previous studies, similarity of the ionic sizes of Sc^{3+} and Fe^{2+} allows the heterovalent substitution of these two elements (Norman and Haskin, 1968), however, diadochic substitution of Sc^{3+} with Fe^{3+} in layer silicates makes possible that these two elements show similar behaviors. In addition to these, equality between crystal field stabilization energies of Sc^{3+} and Fe^{3+} , which are equal to zero, strengthen the belief that scandium has entered into the position of ferric iron (Birgöl, 1981). As a brief summary, the occurrence of scandium in such clayey and lateritic deposits and its strong correlation with iron, are based on the similarity between ionic sizes and crystal field stabilization energies of these elements. Recognition of the correlation between scandium and iron, plays an important role for establishing the most suitable processing route in order to obtain a pre-concentrate that is enriched in terms of scandium content.

2.2 Processing Methods for Scandium Ores

2.2.1 Attrition Scrubbing

Attrition scrubbing, one of the commonly used pre-concentration processes, can be defined as the combination of comminution and separation processes in order to remove the surface cover of a material by making some physical and chemical processes including desorption, adsorption, dissolution, diffusion, dispergation, deagglomeration, and physical liberation (Strazisar and Seselj, 1999). In other words, attrition scrubbing can be thought as washing, de-sliming, and decontamination processes depending upon the type of feed material, which can be soil or surface covered ores. Basically, attrition scrubbing is the separation of material covering the solid surface of the material, which can be thought as tailing or pre-concentrate depending on the content of the material, to the liquid medium phase by the help of

mechanical abrasive motion. Aforementioned mechanical abrasive motion is provided by using attrition scrubbers, which liberate material on the solid surface of particles by applying high shear forces (Spiller, 1992).

Originally, there are three main forces that keep the material on the solid surface of particles (Bayley and Biggs, 2005).

- Chemical bonds between the solid surface and the covering material that provides the retainment
- Surface solubility properties of covering materials including hydrophobicity and hydrophilicity
- Physical bonds based on Van der Waals forces between covering material and solid surface

As the result of attrition scrubbing, liberation of covering material is provided by weakening and detaching the bonds between the solid surfaces and covering material under the effect of high shear forces. Although attrition scrubbing can be seen as a comminution process, because of the newly formed fine particles and size reduction of coarser particles, it liberates the surface covering material, by means of excellent mixing and surface abrasion, without fracturing or breaking the material. Therefore, attrition scrubbing is differentiated from the conventional breaking processes based on its fundamental principles. Effect of attrition scrubbing and its comparison to normal breaking processes is given in Figure 2.5.

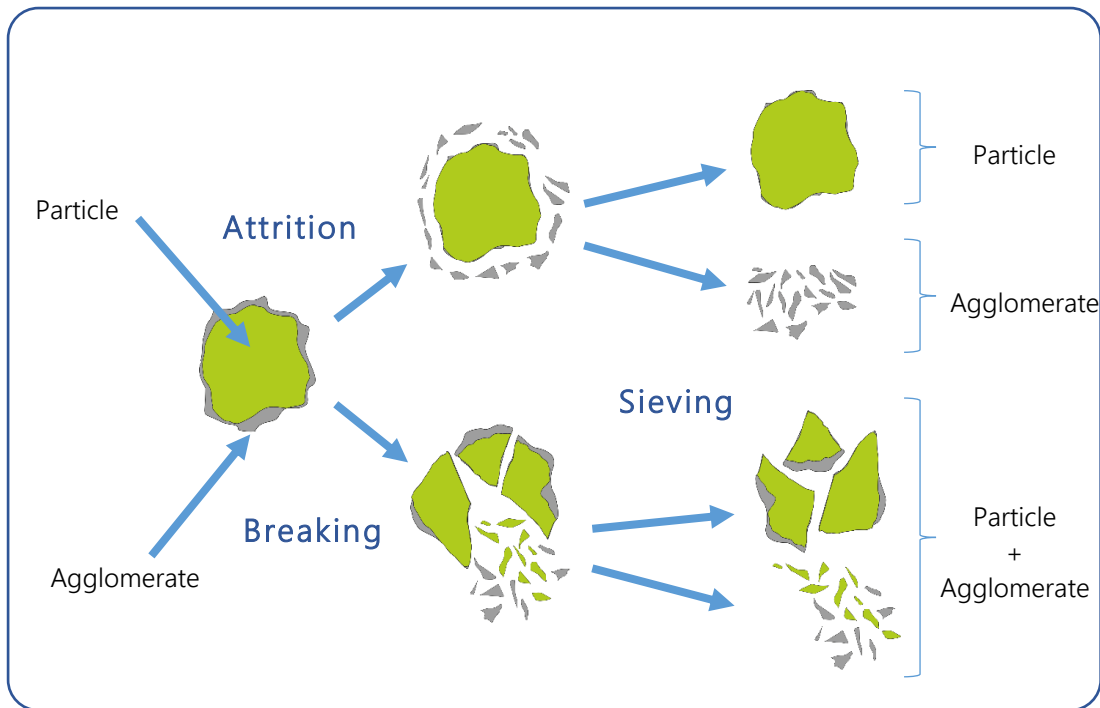


Figure 2.5 Principle of attrition and comparison with conventional breakage process modified from (Petavy and V. Ruban, 2009)

As an industrially widely used pre-concentration technology, attrition scrubbing is used especially for soil processing in addition to the processing of different ores. Based on the fundamental principles of the process, fine particles, generally containing contaminants, are removed (Strazisar and Seselj, 1999). It is generally used for soil remediation process, which normally contains soil washing and decontamination, to remove the contaminants from the surface of soil. By this way, contaminants covering the soil particles are concentrated in finer fractions, thus application of attrition scrubbing removes hydrocarbons with high concentration and high molecular weight. Abrasive and shearing effect of the process cause the interaction of contaminated particles with the liquid medium, walls of the tank, and impellers. The bonds between contaminated particles and soils are detached. After the attrition scrubbing process, clean soil can be obtained following the classification of coarser and finer fractions (Bayley and Biggs, 2005). In addition to these, attrition scrubbing was also used for the decontamination of storm water sediments and it was shown that approximately 70 % of the sediments can be re-processed and re-used in that way (Petavy and V. Ruban,

2009). It is also known that, by the effect of attrition scrubbing, liberation of surface covering materials are provided. In this context, pretreatment of LCD screens, that have low content of indium, via attrition scrubbing, supports the subsequent leaching processes by improving the liberation of indium containing fines. According to the previous studies, 90 % of indium is recovered as 10 times concentrated, from LCD screens (Boundy *et al.*, 2017). Also, other previous studies point out that platinum group minerals are relatively concentrated with higher recoveries by attrition scrubbing from automobile catalytic converters that are made from ceramic-honeycomb (Kim *et al.*, 2010; Liu *et al.*, 2014).

Attrition scrubbing is also used for making the full processing of process tailings by recovering the valuable content in them via economically feasible ways. In this context, surface oxidation of process tailings, which caused recovery losses in subsequent processes, can be removed and also soluble salts covering the surface of process tailings can be dissolved in liquid medium, most commonly water, for recovering the valuable content. In the case of lead-zinc and copper ore processing, surface oxidation of pyrite, which is removed as tailing but having relatively higher cobalt content and can be used as the raw material for the production of sulfuric acid, can be removed by the attrition scrubbing. As another use of attrition scrubbing for the re-processing of tailings, Zn content of electric arc furnace dusts, that also contain high amount of Zn with soluble chlorides, such as NaCl, KCl, PbCl₂, and ZnCl₂, can be increased by dissolving chlorides efficiently in the presence of abrasion effect. In the light of previous studies, approximately 80 % of soluble chlorine, retained on the crude zinc oxide particles, was removed prior to subsequent separation and purification processes (Ding *et al.*, 2015). By this way, through the superior mixing and abrasion ability of attrition scrubbing, the leaching kinetics of gold ores are accelerated by providing the liberation of clayey fractions covering the ore surfaces (Petavy and V. Ruban, 2009).

In addition to the decontamination and remediation applications of attrition scrubbing, it is also used as a washing and de-sliming process for ores with clayey structure, colloidal and fine particles. So, by applying attrition scrubbing process, as a de-sliming effect, clayey, colloidal, and finer materials adsorbed on the surface of material can be removed. Briefly, clay minerals can be generally specified by their fine sizes and weakly bounded particles. Attrition scrubbing of clayey ores in liquid media, such as water, provides the liberation and dispersion of clay particles following the removal of them with subsequent size classification, including de-sliming and sieving operations. It is also known that, attrition scrubbing is beneficial for the removal of clayey fractions from phosphate ores, containing clay minerals, hydrous iron, and aluminum silicates (Abouzeid, 2008). In such phosphate ores, it was observed that the clay minerals, as the main impurities, which can be removed by attrition scrubbing, generally contain silica and silicates. According to the previous studies on phosphate ores, mineralogical analysis, namely XRD, following the sieving of the attrition scrubbed ore from 74 μm , shows that clay minerals are mainly dispersed and liberated in water by enriching in under 74 μm fraction and also phosphate enriched apatite mineral is concentrated in fractions coarser than 74 μm (Guo and Li, 2010). On the contrary, in the case of phosphate ores, including coarse silica and rock chips as the main impurities and phosphate minerals concentrated in finer sizes, finer fraction produced by attrition scrubbing followed by sieving, which is enriched in phosphate minerals, can be taken as the pre-concentrate and also coarser fractions including the main impurities can be removed (Abouzeid, 2008).

Great potential of Turkey in terms of boron minerals has led to the establishment of many boron processing facilities. One of the main challenges for the processing of boron minerals is the removal of their clay content. Therefore, attrition scrubbing, followed by size classification, is generally used for the efficient removal of clay fraction in boron minerals. Clay minerals, adsorbed on the harder and coarser crystalline boron minerals, are removed by the effect of abrasion and a pre-concentrate that is relatively enriched in boron content is produced (Acarkan *et al.*, 2005).

According to the ore to be processed by attrition scrubbing, a general flowchart can be varied in such a way that the clayey fraction can be regarded as concentrate and coarser fractions can be regarded as tailings. In the processing of bentonite ores, which includes high amount of smectite clay minerals, such as montmorillonite, clayey fraction, that is enriched in smectite, is taken as the bentonite concentrate. Associated gangue minerals, which are mainly calcite and quartz, are removed by discarding coarser fractions after size classification (Hassan and Abdel-Khalek, 1998).

The fact that attrition scrubbing is commonly used in the processing of clayey ores for the efficient separation of clay fraction; it can also be used for lateritic nickel-cobalt ores, which contain clay minerals besides their limonite fraction. In other words, attrition scrubbing can be used as a simple beneficiation method for clayey ores such as limonite or manganese (Pryor, 1965). Particularly in lateritic Ni-Co ores, based on the enrichment of valuable elements, such as nickel, cobalt and scandium, in the clayey fraction and the coating of this clay fraction on the common impurity minerals, as calcite, dolomite and silicates, shows that the application of attrition scrubbing for the processing of these type ores is absolutely beneficial. Related with high intensity mechanical mixing and abrasion effect of attrition scrubber that cause intensive collisions between particles, clayey fraction adhering and covering the surface of impurities are liberated and passing to the finer sizes so that a relatively enriched pre-concentrate of Ni-Co-Sc is obtained and exposed to subsequent hydrometallurgical processes. In such processes, the most important effect of attrition scrubbing is providing the removal of impurities, including elements such as Ca, Mg, and Si prior to subsequent hydrometallurgical processes and therefore reducing the chemical reagent consumption and probable operational problems that might be arose due to these impurities. A Similar situation can also be thought for the clayey ores containing rare earth minerals, such as bastnasite, monazite and xenotime. The enrichment of these rare earth minerals at finer size fractions and the presence of impurities at coarser size fractions usually make these ores suitable for the application of attrition scrubbing. According to the previous studies on bastnasite containing ore, it was determined that bastnasite minerals are relatively enriched in finer sizes, namely below 5 μm , and they

are mineralogically locked between fluorite and barite, so attrition scrubbing was used for the purpose of liberating the bastnasite mineral. As the result of classification after the attrition scrubbing, a relatively enriched pre-concentrate, containing 28 % REO, was obtained with 72.6 % recovery (Özbayoğlu and Atalay, 2000).

2.2.1.1 Attrition Scrubber

Attrition scrubbing processes are performed by using attrition scrubbers, which liberate the contaminants or clayey materials covering the solid surface of material, as applying high shear forces on particle surfaces (Spiller, 1992). Attrition scrubbers basically consists of a tank and impellers that are located on a shaft that provides the attrition effect. A simple drawing showing the basic aspects of attrition scrubbers is given in Figure 2-6. By the rotation of impellers at a certain speed, interaction between particles, as collision, friction, dispersion, and abrasion is obtained. In this context, attrition scrubbers consists of a shaft that is located parallel to the vertical axis of rotation. The number of impellers and distance between them can be varied according to the material to be processed. The main purpose of aforementioned impellers are the procurement of interactions between particles at high speeds, but it should not provide interaction between the scrubber and material, as it can cause corrosion of impellers which means reducing the operational lifetime of the attrition scrubber (United States of America Patent No. 7,168,641 B2, 2007). Therefore, blades of the impeller are positioned at opposite angles and so the liquid media, which makes motions in opposite axial directions by the help of rotation, provides the particle to particle interactions (Petavy and V. Ruban, 2009; United States of America Patent No. 7,168,641 B2, 2007). In addition to these, placement of baffles in the tank of the scrubber provides more surfaces for the collision of particles, which results in a more efficient attrition scrubbing process. Apart from these, there are also operational parameters that influence the effectiveness of attrition scrubbing process.

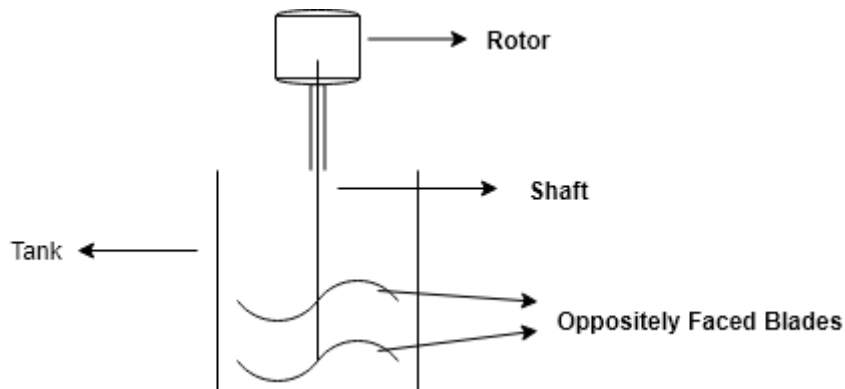


Figure 2.6 Basic aspect of attrition scrubber

2.2.1.2 Operating Parameters of Attrition Scrubbing Process

Operational parameters of attrition scrubbing, which can vary greatly according to the type of the material to be treated, directly influence the cleaning and process efficiencies. These parameters may vary according to the chemical and mineralogical content of the feed material and also vary due to the content of the undesired material that needs to be removed. For instance, the operational parameters during the processing of silica sand or soil are different from the parameters that are used during the treatment of clayey ore minerals. Although contaminant type and concentration, namely the hydrophobicity of the contaminant, which may reduce the cleaning efficiency in the case of soil decontamination, is not a major problem during the processing of ore minerals (Bayley and Biggs, 2005). In this context, there are six main operating parameters that influence the efficiency of attrition scrubbing process, namely solid/liquid ratio, residence time, temperature, rotation speed, particle porosity, and feed particle size.

2.2.1.2.1 Solid - Liquid Ratio

As the first and foremost operating parameter that influences the efficiency of attrition scrubbing, solid/liquid ratio can be mentioned. Based on the fundamental principles of attrition scrubbing, as the intensity and the frequency of the collisions between particles increases, cleaning efficiency also increases, meanwhile increasing the solid/liquid ratio of the pulp causes intense and frequent of collisions between particles (Bayley and Biggs, 2005). Increasing the solid/liquid ratio up to a certain limit value increases the cleaning efficiency already, but also applying attrition scrubbing to a slurry with higher pulp densities reduces the operational efficiency related with high viscosity of the slurry and reduced interaction between particles, for that case slurry acts as a whole particle without perceiving any abrasion effect (Feng *et al.*, 2001). In the case of soil and silica sand decontamination applications, higher pulp densities, as approximately 70 %, was used generally due to the reduced interactions between particles at lower pulp densities (Boundy *et al.*, 2017). Also, the cleaning efficiency and quantity of produced fine particles seems to be limited, because of analogous processes, as de-agglomeration, desorption, dissolution, etc., which are also dependent on the character of the surface of the treated particles (Strazisar and Seselj, 1999). From this point of view, while low solid/liquid ratios can decrease the effect of abrasion, higher solid/liquid ratios may cause a grinding effect according to type of material to be treated. Apart from these, in the case of clayey ores, attrition scrubbing is generally applied at lower slurry densities, because those higher slurry densities induce cake formation which reduces interactions between particles. According to the previous studies, where the aforementioned situation was observed, during the operation of attrition scrubbing, finer fractions including clayey particles, in the slurry behave as pillows due to the higher slurry densities by reducing the inter particular collisions between particles (Acarkan *et al.*, 2005). Therefore, to obtain better results for operational efficiencies of attrition scrubbing for clayey ores, the solid-liquid ratio of the slurry should be adjusted so as to remain around 50 % or below (Özbayoğlu and Atalay, 2000; Hassan and Abdel-Khalek, 1998).

2.2.1.2.2 Residence Time

Residence time is one of the critical parameter that directly affects the efficiency of attrition scrubbing (Bayley and Biggs, 2005). As the residence time increases, the number of collisions between particles increases, which also increases the effect of abrasion on particle surfaces. In fact, direct relation between residence time and operational efficiency until an optimum point was observed but then the increase of residence time to further values did not have any significant effect on the efficiency of the process (Petavy and V. Ruban, 2009). More than that, related with the occurrence state of the undesired material on the solid surface, residence time may not have any effect on the removal rate (Bayley and Biggs, 2005). Increasing the residence time generally promotes the amount of finer products that is produced under the effect of abrasion (Strazisar and Seselj, 1999). However, this cannot be the case sometimes as with the increasing time of residence, bonds between solid surface and valuable elements might be weakened and detached as the result of long term abrasion effect which can lead to loss of recovery and efficiency (Acarkan *et al.*, 2005). For this reason, in the case of recovering clayey fraction covering the harder ore minerals, it may be beneficial to keep the attrition scrubbing residence times relatively longer.

2.2.1.2.3 Rotation Speed

Rotation speed, which is directly related with the torque transmitted to the shaft, is another crucial operational parameter, affecting the efficiency of the process, by increasing both the rate and intensity of collisions between particles. As a result of increases in rotation speed, the amount of produced finer material increases. Increase in rotation speed directly affects the mixing characteristics of slurry, as increasing the impact of collisions, thereby provides the threshold energy value required for weakening or detaching the bonds between undesired material and the solid surface (Bayley and Biggs, 2005). According to the previous studies on pyrophyllite ore, there were drastic changes in the results of attrition scrubbing experiments with rotation speeds of 750 rpm and 1500 rpm. The amount of finer material produced is higher at

1500 rpm, which possibly occurs because of the attainment of required threshold value to weakening or detaching the bonds (Birinci and Sarıkaya, 2004).

2.2.1.2.4 Feed Particle Size

From an operational point of view, it can be said that there is a wide range of feed particle sizes suitable for the attrition scrubbing and also the effect of feed size is generally variable in accordance with the material to be processed. Briefly, removal of undesired material from the coarser fractions can be obtained more efficiently in spite of obtaining it from their finer counterparts due to the intensive effect of abrasion on coarser ones (Strazisar & Seselj, 1999). In the light of previous studies, feed particle top sizes smaller than 6 mm. are found to be appropriate for attrition scrubbing operations (Hassan and Abdel-Khalek, 1998; Özbayoğlu and Atalay, 2000; Birinci and Sarıkaya, 2004).

2.2.1.2.5 Temperature

Attrition scrubbing at certain temperatures, higher than room temperature, can be useful when organic-based impurities are desired to be removed. By increasing the temperature, pulp is getting less viscous and adhesive which caused increased solubility of organic-based impurities (Boundy *et al.*, 2017). In other words, the energy required for breaking or weakening the bonds between undesired contaminants and solid surface is reduced with the increase in slurry temperature. The reduction in the hydrophobicity of hydrocarbon based organic contaminants by the increase in temperature also reduces the energy required to break the bonds, so-called reduces the strength of the bonds, and facilitates the removal of contaminants from the surface (Bayley and Biggs, 2005). In the case of soil decontamination processes, adjustment of temperature may be beneficial for the efficiency of the process; however, it may not have a significant effect in the processing of inorganic mineral species.

2.2.1.2.6 Particle Porosity

The occurrence state of undesired materials, in the ore or soil to be processed by attrition scrubbing, has a crucial importance for the cleaning efficiency. In this context, contaminants can be found as adsorbed on the ore minerals or entered into the crystal lattice of the ore minerals and the occurrence state of contaminants can be determined with regard to the porosity of the solid phase. The ore minerals that have low porosity are generally more suitable for attrition scrubbing, because of the adsorbed state of impurities on them. In addition to these, it may be beneficial to observe the structural strength of the contaminants in order to determine the fragility of the material during the scrubbing process, which is important in terms of recovery (Bayley and Biggs, 2005). In fact, low porosity of the ore mineral to be processed indicates that the impurities are adsorbed on the solid surface of the ore without entering into the crystal lattice and also previous studies have shown that it is easier to remove the undesired surface impurities by using attrition scrubbing process from the calcite which has already low porosity and pore volume (Strazisar and Seselj, 1999).

2.2.2 Enhanced Gravity Separation by Using Falcon Concentrator

2.2.2.1 Gravity Concentration

Prior to the description of enhanced gravity separation techniques, including centrifugal separation by using falcon concentrator, it would be better to explain the conventional gravity concentration techniques and the reasons for the emergence of enhanced gravity separators. In its simplest form, gravity concentration is a mineral processing technique that can be applied to many different types of ores, which contain specifically liberated or denser particles in it, by separating the valuable and the non-valuable material based on the density difference between them (Schriner and Anderson, 2015). Moreover, based on the fundamental principles of conventional gravity separation techniques, minerals are separated due to the differences in their specific gravities under the effect of both their liberation and particle sizes. In order to

achieve a proper gravity separation between minerals, gravitational concentrators are used. Types of conventional gravity concentrators and their main utilization areas are as following (Wills and Finch, 2016);

- Jigs, as the one of the oldest gravitational concentrator, have three common types such as conventional mineral jigs, in line pressure jigs and air pulsated jigs. Industrially, they are generally used for coal cleaning besides the usage for processing of metallic ores.
- Spirals, benefit from centrifugal force as well as gravitational force acted on particles for making the separation between heavier and lighter fractions. Including Humpreys spiral, wash waterless spiral and wash water spiral are generally used for the treatment of heavy mineral sand deposits, chromite ores and recovery of fine coal particles.
- Shaking tables, which separate minerals based on the size and density of particles in slurry, are another important and commonly used gravity concentrator that makes the separation process by the help of motion on an inclined table. Wet shaking tables and air shaking tables can be given as the most commonly known types of shaking tables. Through the lower operational cost and energy efficient structure of the shaking tables, the utilization of them for treating many types of ores including chromium, tin, iron, tungsten, tantalum, mica, barium, titanium, zirconium, and gold ores to a lesser extent.
- Pinched sluices can be basically operated through inclined slopes that are specifically designed for the separation of lighter and heavier particles. Even though they were commonly used in 1960s and 70s, following the generation of spirals their popularity decreased drastically (Falconer, 2003). Trays and cones are the most common types of pinched sluices, which were generally used for the treatment of silica sands, iron, tin and tungsten ores.

As a common mineral processing method applied for the separation of minerals, gravity separation draws attention in terms of its higher efficiency, lower capital and operating costs, and environmentally friendly structure, due to the disuse of chemical reagents (Ottley, 1978). On the other hand, based on the fundamental principles of conventional gravity separators, both texture and particle size distribution of the ore have an important impact on the process efficiency by affecting the density and also the viscosity of the slurry during the operation. However, that may not be the case for the ores containing slimes, namely ultrafine particles which already make the slurry more viscous, through reducing the efficiency, sharpness of separation and also preventing the visibility of cut-point for the separation. Therefore, de-sliming operations by rejecting ultrafine particles to tailings, prior to the application of conventional gravity separation is a common practice for an efficient separation. Although this might be successfully performed for certain types of ores, it results in significant recovery losses for the ores that contain particles with finer size distribution and clay minerals (Wills and Finch, 2016). In the case of these ores, the main problem of separation is the low initial settling velocities of finer particles under the effect of gravity, so there is a critical need for increasing the settling velocities of finer particles for a successful separation. For all these reasons, enhanced gravity separators were developed in order to process ores with finer particle sizes and clayey minerals.

2.2.2.2 Enhanced Gravity Separation

Enhanced gravity separation, namely centrifugal separation, is mainly used for the recovery of fine and ultrafine particles under the influence of ultrahigh gravitational forces (El-Midany and Ibrahim, 2011). As previously mentioned above, gravitational separation of ores containing finer size particles, which is below 38 μm , with conventional gravitational concentrators are challenging due to lower mass and momentum of these particles besides their effect of surface coatings, heteroaggregation, increasing viscosity of the slurry, and also by having higher surface area and surface energy (Sivamohan and Forssberg, 1985). Combination of these effects, reveal the problem of low settling velocity of particles that results in

challenging separation of these particles under gravitational field of 1 g. At that point, enhanced gravity separation provides the increment of gravitational field in order to increase the settling velocities of finer particles, which catalyzes the separation process (Boylu, 2013).

Enhanced gravity separation methods, which was initially applied in the industry at the end of the 1980s- in order to recover free gold present in the placer deposits, have been used as pre-concentration and concentration methods, for the processing of many different types of ores, in combination with other mineral processing technologies. These wide range of applications including, recovery of free gold from heap leach solutions, flotation concentrates, recovery of platinum group elements, and also recovery of valuable elements from tin, tantalum, heavy minerals, coal, and diamonds processes (Cole *et al.*, 2012). In addition to these, enhance gravity separation methods can also be applied to the rare earth ores containing finer size rare earth element bearing particles and clayey minerals (Jordens, et al., 2016). Deposits containing rare earth elements in nature appear in low grades and beneficiation of these ores by conventional methods usually bring along high energy consumption rates and higher processing costs due to the employed hydrometallurgical methods (Dehaine *et al.*, 2017). Therefore, to avoid these effects it may be necessary to employ enhanced gravity methods before the downstream hydrometallurgical processes in order to reduce high costs, high energy consumption, and the adverse effects on the environment to obtain pre-concentrate or concentrate. The same situation is also valid for scandium bearing laterite and clayey ores, which are generally processed by hydrometallurgical methods. Finer particle size distribution and impurity content of these ores, caused to higher cost and also recovery loss during the hydrometallurgical processing of them. Thus, application of enhanced gravity separation on them should be beneficial by obtaining a pre-concentrate enriched in scandium.

Specifically designed devices, namely enhanced gravity separators or centrifugal separators, are used for the application of enhanced gravity separation process. Kelsey Centrifugal Jig, Gekko In-Line Pressure Jig, Mozley Multi Gravity Separator (MGS),

Knelson Concentrator, and Falcon Concentrator are the most commonly used enhanced gravity separators. Basically, these separators increase the initial settling velocity of particles by applying centrifugal forces, thus the separation is enhanced even in the existence of fine particles (Buonvino, 1993). The centrifugal force applied on particles can be expressed by Equation (1);

$$F_c = m[\omega^2 r] = m[N^2(2\pi/60)^2 r] = m a_c \quad (1)$$

where F_c is the centrifugal force, m mass of the particle, ω the angular velocity, r the bowl radius, N the rotational speed, and a_c the centrifugal acceleration (Lins *et al.*, 1992). By applying centrifugal force, settling velocity of fine particles is increased, as scattering radially outwards by accelerating continuously, which comes along with the enhanced separation (Buonvino, 1993). In more details, centrifugal forces applied on the particles, during the separation process in the bowl of centrifugal separators, is much higher than the gravitational force that leads to increase in efficiency of gravity separation of finer particles. As the result of this, finer particles with higher specific gravities are accumulated on the bowl of the separator, while the lower specific gravity particles are transferred with the flowing fluid by being subjected to tailings fraction (Falconer, 2003). In this way, rare earth ores, that have finer particle size distributions, can be processed by using centrifugal separators, namely spinning bowl concentrators. Due to the type of the ore, these separators may have processed feed material with particle sizes as finer as 10 μm , owing to their spinning bowls (Schriner, 2016). In this study, the falcon concentrator, which is one of the common centrifugal separators, will be used in order to make a successful gravity separation in the presence of finer size particles.

2.2.2.3 Falcon Concentrator

The falcon concentrator, which was developed at the end of 1970s for the recovery of free gold contained in placer deposits, is recently used in many industrial applications including physical separation of lighter and heavier particles in the presence of finer particles. Such challenging separation of these particles, by using conventional physical separation methods, accompanies the use of falcon concentrator for the processing of waste electrical and electronic equipment, dredged sediments, coal and clay minerals containing metallic and rare earth ores, and tailings dewatering in the sand industry (Rabotin and Sanders, 2014). Based on the centrifugal separation theory, initial settling velocities of particles are increased by the effect of g-force during the falcon concentrator process, thus it would be useful to examine the working and separation principles of the falcon concentrator in more details.

Simply, falcon concentrator can be thought as an enhanced gravity separator consisting of a fast spinning bowl for the separation of finer size particles according to the differences in their specific gravities. Moreover, in order to increase the initial settling velocities of finer particles, falcon concentrator can provide enhanced gravitational force up to 300 g that leads to successful separation at both lower gravity points and finer sizes, typically finer than 100 μm (El-Midany and Ibrahim, 2011). Therefore, by providing such a higher centrifugal force, falcon concentrator can be classified as the centrifugal separator that has lower separation density and also effective separating density can be decreased by increasing the centrifugal force generated by the fast spinning bowl (Boylu, 2013). During the separation process, the aforementioned fast spinning bowl generates an enhanced gravity field by exerting centrifugal forces on particles which provides the separation of particles, even though they have ultra-fine sizes, directly related with the differential settling velocities of them (Deveau C. , 2006; Deveau and Young, 2005) .

As centrifugal separators have such a crucial role, the falcon concentrator has many advantages. Its ability to operate in the presence of ultra-fine particles, down to 15-20 μm , fulfills the need for processing of ores with such finer particle sizes. In addition to that, under the favor of simple and enduring design of the falcon concentrator, it is not a labor intensive processing method. Directly related with technological developments, up and coming high capacity models of falcon concentrators, make this device industrially attractive. Apart from these advantages, the falcon concentrator also has some disadvantages that the geometry of the bowl and opening of orifices should be finely tuned according to the size distribution of ore to be processed; otherwise higher efficiencies cannot be obtained. Based on the closed structural design of this device, cut points cannot be seen during the operational process. As the last and foremost disadvantage, enrichment ratio of falcon concentrator is relatively lower, thus it can be directly thought as a pre-concentration process, which is mainly used for separating gangue minerals from valuable minerals (Falconer, 2003).

By investigating the separation mechanism occurring in the falcon concentrator, as the slurry is fed through the spinning bowl via the inlet pipe, slurry moves from bottom to top, while reaction of heavier particles to the exerted centrifugal force are more than their lighter counterparts. As the result of this reaction, heavier particles tend to be retained on the wall of the bowl and the lighter particles are disposed to top of the bowl with the flowing water (Falconer, 2003). During the operation, heavy particles are retained on the riffles that are located at the upper part of the bowl (Nayak and Pal, 2013). By this way, separation between lighter and heavier particles is obtained, thereby lighter particles are collected from the tailing discharge by the effect of supplied water from the bottom and heavier particles are collected from the bowl by adjusting the orifices located on that (Falconer, 2003). In a different way, heavier particles can also be collected via the help of fluidization or pressurized back water which results in the migration of heavier particles through the bottom or outside of the bowl (Nayak and Pal, 2013). Partial cross-section of a falcon concentrator showing the behavior of lighter and heavier particles in the bowl is given in Figure 2.7.

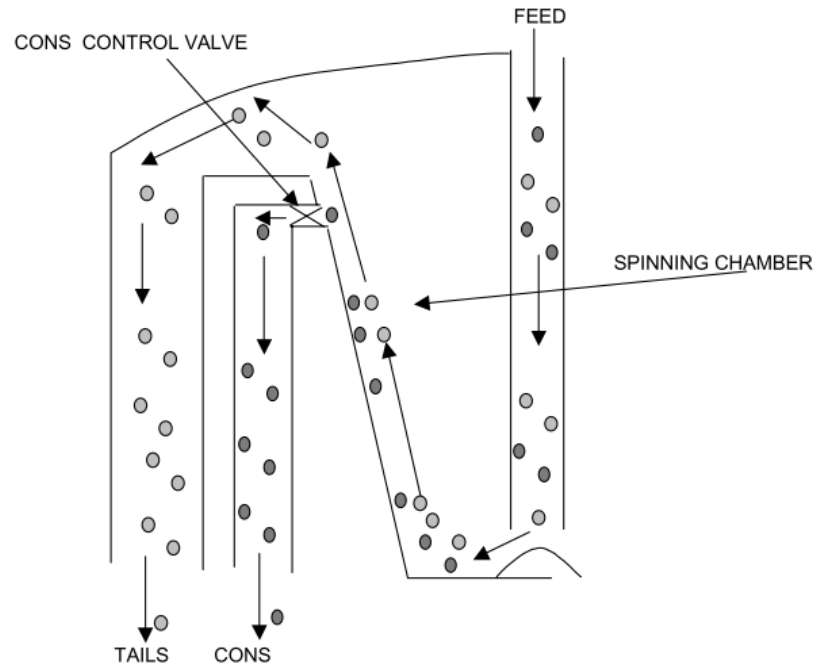


Figure 2.7 Partial cross-section of the falcon concentrator adapted from (Falconer, 2003)

In order to achieve a physical separation in the bowl of the falcon concentrator based on the gravities of particles, external forces are exerted on particles. These are centrifugal force that derived from the spinning of the bowl, frictional force between the particles and solids bed, and drag force that originated from the flowing fluid (Buonvino, 1993). By the effect of centrifugal force, initial settling velocities of particles are increased and the inhibiting effect of finer size ranges are minimized while increasing the effect of density during the separation process. Frictional force between the particles and solid beds provides the retainment of heavier particles on the bowl of the concentrator due to impression of them that is greater than lighter particles (Buonvino, 1993). Moreover, drag force of the fluid completes the separation process by carrying the lighter particles from bottom to top of the bowl through the tailing discharge. Also in the case of higher drag forces overcome the centrifugal forces, removal of lighter particles that located in the heavier fraction, which are already concentrated on the wall of the bowl due to the quick stratification, can be obtained (El-Midany and Ibrahim, 2011).

Under the effect of aforementioned forces, there are mainly three steps for the stratification of heavier particles on the bowl of the falcon concentrator. The initial step is the unselective deposition, which occurs immediately after the feeding of the slurry through the concentrator bowl, in order to form a bed layer (Jordens *et al.*, 2014; Laplante, 1993). Although the duration of this step is considerably short, recoveries are generally higher (Buonvino, 1993). Following the formation of the bed layer, the second step, namely selective deposition, begins with the deposition of heavier particles onto the previously formed bed layer. Selective deposition step is continued until the saturation point of the bed is reached. During this second step, higher recoveries are also obtained due to the selective separation of heavier and lighter particles (Buonvino, 1993; Jordens, *et al.*, 2014; Laplante, 1993). As the saturation point is reached, recovery drastically drops because of both the completely filled capture sites and erosion of the stratum under the effect of flowing slurry (Laplante, 1993). Finally, the recovery of heavier particles almost reaches zero, which means that the bowl of the concentrator should be discharged. By observing the third stratum, formed after the selective deposition, it can be directly seen that the third stratum is mainly composed of gangue particles, which can be easily removed as they are loosely bounded on the second stratum (McAlister, 1992). Discharging of the concentrate retained on the bowl of the concentrator is removed manually for the batch type falcon concentrators; however, it can be removed automatically by recovery slots in the continuous type falcon concentrators which will also be mentioned on the following pages. By examining the above mentioned stratification process in terms of particle sizes, particles are retained on the bowl mainly due to two cases. Coarse particles contained in the feed slurry, entrap themselves to the concentrate bed based on the effect of centrifugal force. Finer particles, as their counterparts, are retained on the bowl under the favor of pores created by the coarser particles. These pores can be thought as the readily formed capture sites and thereby increase of the size of the captured particles also increases the pore volumes resulted with the increase in recovery. At that point, it should be taken into account that, although the recovery is increased, the grade of the concentrate may be decreased due to the chance of the retainment of gangue particles on these pores (Buonvino, 1993).

In conjunction with the type of ore to be processed by using falcon concentrators, the type of the falcon concentrator should be chosen carefully in order to achieve a successful separation. In this context, accomplishment of the separation is directly related with the design, especially geometry of the bowl, of the falcon concentrator. There are mainly three different types of falcon concentrators based on their bowl design and operating principles, as semi-batch (SB), ultra-fine (UF), and continuous (C) falcon concentrators.

The falcon semi-batch (SB) concentrator separates the particles based on the application of enhanced gravitational forces on particles. Slurry is fed through the spinning bowl by the help of a feeding pipe. The bowl of the SB concentrator contains a smooth-walled bottom part and a fluidized collection zone at the top (Cole *et al.*, 2012). Collection riffles are located on the fluidized collection zone and can be specifically designed with regard to the characteristics of the ore to be processed (Rabotin and Sanders, 2014). Directly related with the smooth wall design of the SB concentrator, heavier particles are retained on the wall of the bowl, while lighter particles are discharged under the effect of flowing fluid (Cole *et al.*, 2012). As a SB concentrator, at the end of each stratification cycle, which was previously described, the concentrator is almost stopped and already retained heavier particles are collected from the bottom of the concentrator by making elution with fluidization. The capacity of each cycle and the bowl can also be arranged by the help of the counter-pressure flow rate (Rabotin and Sanders, 2014). Industrially, they are generally used in combination with grinding and cyclone circuits and they have the capability for generating 50 to 300 G centrifugal force and processing 1 to 400 tons of ore per hour (Cole *et al.*, 2012).

As it was in SB concentrator, the falcon ultra-fine (UF) concentrator has almost a similar bowl design with reduced diameter at the top of the bowl (Rabotin and Sanders, 2014). UF concentrator is a specifically designed falcon concentrator especially for processing slurries containing ultra-fine particles. The main difference of UF

concentrator is the lack of fluidizing water (Lins *et al.*, 1992), so that once the particle is retained on the wall of the bowl, it is not possible to substitute that particle with another particle thus preventing the flushing of fine heavier particles to the tailing discharge (Jordens, et al., 2016; Dehaine *et al.*, 2017). Instead of the fluidizing water, UF concentrator contains a pneumatically adjustable lip at the top of the spinning bowl, which tends to be expanded throughout the operating cycle. This special device has the ability of generating up to 600 G force and processing slurries that contain particles below 75 μm (Cole *et al.*, 2012). Although these concentrators may have achieved successful separations up to 3 μm , the most crucial industrial handicap of them is the low operating capacities (Rabotin and Sanders, 2014). Based on the aforementioned characteristics of the SB and UF concentrators, bowl schematics are given in Figure 2.8.

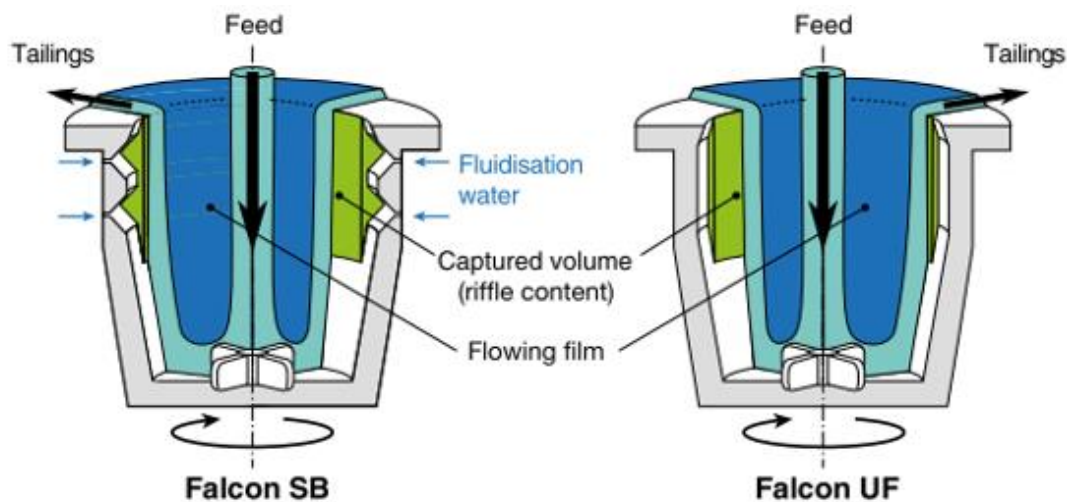


Figure 2.8 Bowl schematics of Falcon SB and UF concentrators adapted from (Dehaine *et al.*, 2017)

The falcon continuous concentrator (C) has adjustable apertures located on the retention zone. Apart from that, the operating principles and design of this concentrator are the same as the UF concentrator (Rabotin and Sanders, 2014). In comparison with the batch type concentrators, C concentrators have the ability for operating continuously owing to their adjustable apertures, which provide the steady state production of the concentrate (McAlister and Armstrong, 1998). Due to the continuous

operating nature of these concentrators, stratification cycles also continues without interruption, and therefore particles pass through the following sequences, namely complete dispersion, partial stratification, complete stratification and retention, from bottom to top of the bowl (Honaker *et al.*, 1996). Briefly, C concentrators have the ability to process 1 to 200 tons of ore per hour with generating 50 to 300 G centrifugal force (Cole *et al.*, 2012).

As it can be seen from the different types of the falcon concentrator, it can be used for the processing of many different types of ore. Prior to discussing the operating parameters of the falcon concentrator, it will be useful to mention about the intended purpose of these concentrators for the processing of different types of ores. Initially the falcon concentrator was specifically used for the processing of gold ores, containing free gold, in order to reduce the operating cost and challenges that were originated from the conventional cyanide leaching process. Also, it was observed that the device was efficient for the recovery of gold particles, less than 37 μm (Laplante, 1993). In this context, falcon concentrators were also used for the recovery of fine coal particles from tailings and also from froth flotation concentrates. Presence of sulfur bearing minerals, such as pyrite, and ultrafine clay minerals, which cover the surface of the coal, decrease the efficiency of coal cleaning process. Therefore, falcon concentrators are commonly used for the removal of gangue minerals to obtain a clean coal concentrate (Zhang, et al., 2011; Boylu, 2013; Oruç *et al.*, 2010).

The falcon concentrator is also used for the processing of low grade fine iron ores, mainly containing hematite, in order to obtain a pre-concentrate enriched in Fe and also remove the silica, which can be problematic for the subsequent processes (Nayak and Pal, 2013; Dey *et al.*, 2012). Similarly, for the processing of celestite (a strontium mineral) bearing ores, which have finer size distribution, falcon concentrators can be used for the selective separation of celestite and calcite (El-Midany and Ibrahim, 2011). Cassiterite, as a tin oxide mineral, is present together with chalcopyrite, sphalerite, which behaves somewhat same as the cassiterite during processing operations, and silica as the main gangue mineral. Generally, cassiterite is found as

locked between interparticle grains in these ores, therefore over-grinding may be needed for the liberation of cassiterite. Falcon concentrator is used for the selective removal of chalcopyrite, sphalerite and silica in the presence of slimes. Silica, as the lighter impurity, is removed first and then chalcopyrite and sphalerite are removed by a second step of the falcon concentration process. As the result of this, relatively enriched cassiterite concentrate is obtained (Buonvino, 1993).

Moreover, based on the mineralogical characteristics of the rare earth ores, conventional gravity separation methods are inefficient, mainly resulting from the similar specific gravities of valuable and non-valuable minerals and also the finer size of the particles. Therefore, in order to obtain a pre-concentrate enriched in rare earth minerals by using falcon concentrators, is an efficient way in terms of both operating cost and recoveries in subsequent processes (Jordens *et al.*, 2015). Within this context, similar to rare earth ores, scandium bearing lateritic Ni-Co ores and ion-adsorbed clay ores, which have finer size distributions, may be processed by using falcon concentrators in order to produce a relatively enriched pre-concentrate and remove gangue minerals. The falcon concentrator, as a widely used centrifugal concentrator, has operating parameters that directly affect the efficiency of separation, and therefore fine tuning of these parameters is crucial for a successful separation process.

2.2.2.4 Operating Parameters of Falcon Concentrator

Operational parameters of falcon concentrator, which can vary greatly according to the type of the material to be treated, directly influence the separation efficiency. These parameters may vary according to the chemical and mineralogical content of the feed material and also vary due to the content of the undesired material that needs to be removed. As mentioned above, separation efficiency of the falcon concentrator is directly related with the geometry of its bowl, but also in order to make a successful separation, fine tuning of operational parameters should be done. In this context, there are four main parameters, namely centrifugal force (G-force), fluidization water pressure, feeding rate and density of the slurry, and feed particle size (Falconer, 2003;

Schriner and Anderson, 2015). In addition to these parameters, temperature of the fluidization water, impurity amount and content of the slurry, and also the frequency of pulse orifices can be important according to the type of the ore to be processed. Briefly, increasing the frequency of pulse orifices directly increases the weight of concentrate to be taken thus increasing the recovery with a decrease in grade (Falconer, 2003). Temperature of the fluidization water is important in the presence of organic impurities; thereby removal of them can be facilitated by taking the advantage of solubility of them in relatively higher temperatures. Due to the limitations on the operating capacity of the falcon concentrator, generally it is not efficient to process the ores with high amount of impurities, causing lower stratification cycle times in batch types and irregular stratification in the continuous type concentrators (Jordens *et al.*, 2013).

2.2.2.4.1 Centrifugal Force

The falcon concentrator operates at higher rotation speeds and so providing an enhanced gravity field by the help of centrifugal forces (G-force). Based on the provided G-force, initial settling velocities of particles are increased; therefore effective specific gravity of separation is decreased (Boylu, 2013; Oruç *et al.*, 2010). In this context, centrifugal force is one of the most dominant factors and directly affects the separation efficiency.

Varying the centrifugal force, namely rotation speed of the bowl, generated by the falcon concentrator can also differentiate the separation mechanism that occurs in the bowl. By increasing the rotation velocity of the bowl, flow regime on the bowl is smoothed via the effect of higher centrifugal forces exerted on particles and also stratification steps are changed due to the mechanical entrapment of lighter particles into the concentrate. In the case of greater centrifugal forces compared to drag forces, the stratification layers become compact and drag forces are not enough to remove lighter particles entrapped in these layers. At that condition, particles are retained on the wall of the bowl by the following sequence: heavier particles of larger sizes,

heavier particles of smaller sizes, lighter particles of larger sizes, lighter particles of smaller sizes. In addition to these, at the condition of higher rotation speeds, heavier particles are directly stratified without exposing the effect of fluidization zone, which also provides the compaction of the stratified bed. On the other hand, under the effect of high rotation speeds, heavier particles of smaller sizes are substituted with lighter particles, thus heavier particles forced the lighter particles to the central part of the spinning bowl by taking advantage of the exerted drag force in order to remove them in the overflow stream (El-Midany and Ibrahim, 2011). Correspondingly, recovery of the separation process is increased at higher rotation speeds but at the same time the grade of the concentrate is decreased related with the entrapment of lighter particles (El-Midany and Ibrahim, 2011; Dey *et al.*, 2012).

In the case of lower rotation speeds, due to the lower centrifugal forces applied on the particles, drag forces are more effective on the separation process. In that way, selective stratification and separation of heavier particles can be obtained, which leads to increase in the concentrate grade besides decreasing recovery (El-Midany and Ibrahim, 2011; Zhang, et al., 2011). Based on above mentioned effects of centrifugal force on the separation process, it is crucial in terms of recovery. Although the grade of the concentrate tends to be decreased by the increasing recovery, this can be thought as a smaller effect besides the effect of it on the recovery (Schriner and Anderson, 2015; Schriner, 2016).

2.2.2.4.2 Fluidization Water Pressure

As the centrifugal force, fluidization water pressure is one of the most important parameters for the efficiency of separation. Based on the separation principles of falcon concentrators, feed slurry is confronted with the fluidizing water, which comes through the orifices once the slurry enters to the spinning bowl. The zone that the feed slurry confronted with the fluidizing water is called the fluidization zone. In this zone, drag forces generated by the fluidizing water are exerted on the particles (El-Midany and Ibrahim, 2011). Under the effect of drag forces, lighter particles have no more

chance to retain or entrap on the wall of the bowl, thus they are subjected to overflow through the tailings discharge. Within this scope, increasing the fluidization water pressure also assists the removal of the lighter material entrapped in the concentrate by means of the effect of drag force (Nayak and Pal, 2013). As a consequence of that, grade of the concentrate increases but the recovery decreases as the heavier particles can also be subjected to the overflow together with the lighter particles. As it can be seen, rate of fluidizing water pressure is mainly related with the concentrate grade unlike the rate of centrifugal force, which is mainly related with the recovery of the desired material.

2.2.2.4.3 Feeding Rate and Density of the Slurry

In order to perform a successful separation by using falcon concentrators, feeding rate and density of the feed slurry should be adjusted accurately. Effect of the density of the feed slurry on the efficiency of the process is directly related with the settling conditions of the particles. According to the previous studies, it was noted that the feed slurry density has a slight effect on the separation process due to that the heavier particles are immediately pushed through the bed for the stratification under the effect of centrifugal force (Schriner and Anderson, 2015; Buonvino, 1993). However, in other studies, it has been found that the slurry density is effective on the separation process up to a certain point and it directly affects the settling conditions of the particles in slurry. By considering the effect of solid content of slurry on the viscosity, it can be thought that there may exist free or hindered settling conditions. In the case of lower pulp densities at which free settling conditions occur, particles are more dispersed, when they are fed into the bowl. Although heavier and relatively coarser particles are retained on the bowl of the concentrator, heavier but finer particles are subjected to the overflow under the effect of the drag forces, which cause increased grade but also the decreased recovery (Dey *et al.*, 2012).

Hindered settling conditions are observed above a certain solid concentration in the slurry which cause the viscous pulp and also affects the settlement of particles. Furthermore, desired dense media can be readily formed based on the nature of feed slurries that contain ultrafine and clayey particles. As the result of hindered settling conditions, heavier but finer particles can also be retained on the wall of the bowl. By that way, the effect of the density of particles on the separation becomes more important than the effect of the size of particles (Boylu, 2013). Operationally, this situation leads to the increase in recovery besides the decreased grade of the concentrate. According to previous studies, it was stated that hindered settling conditions occur at solid concentrations above 20 %, in the presence of ultrafine and clayey particles, resulting in the increased recovery of the process (Boylu, 2013; Zhang, et al., 2011). In terms of the feed rate of the slurry, it affects the recovery of desired material due to the capacity limiting effect. Due to the stratification cycle, when the slurry is fed through the feeding pipe of the bowl, particles in the feed slurry are directly forced to wall of the spinning bowl unselectively. In the case of higher feeding rates, grade of the concentrate decreased because of the entrapped lighter particles located on the stratified bed (Schriner and Anderson, 2015). Therefore, fine tuning of the feeding rate should be done in order to achieve a successful separation with a maximum capacity.

2.2.2.4.4 Feed Particle Size

The falcon concentrator is specifically designed for the recovery of fine and denser particles from the feed slurries containing fine particles (Laplante, 1993). Therefore, the feed particle size has a crucial effect on the grade of the concentrate produced from the falcon concentrator (Schriner and Anderson, 2015; Buonvino, 1993). It will be more accurate to examine the feed particle size in terms of undesired particle size and desired particle size. Related with the drag force exerted on the particles, as the particle size of gangue minerals decreases, the recovery of the desired material increases due to the gangue minerals that are subjected to overflow more easily. If the particle size of gangue minerals increases, the drag force exerted on them is not enough to push

them through the tailings discharge and recovery of the desired material decreases because the capture sites of the bowl is occupied by gangue minerals. On the contrary, if the particle size of the desired material is increased, retainment of them on the surface of the bowl is facilitated and also there are more capture sites available due to the coarser particles retained on the wall of the bowl. In addition to these, based on the increase of capture sites, recovery of the desired material is also increased (Buonvino, 1993).

2.2.3 Magnetic Separation

As an old separation method, usage of magnetic separation in the mineral processing industry dates back to the end of 1700's. Although the magnetic separation method was initially used for the removal of tramp iron, it is being used for the processing of many different ores currently. It will be useful to give the main principles and types of the magnetic separation methods prior to the usage areas and previous studies of magnetic separation.

In the simplest form, magnetic separation is conducted in order to separate the magnetic and nonmagnetic particles contained in a material, based on the differences between magnetic properties of minerals. Magnetic separation process is made by the use of magnetic separators, which provides a magnetic field, to produce a magnetic concentrate, including magnetic minerals, and to remove non-magnetic minerals or to remove magnetic contaminants (Wills and Finch, 2016). On the other hand, magnetic separation is a type of physical separation that separates the individual particles based on the interaction between magnetic forces, gravitational, frictional or inertial forces and also forces of attraction and repulsion between these particles (Oberteuffer, 1974). Among these forces, the most important force that directly influences the efficiency of separation is magnetic force. The behavior of a mineral in the magnetic field mostly depends on the magnetic forces applied.

In accordance with the different behaviors of particles in an exerted magnetic field, three different types of magnetic properties can be observed. These are namely, ferromagnetic, paramagnetic, and diamagnetic behaviors. Starting with the diamagnetic materials, namely non-magnetic materials, they are repelled by the magnetic field lines and have tendency of pulling away from magnetic force lines of the exerted magnetic field to a point of lesser intensity (Fuerstenau and Han, 2003). Since these materials are not attracted by magnetic forces, they end-up in the non-magnetic fraction of magnetic separation (Wills and Finch, 2016). Paramagnetic minerals, which consist of ferromagnetic minerals, are attracted by the magnetic field lines and have tendency for moving to a point of higher intensity of magnetic field (Fuerstenau and Han, 2003). So, the paramagnetic minerals are attracted by magnetic force and so they are subjected to the magnetic fraction of magnetic separation. Briefly, paramagnetic properties of these minerals, called as paramagnetism, are based on the formation of magnetic dipoles due to the presence of unpaired electrons. By the effect of these magnetic dipoles in an exerted magnetic field, alignment of magnetic dipoles leads to occurrence of magnetic moment, which gives rise to magnetization of the material and are affected by the magnetic force lines in a magnetic field (Wills and Finch, 2016). Although ferromagnetic minerals can be thought as a sub-group of paramagnetic minerals, ferromagnetic properties are the special case of paramagnetic properties. In the case of magnetic separation of ferromagnetic minerals, magnetic dipoles of these minerals are aligned quicker than other magnetic minerals, which prove their higher affinity for the magnetic forces, and acquire magnetism, once they are subjected to magnetic field even though the magnetic field is removed (Wills and Finch, 2016; Fuerstenau and Han, 2003).

By considering the aforementioned different magnetic properties of the minerals, they are separated under the effect of different magnetic field intensities. Magnetic separators are classified due to the generated magnetic field, mainly as high intensity and low intensity magnetic separators. In this context, related with their higher affinity for the magnetic forces, ferromagnetic minerals can be selectively separated from their nonmagnetic and paramagnetic counter parts under low intensity magnetic fields. Low

intensity magnetic separators can generate such lower magnetic fields. Also, paramagnetic minerals can be concentrated under the generation of higher magnetic field intensities by using high intensity magnetic separators. Magnetic forces applied on the particles in a magnetic field relatively changes with respect to the intensity of the magnetic field. Therefore, magnetic force is one of the critical aspects for the magnetic separation methods.

Principally, degree of magnetic force generated during the magnetic separation is adjusted by the magnetic induction, so-called magnetic flux density, which can be explained as the number of magnetic force lines passing through a unit area. Magnetic induction is symbolized as B and the unit of magnetic induction is Tesla (T) (Wills and Finch, 2016). Magnetic field intensity (H), generated by the magnetic separator, generates the magnetic force lines through the material. It is also called magnetizing force and has a unit of ampere per meter (Bennett *et al.*, 1978). As the last component of the magnetism, magnetization (M , ampere per meter) is the volumetric density of induced magnetic dipoles. Combination of the magnetic induction (B), magnetic field intensity (H), and magnetization (M) are given in Equation (2) (Fuerstenau and Han, 2003).

$$B = \mu_0(H+M) \quad (2)$$

$$\mu_0 = \text{permeability of free space (N/A}^2\text{)}$$

In the course of magnetic separation, magnetic susceptibilities of minerals have also a crucial importance for the effectivity of magnetic force exerted on them. Magnetic susceptibility of a mineral can be expressed as the ratio of M to H (Fuerstenau and Han, 2003). Based on the increase of the field intensity affected on particles towards the surface of the magnets, particles having higher magnetic susceptibilities (paramagnetic and ferromagnetic minerals), which are already concentrated on the magnetic field lines, are attracted more towards the magnetic force. In fact, the main separation mechanism is based on this principle and therefore the diamagnetic

minerals, which have lower magnetic susceptibilities, are not affected by the applied magnetic field (Wills and Finch, 2016). During the separation; magnetic, gravitational, frictional, and hydrodynamic forces have tendency to enhance the separation, but also inter-particle forces try to reduce the degree of separation. In this context, magnetic force exerted on a particle under an externally applied magnetic field is given in Equation (3) (Oberteuffer, 1974).

$$F_x = V(\chi_p - \chi_m)H \frac{dB}{dx} \quad (3)$$

F_x = Magnetic force (N)

V = Particle volume (m³)

χ_p = Magnetic susceptibility of the particle

χ_m = Magnetic susceptibility of fluid medium

Variation of magnetic force with regard to the externally applied magnetic field and the difference in magnetic properties of minerals, necessitate the usage of magnetic separators with different magnetic field intensities. In addition to the most common magnetic separators, as low intensity and high intensity, high gradient and superconducting separators also have become popular with the technological developments. In order to process ferromagnetic and highly paramagnetic minerals containing ores, low intensity magnetic separators are used. Removal of tramp iron can also be performed by the usage of low intensity magnetic separators (Wills and Finch, 2016). Low intensity magnetic separators can generate magnetic field with intensities up to 0.2 T (2000 Gauss) having both dry and wet operating types (Fuerstenau and Han, 2003). Industrially, dry low intensity magnetic separators are mainly used for the processing of coarse sands that have higher magnetic susceptibilities. Dry low intensity magnetic separators cannot be operated efficiently below a certain particle size, due to the loss and inefficient separation. In those cases, wet low intensity magnetic separators are used by taking the advantage of the fluid medium, which results in increased recovery and separation efficiency. They are mainly used for the recycling of dense medium separation media and for the treatment

of ferromagnetic sands (Wills and Finch, 2016). In addition to these, lower grade iron ores, including magnetite and in some cases finer hematite concentrated in siliceous rocks, can be further upgraded for the production of concentrate enriched in iron with the low intensity magnetic separators. Drum separators, cross-belt separators and rare earth roll separators are grouped under the low intensity magnetic separators.

High intensity magnetic separators are used for the processing of paramagnetic and weakly paramagnetic minerals by generating magnetic fields up to 2 T (20,000 Gauss). In the case of dry high intensity magnetic separation, mainly induced roll magnetic separators are used for the separation of paramagnetic impurities from industrial minerals such as quartz, feldspar, and calcite (Fuerstenau and Han, 2003). Separation efficiency of dry magnetic separators decreases with the presence of finer size particles, due to the effects of air currents, adhesion between particles, and particle-rotor adhesion (Wills and Finch, 2016). Wet high intensity magnetic separators, which are also used in the presence of finer particles, mainly include Jones Separator, Frantz Isodynamic Separator, and high intensity drum magnetic separator (Fuerstenau and Han, 2003). Generally, wet high intensity magnetic separators are used for the processing of low-grade iron ores containing hematite and goethite in order to produce a concentrate enriched in iron. In addition to this, these separators are also used for the removal of magnetic impurities from cassiterite, asbestos, and the ferrosilicate minerals from clayey ores, Fe and Ti oxides from zirconium and rutile from heavy mineral beach sands (Corrans and Svoboda, 1985). In all these cases, high intensity magnetic separators are used to remove the impurities and to obtain a pre-concentrate prior to subsequent processes. High gradient magnetic separators have also improved with the usage of high technologies for the separation of weakly paramagnetic minerals. These devices, including SLon, and Eriez, are used for the removal of iron bearing minerals from kaolinite, quartz, garnet, and also the concentration of iron-bearing minerals and rare earth minerals (Wills and Finch, 2016).

Based on the usage of magnetic separators for processing of such different ores, in order to make a successful separation, field strength should be carefully selected according to the material to be processed, as higher field strengths may lead to increased recovery of weakly magnetic gangue particles (Wills and Finch, 2016). In this context, it was shown that the magnetic separation could be used for the beneficiation and pre-concentration of rare earth minerals due to their inherently strong paramagnetism. Such separations can be exemplified as the separation of strongly paramagnetic monazite and xenotime from heavy mineral sands (Jordens, et al., 2016; Jordens *et al.*, 2014).

As another application of magnetic separation, weakly magnetic impurities contained in clayey ores can be removed (Oberteuffer, 1974). Within this scope, due to the paramagnetic properties of scandium bearing clay minerals, namely Pyroxene and diopside, separation of them from their diamagnetic quartz and serpentine counterparts may be possible. In addition to these, paramagnetic property of scandium renders the magnetic separation possible. According to the previous study on the magnetic separation of scandium from biotite, tremolite, ilmenite, and titanite containing clayey ore, a scandium pre-concentrate was obtained by making high intensity magnetic separation (Likun and Yun, 2010). In summary, scandium is mainly enriched in paramagnetic minerals as biotite, tremolite and ilmenite. Following the grinding of the ore under 74 μm because of the presence of the ferromagnetic minerals, low intensity magnetic separation at 1500 G was conducted prior to high gradient magnetic separation. After the low intensity magnetic separation, two-stage high gradient magnetic separation, including one rougher and one cleaner stage, was conducted at 18000 G in order to obtain a pre-concentrate enriched in scandium. As a result, a scandium pre-concentrate containing 314.89 ppm Sc was obtained at 77.53 % recovery from run-of-mine containing 49.8 ppm Sc. Besides that, most of rare earth elements are enriched in scandium pre-concentrate (Likun and Yun, 2010). In another study conducted on the magnetic separation of scandium from Sc-bearing ultrabasic rock, following the crushing and grinding of the ore under 74 μm , high intensity magnetic separation at 6000-6500 G was made. As a consequence, a scandium pre-concentrate

containing 350-400 ppm Sc was obtained at 37.54 % recovery from run-of-mine containing 45-80 ppm Sc. Although the recovery of scandium at the magnetic concentrate seemed to be lower, tailings of the magnetic separation was subjected to flotation for obtaining a pre-concentrate (China Patent No. CN104263926A, 2014). Due to all of these reasons, magnetic separation applied on the Sc-bearing ores can be thought as a potential pre-concentration step for increasing the efficiency of subsequent leaching and SX processes.

As aforementioned above, magnetic force is one of the most important parameters that affects the separation efficiency and it is mainly related with the intensity of magnetic field. By the increase of magnetic field, so the magnetic force, grade of the concentrate decreases but also the recovery of the magnetic minerals increases due to the increased effect of magnetic field and magnetic force for capturing the magnetic and weakly magnetic particles (Likun and Yun, 2010). On the other hand, contrary to the effect of the magnetic force, decrease in the recovery by the effect of the increase in competing forces causes an increase in the grade of the concentrate (Oberteuffer, 1974). Apart from that, volume of rinsing water in wet magnetic separation processes directly affects the washing quality and so the grade of the concentrate. Fine tuning of the volume of rinsing water should be made to obtain optimum grade and recovery, because excessive volumes cause decreased in recoveries and also too smaller volumes influence the grade of the magnetic concentrate (Likun and Yun, 2010).

2.2.4 Flotation Concentration

Flotation is one of the oldest and most common mineral processing methods for the beneficiation of sulfide, oxide, and industrial minerals. From past to present, its application areas have been continuing to expand, as including beneficiation of sulfide ores, oxide ores, low-grade complex ores, and tailings of mineral processing plants (Rao, 2004; Wills and Finch, 2016). Principally, flotation is the separation of minerals based on the differences of surface properties of minerals, so called wettability (Fuerstenau and Han, 2003). Apart from that, minerals can be classified as

hydrophobic and hydrophilic with respect to wettability of their surfaces. In this context, minerals that repel the water are called hydrophobic; on the contrary particles that make interaction with water are called as hydrophilic (Wills and Finch, 2016). In more details, flotation process contains three phases principally, which are solid, liquid, and gas phases. In order to float the particle, it should be attached to the air bubbles, which represent the gas phase. Naturally hydrophobic particles have more tendency to attach to air bubbles than their hydrophilic counterparts and thus flotation of them is achieved more easily (Wills and Finch, 2016).

In the case of hydrophilic particles, flotation method is focused on making these particles hydrophobic for their attachment to air bubbles. For this, flotation chemicals that affect the surface of particles are used (Fuerstenau and Han, 2003). Prior to briefly explaining these chemicals, it will be useful to explain the mechanism of flotation. It mainly contains three consecutive mechanisms, attachment of desired minerals selectively on air bubbles, dragging of the water by passing through the froth, and the physical retainment between particles in the froth as attached to air bubbles (Wills and Finch, 2016). Mechanism of flotation is given in Figure 2.9.

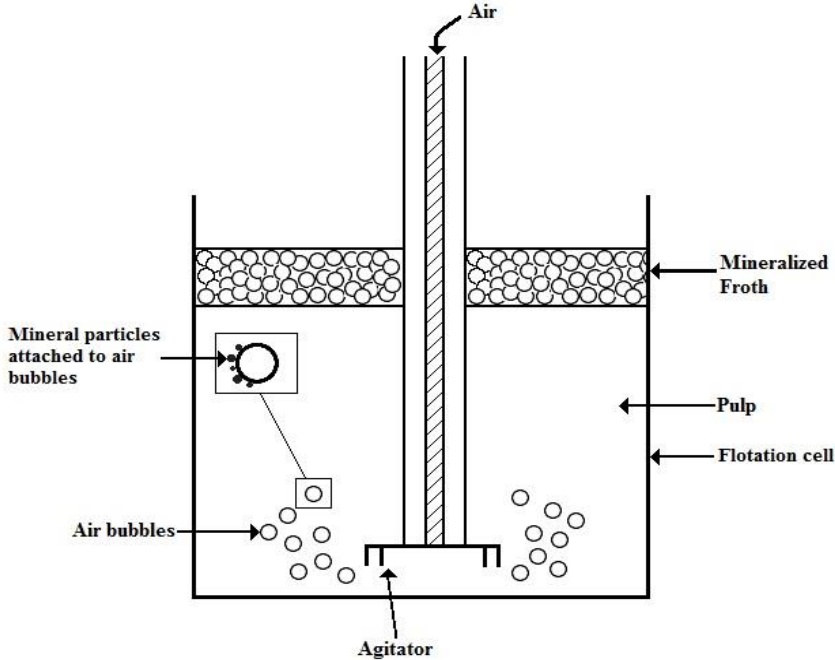


Figure 2.9 Illustration of flotation mechanism

Attachment of mineral particles to air bubbles is mainly related to their hydrophobicity, so called wettability. Regarding this, the main mechanism that causes the presence of wettability is the adsorption of these particles to air bubbles, which is mainly expressed by the contact angle (θ). Contact angle, which is based on the surface tension (γ) due to the interactions between the gas (G), solid (S), and liquid (L), can be calculated by the Young-Dupre equation, given as Equation (4) (Young, 1805).

$$\gamma_{s/g} = \gamma_{s/l} + \gamma_{l/g} \cdot \cos \theta \quad (4)$$

Based on the formation of solid-gas interfaces, the force required for breaking the particle bubble attachment is called the work of adhesion and it is equal to the force required for the separation of the solid-air interface, thus producing air-water and solid-water interfaces. Work of adhesion can be calculated by the following Equation (5 and 6).

$$W_{s/g} = \gamma_{l/g} + \gamma_{s/l} - \gamma_{s/g} \quad (5)$$

$$W_{s/g} = \gamma_{l/g} (1 - \cos \theta) \quad (6)$$

By examining Equation (6), it can be deduced that the work of adhesion between particle and bubble is proportional to the contact angle. In this context, the increase of contact angle also increases the hydrophobicity of the mineral by making the mineral as aerophilic, which means higher affinity for air than water (Wills and Finch, 2016). Interaction between three phases of flotation and the representation of contact angle are given in Figure 2.10.

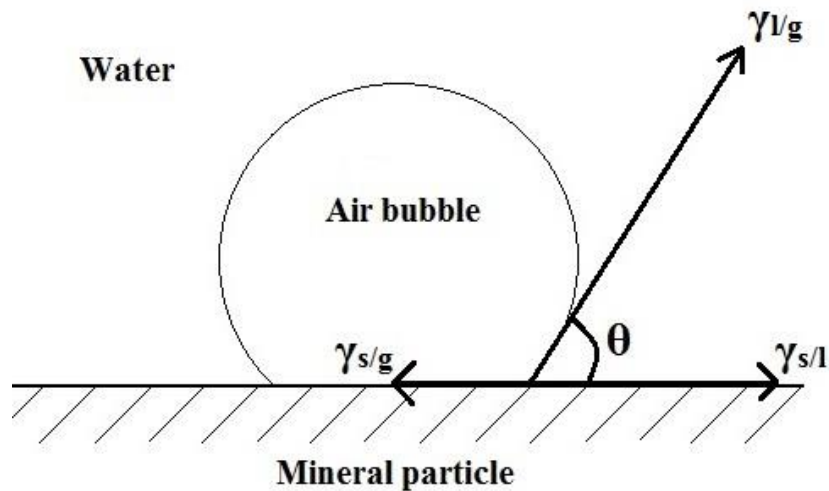


Figure 2.10 Three phases of flotation and contact angle (θ)

Surface properties of minerals, namely hydrophobicity and hydrophilicity, are directly related with the contact angle and so the value of contact angle affects the floatability of a mineral. Therefore, if the contact angle is greater than 90° , the particle can be defined as hydrophobic. On the contrary, for contact angles less than 90° , particle can be referred to be as hydrophilic. In order to change the surface properties of a mineral, changing the surface of a mineral from hydrophilic to hydrophobic and providing the suitable conditions for achieving a successful flotation process, flotation reagents are used (Fuerstenau and Han, 2003). These flotation reagents can be classified into three main groups, as collectors, frothers, and regulators.

- **Collectors:** As aforementioned above, surface of the mineral to be floated should be hydrophobic. In order to make the surface of a hydrophilic mineral hydrophobic, organic type chemical surfactants, namely collectors are used (Wills and Finch, 2016). In other words, collectors provide the selectivity of the separation by forming a hydrophobic layer on the desired mineral surface and providing the particle-bubble attachment. Thus, these minerals can be recovered from the froth product (Bulatovic, 2007). Based on the dissociation characteristics of the collectors, they can be divided into two groups as ionizing and non-ionizing collectors. Ionizing collectors are divided into two sub-

groups as cationic and anionic collectors. Due to the solidophilic properties of anionic collectors, they can be oxhydryl or sulfhydryl type. Cationic collectors, which are mainly composed of amines have protonized hydrocarbon radical structures. Apart from these, non-ionizing collectors are also divided into two sub-groups according to their content of bivalent sulfur and non-polar hydrocarbon oils (Bulatovic, 2007).

- **Frothers:** Principally, frothers, as heteropolar surface-active compounds, enable the adsorption between an air bubble and a water interface by reducing the surface tension of water (Bulatovic, 2007). Frothers have three main functions in the flotation process as, providing stability of froths including prevention of the destruction of small bubbles, lowering the rise velocity of bubbles, and supporting the formation of the stable froth (Klimpel and Isherwood, 1991). Effectivity of frothers is directly related with the pH of the pulp and therefore they can be classified according to the effectiveness at different pH values. For the acidic frothers, frothing ability is decreased by the increased pH. Phenols and Alkylsulfonates are the two sub-groups of acidic frothers. On the contrary, frothing ability of neutral frothers increases with increasing pH. Cyclic alcohols, aliphatic alcohols, alkoxy paraffins, and glycols are the four different subgroups of neutral frothers (Bulatovic, 2007).
- **Regulators:** Mainly composed of activators, depressants and pH regulators are also called modifiers. Theoretically, they have a modifying effect on the action of collectors covering the mineral surfaces. In addition to these, they provide the selectivity and also higher process efficiency. Activators and depressants have a direct effect on the mineral surface for the modification of surface properties of desired or undesired minerals. Namely activators act on the surface of desired particles, by making their surface hydrophobic. On the contrary, depressants are used for the depression of undesired minerals to make a selective separation. Apart from these, pH regulators are used for changing the ionic composition of the pulp by changing the hydrogen ion concentration.

Even though they are used for improving the collector – mineral interactions, they can be also used for the depression of undesired minerals (Bulatovic, 2007).

Following the brief description of flotation theory and flotation reagents, it will be useful to mention flotation practices. Based on the wide application areas of flotation, it is known that flotation is a common method for the beneficiation of rare earth minerals (Anderson *et al.*, 2016). According to the previous studies, flotation was applied on a scandium bearing ultra-basic rock in order to obtain a pre-concentrate enriched in scandium. Within the scope of this study, tailings of magnetic separation that contained scandium was subjected to flotation. Prior to collector addition, pH of the pulp was lowered by adding sulfuric acid, then modified hydroxamate collector (MOH) was added for the flotation of scandium bearing minerals. As the result of flotation, a scandium pre-concentrate containing 350-400 ppm Sc was obtained (China Patent No. CN104263926A, 2014). Although this study seems to be successful in terms of the grade of the concentrate, in the case of scandium bearing clayey ores, application of flotation is expected to be challenging due to presence of excess slimes. Generally, slimes and finer particle size clay minerals both increase the viscosity of the pulp and coat the mineral surfaces so preventing their interactions with collectors and therefore decreasing the recovery and selectivity of separation (Yu *et al.*, 2017; Taner and Onen, 2016). In addition to these, the dispersed state of scandium in these clayey ores also makes the flotation process problematic due to the uncertainty of the desired mineral to be floated.

CHAPTER 3

STATEMENT OF THE PROBLEM

Rare earth metals, namely critical metals, have been in great demand due to their usage in industrial applications and their higher economic return in comparison with the base metals whose prices are falling continuously. Scandium, as one of the most critical rare earth elements, is used in high technology critical applications, such as military applications, aircraft applications, and solid oxide fuel cells. High prices of its end products and lower rate of current production make this superior metal attractive in terms of both economic and technological developments. Due to scarcity of scandium, no single deposit and occurrence of it in dispersed state in ore deposits, recovery of scandium from these deposits is challenging. In addition to the lower grade of the scandium bearing deposits, deposition of the scandium in the clayey minerals and also the usage of hydrometallurgical processes in order to recover the scandium make these processes costly due to the impurities contained in these ores. For these reasons, application of mineral processing techniques to obtain a scandium pre-concentrate by rejecting impurities as much as possible become an important issue during the processing of these types of ores. This study will be unique in terms of filling the gap in the literature on the pre-concentration of scandium containing clayey ores.

The objective of this study is to investigate the possibility of obtaining a scandium (Sc) pre-concentrate from scandium bearing clayey ore by using mineral processing methods. The motivation of obtaining a pre-concentrate is the preparation of a feed relatively enriched in scandium content in order to increase the efficiency and effectiveness of subsequent hydrometallurgical process and thereby decreasing the processing costs. Through pre-concentration, rejection of gangue minerals that would cause negative impacts during hydrometallurgical treatment, at the highest possible extent, is also targeted.

In order to obtain a scandium pre-concentrate, this study is focused on separate but sequential mineral processing unit operation stages. These are;

1. Chemical and mineralogical characterization of the ore sample
2. Attrition scrubbing studies
3. Falcon concentration studies
4. Magnetic separation studies
5. Flotation studies

For each of the above mentioned mineral processing technique, the conditions and parameters that would enable the most effective means of scandium pre-concentration and gangue mineral rejection will be identified and discussed.

CHAPTER 4

MATERIALS AND METHODS

4.1 Scandium Ore Sample

Scandium ore samples from Eskişehir-Mihalıççık were used for the investigation of the pre-concentration possibility of scandium from that types of ores. Prior to the delivery of the samples, the reserve areas were visited and previous geological studies were investigated. According to the on-site observations, the reserve is surrounded by lateritic nickel-cobalt deposits. Previous site investigation studies include drilling operations for the resource estimation; however, related with the clayey structure of the deposit, drill cores could not be obtained appropriately. Therefore, trenches with 5 m depth and 5 m height were excavated at different sites of the reserve in order to take representative samples. Approximately 180 kg of ore was delivered by combining the samples as representative as possible from the different trenches. In addition to these, samples that represent different lithological units were taken from the drill cores for making further geological, geochemical, and mineralogical characterization of the ore deposit.

The present study for investigating the scandium pre-concentration possibility of Eskişehir-Mihalıççık ore through mineral processing methods consisted of six main stages as detailed mineralogical, geochemical and geo-metallurgical characterizations, preparation of representative samples for the pre-concentration studies, pre-concentration of the ore by using different methods, namely attrition scrubbing, falcon concentrator, magnetic separation and flotation, separately but sequentially. Prior to the pre-concentration studies, detailed characterization of the ore samples was performed and also representative samples were prepared.

4.2 Characterization of the Scandium Ore

Scandium bearing clayey ores are rarely seen and lack of detailed information about these ores in the literature make the characterization of these ores more important in terms of the identification of the ore and the selection of the suitable processing methods. For the purpose of identifying the ore in details, characterization of both trench samples and drill core samples were made. Detailed characterization of the trench sample, which were used as a feed material in pre-concentration studies, will be given in the following chapter. In this context, selected drill core samples that represent the different lithological parts of the ore were subjected to detailed characterization studies for getting the basic knowledge about the deposit. These characterization studies include mineralogical investigations by making XRD (X-Ray Diffraction) analysis and geo-metallurgical, geochemical investigations by making elemental analysis using ICP-OES method.

4.2.1 Mineralogical, XRD Analyses of Drill Core Samples

Mineralogical properties of the ore deposit, in order to identify the surface and the compositional characteristics qualitatively, were examined by performing XRD analyses. XRD analyses were used not only for the characterization of the ore but also for the observation and comparison of the differences in the concentrate and the tailing fractions during the pre-concentration studies. For the purpose of getting basic knowledge about the mineralogical properties of the ore deposit, drill cores that represent different lithological parts were used for the XRD analyses. Image of the selected drill core samples are given in Figure 4.1.

For XRD analyses, drill core samples were ground to $-150\ \mu\text{m}$ size and representative portions were taken from each of them, following the drying of samples at $105\ ^\circ\text{C}$ in a laboratory furnace for approximately 24 hours. XRD analyses were conducted using a Bruker D8 Advance XRD device. The analyses were done in $10^\circ - 80^\circ$ range with

1°/min scan rate. X-ray diffractograms of the drill core samples are given in Appendix A (Figure A.1-A.6).



Figure 4.1 Selected drill cores, representing different lithological parts

In order to determine the general mineralogical properties of the Eskişehir-Mihalıççık scandium ore, XRD analyses were made on six drill core samples taken from different lithological parts. Through the XRD analyses, ore minerals were observed and also prior knowledge about the ore was obtained. Drill core samples were coded from 1 to 6 before the XRD analyses as CS1 to CS6. The characteristic peaks and the 2 theta values of the major minerals were mentioned below.

As the result of the XRD analyses, although they were taken from different lithological parts of the ore, it was seen that CS1 and CS3 samples had similar mineralogical properties. In this context, dolomite was the dominating mineral phase in these samples and was responsible for the most intense peak at 31.02° . In addition to this, relatively less intense characteristic peaks of dolomite were observed at 24.08° , 37.43° , 41.21° , 45.01° , 49.33° , 50.61° , 51.15° , 59.93° , 63.51° , and 67.41° . Apart from that, quartz and nontronite (iron rich, smectite group clay mineral) were observed as the dominating

silicate mineral phases. Quartz and nontronite peaks were frequently overlapped and created a moderately intense peak at 26.63°. Quartz peaks with less intensity were observed at 20.85°, 36.54°, 59.98°, 68.13°, and 70.57°. Similarly, peaks of nontronite were observed at 20.85°, 25.55°, 39.48°, 50.17°, and 70.57°. As another clay mineral, moderate intense peaks of sepiolite were observed at 24.08°, 26.63°, and 22.11°. Additionally, as the potential Sc-bearing mineral, pyroxene (so-called pyroxenite, a rock-forming silicate mineral) peaks were observed at 27.96° and 45.51°. As pyroxene, potentially Sc-bearing mineral, characteristic peaks of diopside (Ca, Fe, Mg silicate mineral) with lower intensities were observed. Lastly, presence of (Mg, Al, Fe) silicate was also seen with very low intensity.

X-ray diffractogram of sample CS2 showed that quartz and nontronite were the dominating mineral phases. At that point, peaks of them were frequently overlapped and created the most intense peak at 26.63°. Relatively moderate intense characteristic peaks of quartz were observed at 20.85°, 36.54°, 42.48°, 54.90°, 59.98°, 68.13°, and 70.57°. Also, moderate intense characteristic peaks of nontronite were observed at 20.85°, 25.55°, 39.48°, and 50.17°. Overlapped peak of dolomite and sepiolite was observed at 24.08° with a moderate intense besides the lower intensity peak of dolomite located at 32.93°. In addition to these, low intensity peaks of goethite and (Mg, Al, Fe) silicate were observed. Although the peak of goethite had lower intensity, it was thought that the relatively dark color of the sample was based on the presence of this mineral.

In the sample CS4, as the excessive clay containing lithological part of the ore, montmorillonite (Al, Mg containing, smectite group clay mineral), nontronite, and quartz were observed as the dominating mineral phases. Characteristic peak of montmorillonite was observed at 19.64°. As in the other samples, characteristic peaks of nontronite and quartz were overlapped frequently and created a higher intensity peak at 26.63°. Moderate intense quartz peaks were observed at 20.85°, 36.54°, 54.90°, 59.98°, and 68.13° and also the moderate intense nontronite peaks were observed at 20.85°, 25.55°, 39.48°, 50.17°, and 61.29°. In addition to these, characteristic peaks of

dolomite were observed at 24.08°, 30.97°, 41.21°, 45.01°, 51.15°, 59.93°, and 61.29°. Different than the mineralization of the other samples, moderate intense characteristic peaks of (Na, Al, Fe) silicate were observed at 10.06° and 17.73°. Characteristic peak of sepiolite was observed at 24.08° and overlapped with the characteristic peak of dolomite. Additionally, as the potential Sc-bearing mineral, diopside peaks were observed at 29.50° and 34.76°. As diopside, potentially Sc-bearing mineral, characteristic peak of pyroxene with lower intensity was observed.

For the sample CS5, dolomite, quartz and nontronite were observed as the most dominant mineral phases. In this context, the most intense characteristic peak of the dolomite was located at 31.02°. Relatively less intense characteristic peaks of dolomite were observed at 24.08°, 37.43°, 41.21°, 45.01°, 50.61°, 51.15°, 63.51°, and 67.41°. As in the other samples, characteristic peaks of nontronite and quartz were overlapped frequently and created a higher intensity peak at 26.63°. Moderate intense quartz peaks were observed at 20.85°, 36.54°, 42.48°, 59.98°, and 68.13° and also the moderate intense nontronite peaks were observed at 20.85°, 25.55°, 39.48°, and 70.50°. Characteristic peaks of sepiolite were observed at 32.50° and 24.08°, which was overlapped with the characteristic peak of dolomite. As the potential Sc-bearing mineral, diopside peaks were observed at 29.50° and 34.76°, while pyroxene peaks were observed at 27.96° and 45.51°. In addition to these, calcite and monticellite (Ca, Mg silicate) were observed with less intense characteristic peaks. Lastly, presence of (Mg, Al, Fe) silicate was also seen with very low intensity.

For the sample CS6, quartz and nontronite were observed as the dominant mineral phases. As in the other samples, peaks of them were frequently overlapped and created the most intense peak at 26.63°. Relatively moderate intense characteristic peaks of quartz were observed at 20.85°, 36.54°, 39.50°, 42.48°, 54.90°, 59.98° and 68.13°. Also, moderate intense characteristic peaks of nontronite were observed at 20.85°, 25.55°, and 50.17°. In addition to these, characteristic peaks of dolomite were observed at 24.08°, 45.01°, and 67.41°. The characteristic peak of sepiolite was observed at

24.08° and overlapped with the characteristic peak of dolomite. Lastly, presence of (Mg, Al, Fe) silicate was also seen with very low intensity.

By considering the results of the mineralogical studies on drill core samples, presence of nontronite, montmorillonite, and sepiolite minerals was a strong evidence for the clayey structure of the Eskişehir-Mihalıççık scandium ore. In addition to these, based on the literature information on Sc-bearing possibilities of silicate minerals, namely diopside and pyroxene, (Chasse *et al.*, 2016; Devore, 1955) showed that they could be the main scandium carriers in the ore. On the other hand, based on the possibly lower scandium content of quartz and dolomite minerals it would be accurate to classify them as the main gangue minerals. Thus, the pre-concentration studies were focused on the removal of these calcium, magnesium, and silica containing gangue minerals with the highest possible extent of scandium recovery.

4.2.2 Geo-metallurgical and Geochemical Investigations

In order to determine the general composition of the ore and occurrence state of scandium with regarding to different parts of the ore, selected drill core samples from different lithological parts and also samples taken from a drill hole, which were believed to represent the whole ore deposit, were subjected to elemental analysis by using ICP-OES. According to the geological interpretations, the ore deposit was developed on serpentinites and peridotites that were interlayered in a Jurassic Sedimentary package of limestone and associated sediments. The ore body contains clay, altered serpentinite, silica, limonite, and saprolite as the geological units. The main lithological units of the deposit are clays, altered serpentinite, and silica, so the deposit contains limonite and saprolite to a lesser extent. Scandium content of the deposit fluctuates in a range between 27 and 149 ppm throughout the ore body. That situation also shows that the ore body includes various mineralogical formations. Based on this, analysis results for the scandium content of the different lithological units are given in Table 4.1.

Table 4.1 Scandium amounts with respect to different lithologies

Lithology	Sc (ppm)
<i>Limonite</i>	77
<i>Saprolite</i>	78
<i>Clay</i>	149
<i>Altered Serpentinite</i>	81
<i>Silica</i>	27

As it can be seen from Table 4-1, clay minerals are the main scandium bearing part of the ore with their 149 ppm Sc content. The other main lithological part of the ore deposit, as altered serpentinite that contains 81 ppm Sc. Contrary to these, lower scandium content of silica, as 27 ppm, indicated that silica is the main impurity to be removed during the pre-concentration studies. In addition to these, scandium amount of the representative samples taken from the different depths of a drill hole fluctuates between 44 and 127 ppm, and also it was observed that the scandium amount is not related with depth. By examining the analysis results in more details, it was observed that scandium content of the ore is directly related with its iron content. In other words, scandium amount of the ore has peaked between 27 and 35 meters of depth, while iron content of the ore also making peak by reaching 10.90 %. Apart from these, no correlation between scandium, calcium and magnesium was observed. As a consequence of this, it was thought as a preliminary evidence for the removal of calcium and magnesium during the pre-concentration studies with as possible as minimal scandium loss. Scandium and iron amounts with regarding to the different depths of the drill hole are given in Table 4.2. Detailed analysis results are also given in Appendix B (Table B.1).

Table 4.2 Scandium and iron content of the core samples

Drill Core Sample No	Start (m)	End (m)	Interval (m)	Sc (ppm)	Fe (%)
1	1.00	16.40	15.40	90	6.07
2	16.40	19.40	3.00	58	3.84
3	19.40	23.60	4.20	87	5.31
4	27.00	35.00	8.00	127	10.90
5	37.00	53.70	16.70	76	6.01
6	58.00	67.00	9.00	44	4.09

4.3 Preparation of Representative Samples (Feed) for the Pre-Concentration Studies

For the representation of the whole ore deposit, approximately 180 kg of samples, taken from different trenches opened on the site, were used in order to prepare representative samples for the pre-concentration studies. After obtaining the samples, it was observed that the moisture content of the ore was relatively higher due to the clay content of the ore, so they were laid out on a smooth floor for drying at atmospheric conditions. Prior to drying of the sample, representative samples were taken for the moisture content analysis.

In order to determine the moisture content of the ore sample, moisture content analysis were performed according to ASTM D2216 – 10 standards. For these tests, 50 grams of representative samples were used and 6 parallel analysis were conducted. Briefly, each representative sample was placed into the container and total weight was recorded. Following this, the container was placed into a laboratory furnace, which was adjusted to 105 ± 5 °C. After periodic weight measurements, samples that reached constant weight values were taken out from the furnace and the moisture content of them were calculated by using the following Equation (7);

$$w = [(M_{cms} - M_{cds}) / (M_{cds} - M_c)] * 100 = (M_w / M_s) * 100$$

w = moisture content, %,

M_{cms} = mass of container + moist sample, g,

M_{cds} = mass of container + oven dry specimen, g, (7)

M_c = mass of container, g,

M_w = mass of water ($M_w = M_{cms} - M_{cds}$), g,

M_s = mass of oven dry sample ($M_s = M_{cds} - M_c$), g

As a result of the moisture content analysis, average moisture content of the ore was found as 24.38 %. It was thought that such high moisture content was based on the clay content of the ore. Constructed weight – time graphs for determining the moisture content of samples are given in Figure 4.2.

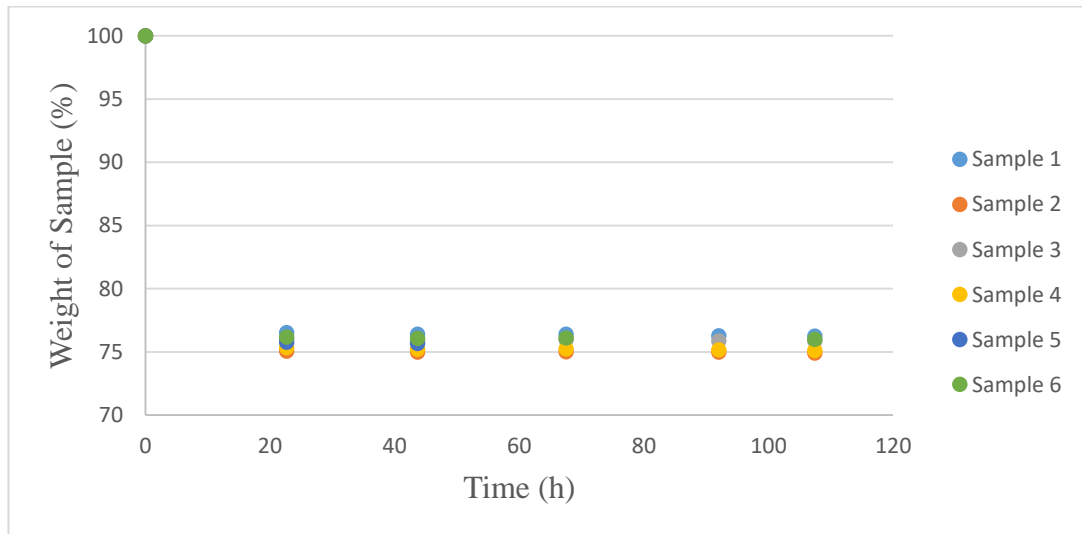


Figure 4.2 Weight of sample (%) versus time (h) graph for the determination of the moisture content

After drying of the samples at atmospheric conditions, they were entirely subjected to particle size reduction by coarse crushing using jaw crushers. Although the samples have already fine particle size distribution, coarse crushing was performed in order to reduce the size of relatively bigger rock chips. Following the size reduction of the samples, crushed ore was put into a cone-shaped ore pile for representative sampling

by applying cone and quartering method. Sampling by using cone and quartering method was performed until 5 kg representative samples were obtained (Figure 4.4). Each 5 kg portion was representatively sampled by using the Jones Riffle separators. As the result of this, representative samples weighing approximately 650 gr were obtained for using as the feed materials of the pre-concentration tests. Representative sampling flowsheet is given in Figure 4.3.

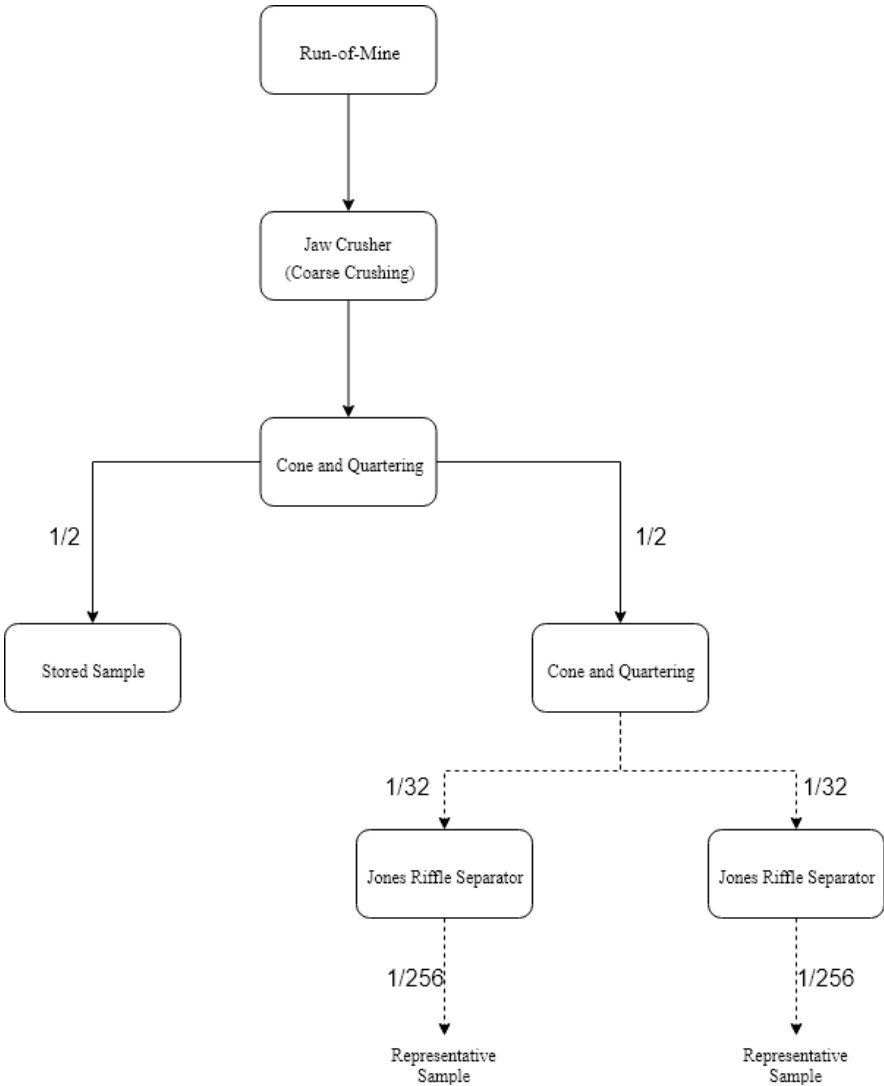


Figure 4.3 Representative sampling flowsheet



Figure 4.4 Preparation of Eskişehir-Mihalıççık ore for pre-concentration studies: a) Run-of-mine after drying, b) Coarse crushing of the run-of-mine, c) Dividing the ore pile into representative fractions by cone and quartering, d) Representative sampling of the sample by using Jones Riffle separator for obtaining smaller representative fractions

4.4 Pre-Concentration Studies

4.4.1 Attrition Scrubbing

As the initial step of pre-concentration studies, representative samples were subjected to attrition scrubbing, in order to liberate the clay minerals, which were already interlocked and covered the rock chips. In this context, the main purpose of attrition scrubbing was the removal of the undesired gangue particles, containing calcium, magnesium and silicon, and also obtaining a pre-concentrate enriched in scandium. The attrition scrubbing process has six important operational parameters, these are

solid-liquid ratio, residence time, rotation speed, feed particle size, temperature, and particle porosity. The fact that, temperature is important when organic material presents in the feed sample (Boundy *et al.*, 2017; Bayley and Biggs, 2005) led to the, experiments being conducted at room temperature. In addition to these, according to the previous studies on attrition scrubbing, it was shown that feed particle top sizes smaller than 6 mm are found to be appropriate (Hassan and Abdel-Khalek, 1998; Özbayoğlu and Atalay, 2000; Birinci and Sarıkaya, 2004) so, that the feed material was not exposed to further size reduction due to the finer particle size distribution. Also based on the presence of clay minerals in the ore, it was thought that the ore has higher porosity and the clay minerals could be liberated by applying attrition scrubbing. So, the present study was focused on the effect of solid-liquid ratio, residence time, and rotation speed.

Attrition scrubbing experiments were conducted by using an attrition scrubber. A Booth laboratory type attrition scrubber machine (71-220) was used for the attrition scrubbing experiments. The machine has an adjustable agitator rotor, agitation shaft with 3 gentle impellers, and a rigid plastic container that has a capacity of 5 L. Image and cross sectional view of the laboratory type attrition scrubber are given in Figure 4.5.

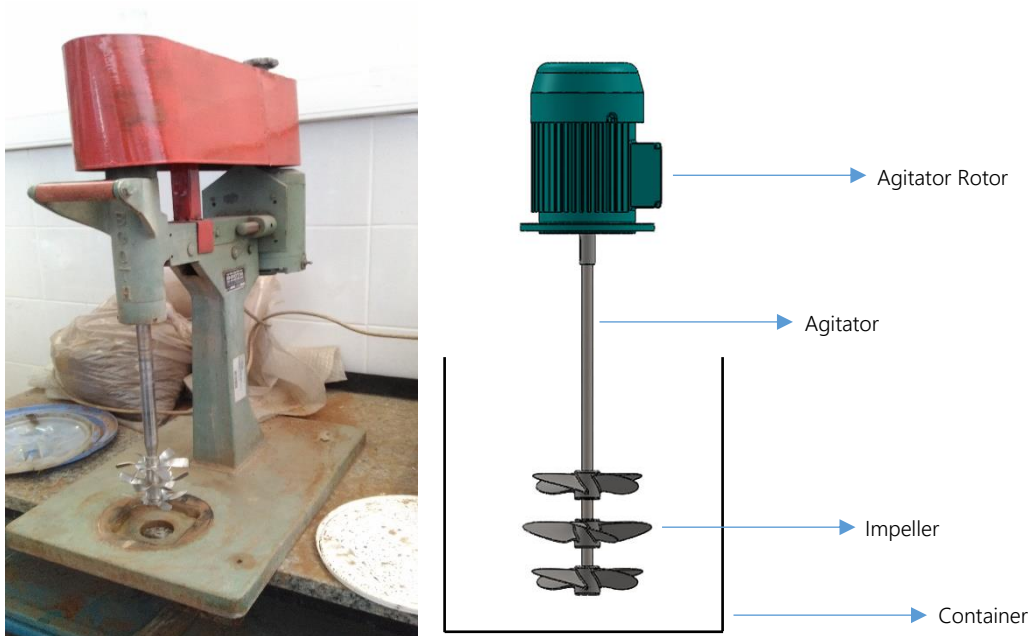


Figure 4.5 Laboratory type attrition scrubber and cross sectional view of it

During the experiments, previously prepared representative samples were used as the feed material. After pouring the representative sample into the tank of the scrubber, required amount of tap water, according to the calculated pulp density was added. In order to provide the desired rotation speed, agitation rate was adjusted via the agitation valve and the rate of agitator was measured by using rotameter. Following these, the sample was subjected to scrubbing at desired amount of retention time. At the end of the retention time of the experiment, the scrubber was switched off and the material plastering on agitator shaft was gently washed into the tank of the scrubber. For the classification of the attrition scrubbed ore, wet sieving was performed with the selected optimum size. Finally, both oversize and undersize material obtained from the classification, were dried in a laboratory furnace at 105 °C for further analysis. After drying, weights of the products were recorded and prepared for the ICP-OES analysis. In this context, material balance tables for each test were constructed and also metallurgical performance of each test was evaluated in terms of grade and recovery values.

Initially, preliminary tests of attrition scrubbing were made for the selection of optimum parameter ranges. Correspondingly, optimization of the attrition scrubbing process parameters, in terms of grade and recovery of the concentrate and also the removal rate of gangue minerals, were made by using statistical modeling.

4.4.1.1 Optimization of the Attrition Scrubbing Process by Statistical Modeling

Following the determination of the most favorable attrition scrubbing operating parameters range, namely solid-liquid ratio, retention time, and rotation speed, for Eskişehir-Mihalıççık scandium ore, the process was further optimized using statistical modeling and a systematic design of experiments, including the variations in aforementioned parameters. In this point of view, attrition scrubbing experiments were designed according to the two level full factorial statistical design (Box, Hunter, & Hunter, 2005). For the statistical design, effects of three above mentioned parameters of attrition scrubbing were studied. The details of statistical modeling and its pattern are given the Chapter 5. In order to optimize the attrition scrubbing process, single stage batch attrition scrubbing tests were conducted based on the previously determined optimum parameter range. As the result of the statistical modeling, the optimum parameters that provides the maximum amount of removal of gangue particles with as minimal scandium loss as possible were determined. In other words, the efficiency of the attrition scrubbing process was evaluated in terms of rate of the gangue particle rejection, scandium recovery and grade of the pre-concentrate and tailings.

4.4.2 Enhanced Gravity Separation by Using Falcon Concentrator

Based on the fundamental principles of the enhanced gravity separation methods, falcon concentrator was used as the second processing method following the attrition scrubbing of the Eskişehir-Mihalıççık scandium ore. In simple terms, enhanced gravity separation was used for the recovery of the fine and ultrafine particles under the influence of very high gravitational forces by the effect of increased initial settling

velocities of finer particles (El-Midany and Ibrahim, 2011; Boylu, 2013). In this context, the main reason of selecting falcon concentrator was its ability to increase the gravitational field due to the applied intense centrifugal forces and it is the gravity separator which has the lowest effective separating density. Therefore, based on the finer particle size and clay composition of the ore, falcon concentrator was used for the separation of clay minerals from their silicon, magnesium, and calcium counterparts, which have relatively higher density and particle sizes. Falcon concentrator study was mainly focused on the separation of clay minerals and gangue minerals, containing Ca, Mg, Si, rather than obtaining a pre-concentrate enriched in scandium with higher grades due to the lower upgrading ratios of the falcon concentrator.

The falcon concentrator has four important operational parameters, these are centrifugal force, fluidization water pressure, feeding rate and density of the slurry and feed particle size. The density of the slurry has a significant effect on the efficiency of the process by affecting on the settling conditions of particles defined as free settling or hindered settling. Above a certain value of solid content of the feed slurry, desired dense media was formed due to the increasing viscosity effect of the fine and ultrafine clay particles. In this case, viscous pulp leads to the occurrence of hindered settling conditions, which also provided the recovery of heavier but finer particles on the heavier fraction. By that way, effective separation density is decreased and the density difference of particles becomes more important than the size difference of particles during the separation. According to the previous studies on the falcon concentration process, in the presence of ultrafine slime particles, hindered settling conditions were provided successfully at solid concentrations above 20 % (Boylu, 2013; Zhang, et al., 2011). Thus, in the present study solid concentration of the feed slurry was fixed at 20 % through the falcon concentration experiments, which was thought to be enough for providing viscous pulp required for the hindered settling conditions. Due to the limitations of the falcon concentrator that was used in the experiments, feeding rate of the slurry was manually adjusted and tried to be finely tuned and fixed, in order to prevent immediate entrapment of lighter particles on the wall of the bowl, during the

experiments. By considering the finer particle size of the feed material obtained as the result of classification after the attrition scrubbing process, and coarser particle size of the heavier particles, namely gangue minerals, it was thought that the efficiency of the falcon concentrator is increased based on the increase in capture sites. Thus, any further size reduction was not made before the falcon concentrator experiments. Based on these, the present study was focused on the effect of centrifugal force and fluidization water pressure of falcon concentrator on the scandium ore.

Enhanced gravity separation experiments were conducted by using Falcon Concentrator. A Falcon SB-40 type laboratory semi-batch concentrator was used during the experiments. The machine has an adjustable rotor that provides the fast spinning of the bowl, which also has fixed orifices located on the walls of it, and a manometer for adjusting the fluidized water pressure. In this context, frequency values generated by the rotor and their corresponding centrifugal force (g) values are given in Table 4.3. Based on the fundamental principles of this machine, it was operated as semi-batch with having a heavier fraction collection slot and a discharge opening slot located at the outside of the bowl for the collection of lighter fraction. Images of the laboratory type Falcon SB-40 concentrator are given in Figure 4.6.

Table 4.3 Frequency values (Hz) and their corresponding centrifugal force values (g)

Frequency (Hz)	Centrifugal force (g)
20	20
25	31
30	44
35	60
40	78
45	99
50	123
55	148
60	176
68	225
71	250
75	275
78	300

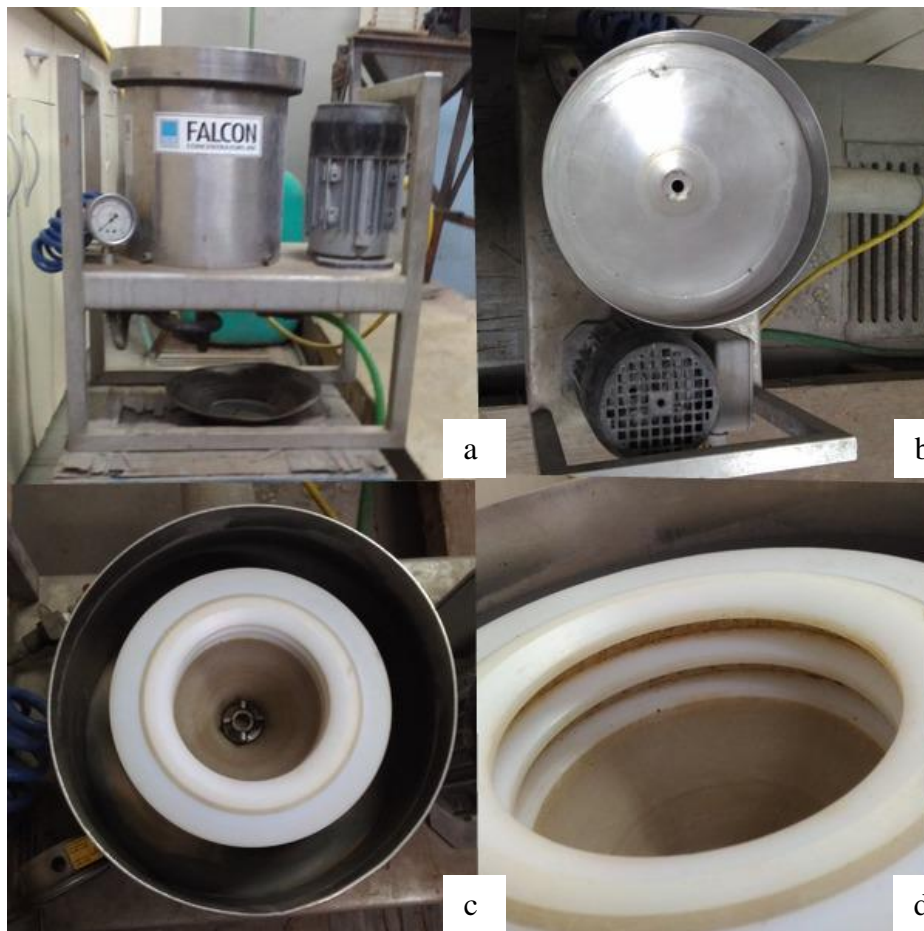


Figure 4.6 a) Cross-sectional view of falcon concentrator b) Top view of falcon concentrator c) Spinning bowl of falcon concentrator d) Orifices located on the walls of the bowl

For the falcon concentrator experiments, undersize of the classification of the previously attrition scrubbed samples were used as the feed material. Due to the samples being dried after the classification, prior to each falcon concentrator experiment, samples were mixed with water in order to make a pulp at desired solid concentration ratio as 20 %. Prepared pulp was agitated by using magnetic stirrers for approximately 4 hours without changing the particle size distribution of the material, which was also determined as the suitable time period that was required for the dispersion of agglomerates originated by the clay content of the ore. After adjusting the desired frequency of the rotor and fluidized water pressure, agitated pulp was fed into the falcon concentrator bowl through the feed inlet pipe. As the result of the

preliminary tests, amount of sample that was favorable for the stratification cycle of the falcon concentrator was determined as 250 gr, so that amount of representative samples were used during each experiment. At the end of each experiment, fluidized water and the rotor of falcon concentrator were switched off sequentially. Thereby, due to the reason that lighter particles were already collected from the upper discharge part of the falcon concentrator bowl, heavier particles were collected from the bottom part and also from the walls of the bowl. In these circumstances, it was expected to be that clay minerals, enriched in scandium, were collected at lighter fraction and also the relatively heavier gangue minerals, containing main impurities such as Ca, Mg and Si, were removed with the heavier fraction.

Finally, both heavier and lighter fractions, obtained from the falcon concentrator, were dried in a laboratory furnace at 105 °C for further analysis. After drying, the weights of the products were recorded and prepared for the ICP-OES analysis. In this context, material balance tables for each test were constructed and also metallurgical performance of each test was evaluated in terms of grade and recovery values. Correspondingly, in order to obtain a scandium pre-concentrate by using falcon concentrator, the parameters that provide the optimum conditions, for the removal of gangue minerals and the recovery of scandium with the highest possible grade were found.

4.4.3 Magnetic Separation

As an alternative method to enhanced gravity separation by using falcon concentrator, magnetic separation method was tried as the second processing method following the attrition scrubbing of the Eskişehir-Mihalıççık scandium ore. Briefly, magnetic separation method was used in order to separate magnetic and non-magnetic parts of an ore. By considering the magnetic properties of ore minerals, ferromagnetic, and paramagnetic minerals are generally enriched in magnetic fraction and the diamagnetic minerals are readily removed in the non-magnetic fraction. Based on the differences of magnetic properties of minerals, in the present study, magnetic separation was

applied for the recovery of Sc-bearing paramagnetic minerals, while removing diamagnetic gangue minerals containing Ca, Mg, and Si in the non-magnetic fraction. According to the previous studies of magnetic separation on scandium containing clayey ores, it was seen that Sc-bearing paramagnetic minerals, as biotite, tremolite and ilmenite were able to concentrate in magnetic fraction and also their diamagnetic counterparts, quartz and serpentine, were removed in the non-magnetic fraction (Likun and Yun, 2010). Therefore, based on the mineralogical properties of Eskişehir-Mihalıççık scandium ore, it was thought that the diamagnetic gangue minerals, such as quartz and Ca, Mg contained minerals, could be removed by recovering scandium in the magnetic concentrate. In this context, high intensity wet magnetic separation was conducted in order to recover paramagnetic minerals by using the previously attrition scrubbed ore samples as the feed material.

The high intensity wet magnetic separation process has two important operational parameters, namely magnetic force that is generated by the applied magnetic field and the volume of rinsing water for both the elution and the cleaning of the magnetic concentrate. Although the fact that solid-liquid ratios of the feed slurries may also be important for the efficiency of the wet magnetic separation processes, solid-liquid ratio of the feed slurry was kept constant during the experiments as it was considered that the required liberation degree of particles was already provided. Thus, solid concentration of the feed slurry was fixed at 20 % during the magnetic separation experiments. By the way, particle size distribution of the feed sample was thought to be finer enough for the magnetic separation process. Apart from these, due to the limitations of the magnetic separator, rinsing water was added manually by trying to be finely tuned, so the present study was not focused on the volume and flow rate of the rinsing water. Based on these, the present study was focused on the effect of magnetic field, so called the magnetic force, on the recovery and grade of scandium in order to obtain a pre-concentrate.

Magnetic separation experiments were conducted by using laboratory type high intensity wet magnetic separator. A laboratory type Carpco High Intensity Wet Magnetic separator was used during the experiments. The machine has an adjustable current generator that provides the desired magnetic field between the plates of the magnetic separation chamber. Current values generated by the current generator and their corresponding magnetic field values are given in Figure 4.7. In order to provide the retainment of paramagnetic and ferromagnetic minerals on the separation chamber under the effect of generated magnetic field, chrome-steel balls, having a diameter of 6 mm were used as the magnetic separation media. Based on the operating principles of the machine, it was operated as batch and so the magnetic chamber was cleaned by rinsing at certain time periods. In addition to these, magnetic and non-magnetic fractions were collected from the same slot sequentially. In the case of the desired magnetic field being generated, diamagnetic particles could not be retained in the magnetic chamber and discharged from the bottom part of the chamber as non-magnetic fraction. By rinsing of the spinning chamber gently while the magnetic field had already been generated, non-magnetic particles that covers the retained magnetic particles were removed through the bottom discharge opening. Following this, magnetic field was closed and the magnetic particles that retained on the chamber were readily collected from discharge opening under the effect of the force applied by the rinsing water. Images of the laboratory type Carpco High Intensity Wet Magnetic Separator are given in Figure 4.8.

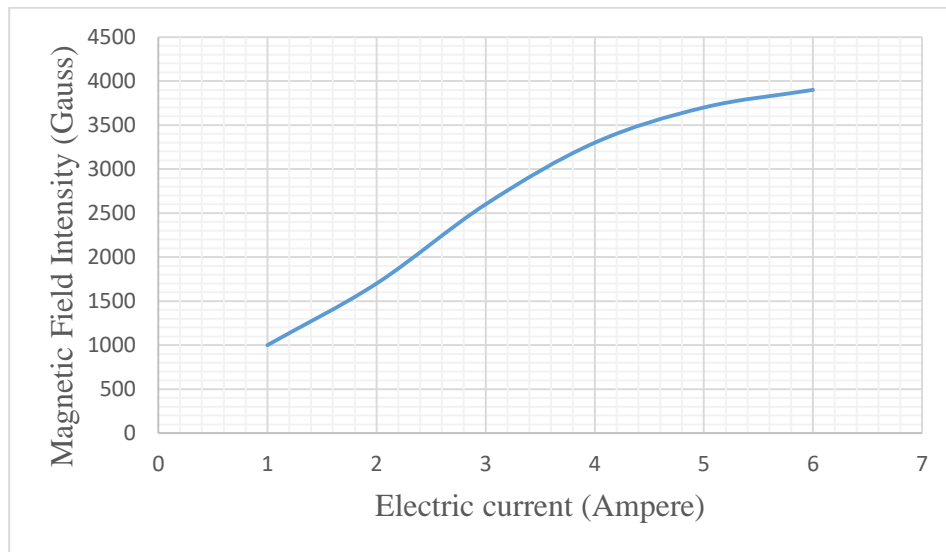


Figure 4.7 Generated electrical current values (A) and their corresponding magnetic field intensities (G)

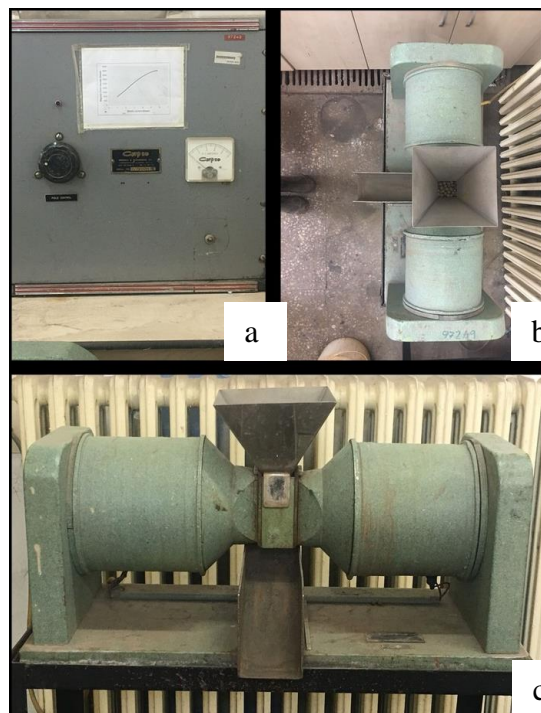


Figure 4.8 a) Current generator of Carpco Magnetic Separator b) Top view of Carpco Magnetic Separator c) Cross-sectional view of Carpco Magnetic Separator

For the magnetic separation experiments, undersize of the classification of the previously attrition scrubbed samples were used as the feed material. As the samples were dried after the classification, prior to each magnetic separation experiment, samples were mixed with water in order to make a pulp at desired solid concentration as 20 %. Prepared pulp was agitated by using magnetic stirrers for approximately 4 hours, without changing the particle size distribution of the material, which was also determined as the suitable time period that was required for the dispersion of agglomerates, originated by the clay content of the ore. After adjusting the desired magnetic field intensity, the agitated pulp was fed into the chamber of the magnetic separator through the feeding cone. In the course of each experiment, chamber of the magnetic separator was rinsed by water gently without affecting the retained magnetic particles, and also the electric current generator could be stopped regarding the occupancy rate of the chamber and the magnetic concentrate was collected by regenerating the chamber concurrently. At the end of each experiment, magnetic separator was switched off and the magnetic concentrate was collected sequentially. As the result of the magnetic separation experiments, it was expected that the Sc-bearing paramagnetic clay minerals were concentrated in the magnetic fraction and also the diamagnetic gangue minerals, containing impurities such as Ca, Mg, and Si were removed with the non-magnetic fraction.

Finally, both magnetic and non-magnetic fractions, obtained from the magnetic separator were dried in a laboratory furnace at 105 °C for further analysis. After drying, the weights of the products were recorded and prepared for the ICP-OES analysis. In this context, material balance tables for each test were constructed and also metallurgical performance of each test was evaluated in terms of grade and recovery values. Correspondingly, possibility of obtaining a scandium pre-concentrate by using high intensity wet magnetic separator was evaluated and if possible, the parameters that provide the optimum conditions for the removal of the gangue minerals and the recovery of scandium with the highest possible grade were found.

4.4.4 Flotation

For obtaining a pre-concentrate enriched in scandium, flotation method was tried as the second processing method following the attrition scrubbing of the Eskişehir-Mihalıççık scandium ore, which was thought as an alternative method to enhanced gravity separation and magnetic separation. In addition to these, separation of gangue minerals containing Ca, Mg, Si from the Sc-bearing minerals were also targeted during the flotation studies. In this context, for the flotation concentration of such clayey ores as in the present study, the presence of ultra-fine slimes and instead of the presence of scandium minerals, presence of scandium as dispersed state in clayey minerals seems to make the flotation concentration of the ore challenging.

Based on the previous studies, flotation experiments were conducted by using different types of collectors in order to obtain a scandium pre-concentrate. According to these previous studies, a scandium pre-concentrate containing 350-400 ppm Sc was obtained by lowering the pH with sulfuric acid prior to the addition of hydroxamate type collector (China Patent No. CN104263926A, 2014). Therefore, it was thought that the Sc-bearing minerals could be recovered in the flotation concentrate while the gangue minerals could be removed in the tailings of the flotation. In the present study, different ionizing collectors of hydroxamate and sulfonate based types were used in order to recover the iron containing clay minerals and so recovering the Sc-bearing minerals. As a dispersing agent, sodium silicate (Na_2SiO_3) was used for both depressing Ca, Mg, and Si containing gangue minerals and providing the sufficient degree of liberation. pH of the pulp was adjusted by using sulfuric acid and the type and amount of the frother was kept constant during the study. Technical grade reagents and chemicals used in flotation experiments are listed in Table 4.4.

Flotation experiments were conducted by using a laboratory scale Denver type flotation machine and a one-liter cell. Image of the flotation machine is given in Figure 4.9. Flotation experiments were made by using undersize of the classification of the previously attrition scrubbed samples as the feed material. For the each test, 350 grams

of representative sample was used and the solid content of the pulp was adjusted as 35 %. Based on the finer particle size distribution of the feed material, it was thought that further grinding was not necessary. During the experiments, after the pulp at the desired solid-liquid ratio was obtained, pH of the pulp was adjusted by using sulfuric acid and then sodium silicate was added as a dispersing agent. Following the conditioning of the pulp for a certain period of time, the collector was added and again waited for the conditioning of the pulp. Finally, prior to the aeration, frother was added and after that collection of the froths which was carrying the pre-concentrate enriched in scandium, was aimed. As a consequence of the flotation experiments, it was expected that the Sc-bearing minerals were recovered in the concentrate and also the gangue minerals, containing impurities such as Ca, Mg, and Si were removed with the tailings.

Finally, both concentrate and tailing, obtained from the flotation, were dried in a laboratory furnace at 105 °C for further analysis. After drying, the weights of the products were recorded and prepared for the ICP-OES analysis. In this context, material balance tables for each test were constructed and also metallurgical performance of each test was evaluated in terms of grade and recovery values. Correspondingly, possibility of obtaining a scandium pre-concentrate by using flotation method was evaluated and if possible, the parameters that provide the optimum conditions for the removal of gangue minerals and the recovery of scandium bearing minerals with the highest possible grade were found.

Table 4.4 Chemicals and reagents used during the flotation experiments of the study

pH Modifiers	Depressants	Collector (Promoter)	Frother
Sulfuric Acid (H ₂ SO ₄) (Merck)	Sodium Silicate (Na ₂ SiO ₃) (Merck)	Aero 6493 (Hydroxamate Based) (Cytec) Aero 866 (Sulfonate Based) (Cytec)	F-533 (Cytec)



Figure 4.9 Laboratory type Denver Flotation Machine

CHAPTER 5

RESULTS AND DISCUSSION

5.1 Characterization of the Scandium Ore Samples

Characterization of the material, namely including ore, mineral and etc. is the first step for a properly conducted mineral processing study. According to the characterization results and types of characterization methods, an accurate process route for a material could be chosen and also the process could be fictionalized. In the simplest form, characterization is the identification process of ore, therefore an accurately conducted characterization study prior to the mineral processing studies increases the process efficiency and also plays a crucial role in the determination of the most suitable and economic process. The characterization method that will be performed can be differentiated based on the type of the ore to be processed and the type of the process to be foresighted.

By considering that each ore is also differentiated even within itself, the importance of characterization is better comprehended. This case also shows that the characterization study is required to be done prior to the mineral processing study even for the ores that are very common in nature and have many previous characterization studies. In the context of the present study, characterization of the clayey scandium ore, which has a complex and variable structure, was carried out and also it was thought that the characterization study seems to be a guide for future studies as there were no previous studies about the characterization of this type of ores.

In the present study, characterization of the Eskişehir-Mihalıççık scandium ore sample was performed in order to identify the mineralogical, chemical, physical and thermal properties of the ore and also the minerals of it. During the detailed characterization

studies, representative samples of trench samples, which were the feed material in pre-concentration studies, were used. Elemental analysis were done to investigate the chemical composition of the ore in a quantitative manner by using the Inductive Coupled Plasma – Optical Emission Spectrometry (ICP-OES) method. Mineralogical, XRD (X-Ray Diffraction) analysis were performed to identify the surface and the compositional characteristics of the sample qualitatively. Based on the combination of the results obtained from these two analyzes, the content of the pre-concentration study were started to be embodied. In addition to these, specific gravity determination, by using helium pycnometer, and thermogravimetric and differential thermal analysis, by using TGA-DTA analysis system, were performed in order to identify the physical and thermal properties of the ore sample. Moreover, size classification of the ore was made, by applying wet sieving, for the purpose of identifying the weight distributions and elemental distributions with respect to the sizes of the particles. Development of the pre-concentration methods to be applied during the study was based on the results obtained from the characterization study.

5.1.1 Chemical Analysis

As the first step of characterization, previously prepared representative trench samples of Eskişehir-Mihalıççık scandium ore were used for the chemical analysis in order to examine the ore samples in more detail and to determine the head assays. In the context of the chemical analysis, elemental analysis were conducted by using ICP-OES method and general composition of the ore was determined. The elemental analysis results of the representative ore sample, which was used as the feed material during the pre-concentration studies is given in Table 5.1.

Table 5.1 Elemental analysis of the representative trench sample

Sample Code	Al (%)	Ca (%)	Co (ppm)	Cr (ppm)	Fe (%)	Mg (%)	Ni (ppm)	Sc (ppm)	Si (%)
Sarıçtepe Head	3.61	4.44	77	3500	8.01	5.16	668	105	24.45

Chemical analysis results of the ore sample showed that the scandium content of the ore is 105 ppm. By comparing the scandium content of the ore with respect to the scandium content of the Earth's crust, it can be inferred that the deposit has a relatively higher scandium content. In terms of the nickel and cobalt content of the ore, it was thought that the grades of these are lower for obtaining a feasible pre-concentrate or concentrate. So it was also proven that the nickel and cobalt content of the ore were too low for the deposit to be a laterite deposit. By considering the previous mineralogical investigations, the deposit was possibly classified as a clayey deposit.

Another prominence point of the chemical analysis results was the relatively higher iron content of the ore as 8.01 %. Based on this, it was thought that ore could be included into a classification of iron-bearing clay minerals. It was also thought that the aluminum content of the ore was derived from these clay minerals. Therefore at that stage of the study, iron and aluminum were not seen as the main impurities because of the possible similar behaviors of iron, aluminum and scandium. Detailed investigations about this situation were given in the wet sieving analysis part.

In terms of the impurity content of the ore sample, silicon, magnesium and calcium were thought as the main impurities to be removed during the pre-concentration studies. Higher silicon content of the ore as 24.45 % showed that silicon was not only originated from the silica mineral, but also from the clay minerals. Based on the previous elemental analysis, scandium content of the silica mineral was determined as 27 ppm that makes silica one of the most significant impurity to be removed during the pre-concentration studies. Relatively higher content of calcium and magnesium as 4.44 % and 5.16 % proved the presence of carbonaceous minerals. Due to the higher reagent consumptions of these carbonaceous minerals in the subsequent hydrometallurgical processes, removal of them prior to these processes was considered as crucial. In addition to these, it was also considered that the removal of Si, Ca, and Mg bearing impurities provided a relative enrichment of Sc in the obtained pre-concentrate.

5.1.2 Mineralogical, XRD Analyses

Mineralogical, XRD analysis of the Eskişehir-Mihalıççık representative ore sample showed that quartz, nontronite and diopside were the dominating mineral phases. By investigating the X-ray diffractogram of the representative trench sample, diopside was frequently overlapped with augite and created one of the most intense peak at 30.35° . Relatively moderate intense peaks of diopside were observed at 29.50° , 34.76° , 35.70° , 39.19° , 41.95° , 44.43° , and 57.01° . Apart from that, quartz and nontronite peaks were also frequently overlapped and responsible for the other most intense peak at 26.63° . Relatively moderate intense characteristic peaks of quartz were observed at 20.85° , 36.54° , 42.48° , 59.98° , and 68.13° . Also, moderate intense characteristic peaks of nontronite were observed at 19.94° , 20.85° , 25.55° , 39.48° , 50.17° , 61.29° , and 68.13° . In addition to these, characteristic peaks of dolomite were observed at 30.97° , 45.01° , and 50.45° . Moderately intense characteristic peaks of augite (calcium, magnesium, iron, aluminum containing pyroxene mineral) were observed at 27.96° , 29.50° , 39.19° , 41.95° , 57.01° , and 59.16° . Moreover, the characteristic peak of pyroxene was overlapped with the characteristic peak of augite at 27.96° . As another smectite group clay mineral as nontronite, moderate intense characteristic peaks of montmorillonite were observed at 12.46° and 19.94° . Furthermore, lower intensity characteristic peaks of sepiolite and calcite were observed. X-ray diffractogram of the representative trench sample is given in Figure 5.1.

In addition to the XRD analysis of the representative trench sample, due to the clayey structure of the ore sample, previously prepared representative ore samples were dispersed by the help of an agitator for obtaining the particles under $10\ \mu\text{m}$, which was thought as the most intensely clay containing fraction, via sedimentation by using Stoke's Law. Following the drying of the obtained fraction, XRD analysis were conducted in order to determine the clay minerals of the ore sample in more detail. X-ray diffractogram of the minus $10\ \mu\text{m}$ fraction is given in Figure 5.2.

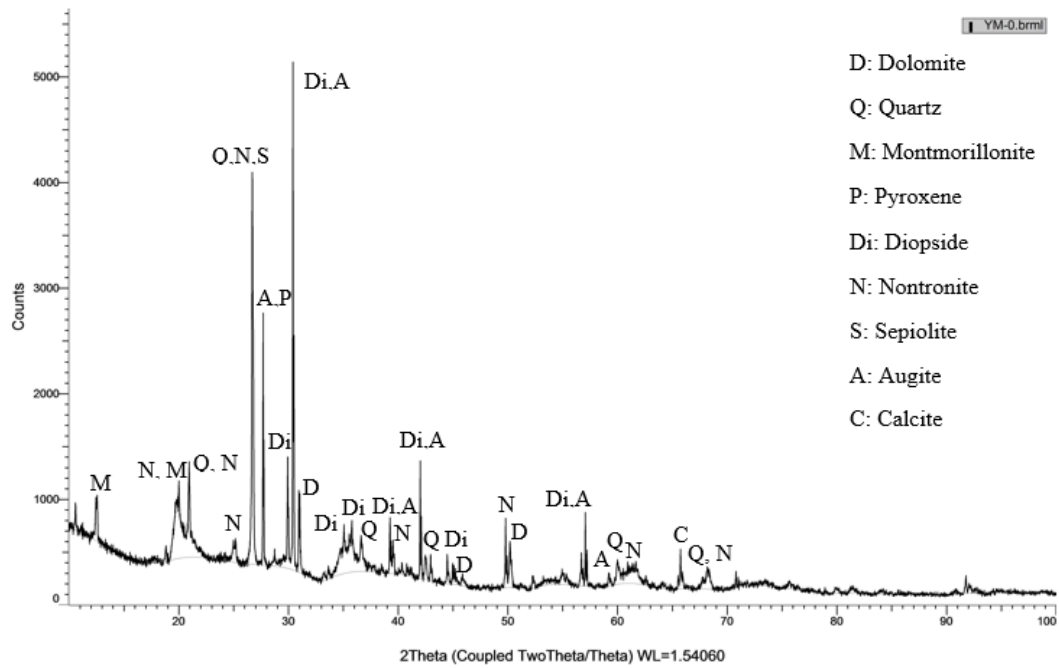


Figure 5.1 X-ray diffractogram of the representative trench sample

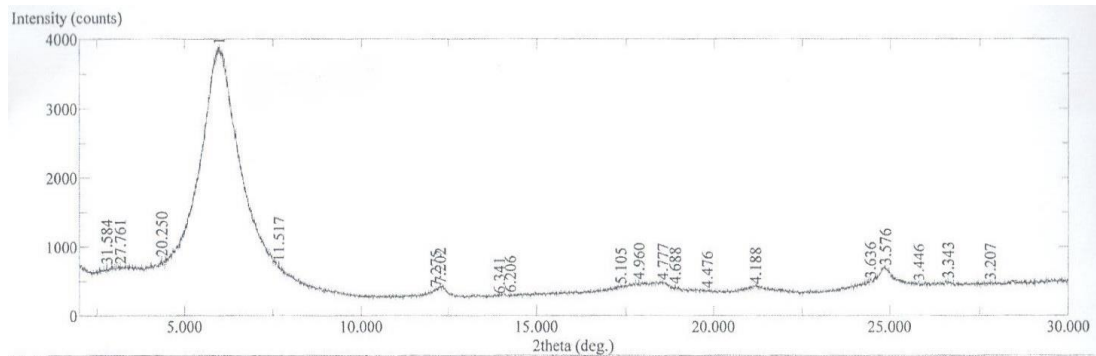


Figure 5.2 X-ray diffractogram of the clay minerals fraction

XRD analysis of the clay minerals fraction was done in 0° - 30° range with $0.5^\circ/\text{min}$ scan rate. As the result of the conducted XRD analysis, obtained X-ray diffractogram showed that the calcium smectite minerals were the dominating mineral phase and were responsible for the most intense peak at 5.985° . On the other hand, smectite group includes clay minerals such as montmorillonite and nontronite, thus it can be seen that the obtained results are consistent with the previous XRD analysis of the representative

ore sample. Apart from these, it was observed that the clay minerals fraction also included plagioclase (feldspar mineral), dolomite, and quartz to a lesser extent.

By the combination of the characterization results of the chemical and mineralogical analysis of the representative trench sample, it was seen that the ore sample not only contains iron-rich clay minerals, such as nontronite and montmorillonite, but also it contains carbonaceous minerals, such as calcite and dolomite. Moreover, it was observed that the relatively higher silicon content of the ore was originated from quartz, clay minerals and also silicate minerals, such as pyroxene, augite and diopside. In the present case, it was considered that the scandium content of the clay minerals, such as nontronite and montmorillonite, and the silicate minerals, such as diopside and pyroxene, are relatively higher than their quartz, dolomite and calcite counterparts (Chasse, Griffin, O'Reilly, & Calas, 2016; Devore, 1955; Schock, 1975). For this reason, pre-concentration studies were emphasized on the removal of impurity minerals, such as quartz, dolomite and calcite. Furthermore, the possibility of obtaining a pre-concentrate enriched in scandium content could be investigated by the removal of these gangue minerals.

5.1.3 Further Characterization Studies

5.1.3.1 Thermal Characterization

In order to determine the thermal behaviors of the Eskişehir-Mihalıççık ore sample, thermal characterization was done by making thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Thermal characterization was thought to be important due to the presence of carbonaceous minerals, which are decomposed at higher temperatures by releasing their carbon dioxide content. For this reason, thermal behavior of the representative trench sample was determined by using a Perkin Elmer Simultaneous Thermal Analyzer (STA 6000). Analysis were done by pyrolysis method. Analysis were carried out between 25 – 1000 °C and 20 ml/min gas flow at

an incremental rate of 10 °C/min. Results of the thermal analysis were given in Figure 5.3.

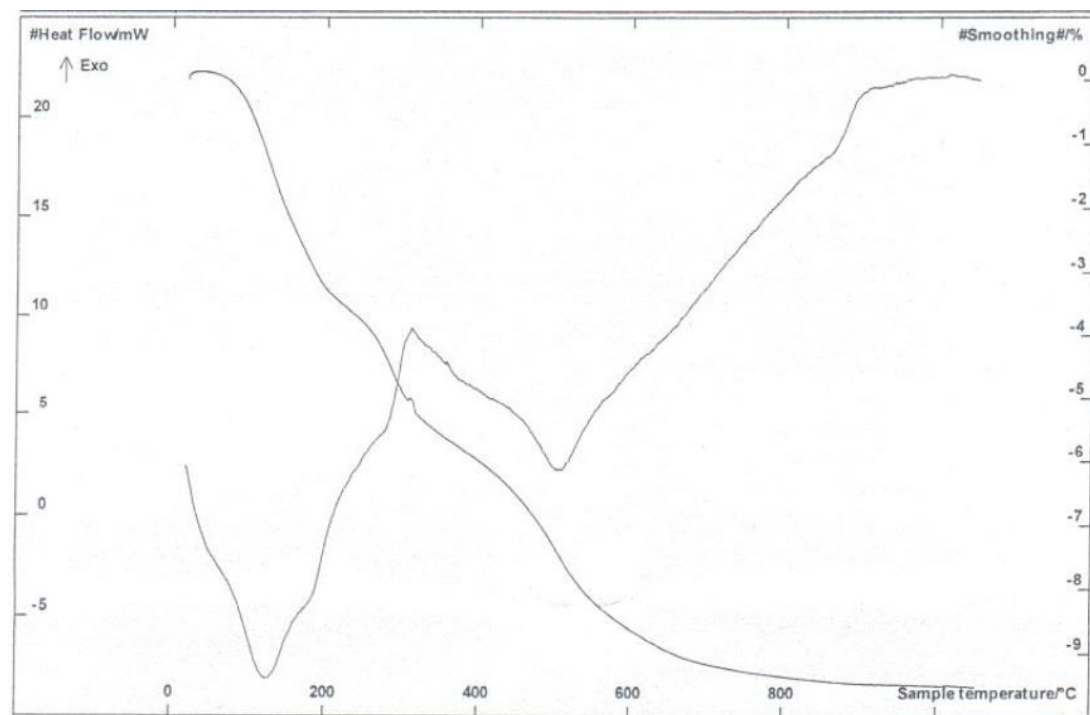


Figure 5.3 Thermal analysis results of representative trench sample obtained by pyrolysis method

Based on the thermal analysis results of the ore sample, the differential scanning calorimetry (DSC) graph shows that there were three main reaction areas as 150 to 200°C, 350 to 400°C and 550 to 600 °C. Thermal peak between 150 and 200 °C indicated that the reaction is endothermic and most probably related with the surface moisture of the ore sample. The exothermic peak between 350 and 400 °C was thought to be derived from the presence of hydroxide containing clay minerals. For the third reaction zone, namely between 550 to 600 °C, decomposition of dolomite, by releasing its carbon dioxide content, formed an endothermic peak. Although the decomposition temperature of magnesium carbonate is lower than 550 °C, due to the presence of calcium in the structure of dolomite, its decomposition temperature was directly increased. Lastly, it was considered that the relatively smaller exothermic peak at approximately 850 °C was formed because of the decomposition of serpentinite (magnesium containing silicate hydroxide) to forsterite, which is the magnesium

silicate counterpart of the serpentinite mineral. As the consequence of the thermal analysis, it was seen that the carbonate content of the ore sample was released at higher temperatures and the continuous weight losses of the ore sample at these temperatures were based on these aforementioned decomposition reactions.

5.1.3.2 Specific Gravity Determination

In the context of further characterization studies, specific gravity of the Eskişehir-Mihalıççık ore sample was determined by using helium pycnometer. A previously prepared representative trench sample was used for the experiment. The main purpose of specific gravity determination was to measure the possibility of trying mineral processing methods based on gravity separation during the pre-concentration studies. Experimental conditions of the specific gravity determination and the determined specific gravity value are given in Table 5.2.

Table 5.2 Specific gravity determination of the representative trench sample

Sample Code	Temperature (°C)	Weight (g)	Volume (cc)	Specific Gravity
Sarnıçtepe Head	25.1	5.38	2.01	2.67

As the result of the conducted specific gravity determination experiment, specific gravity of the ore sample was determined as 2.67. Considering the relatively low specific gravity of the ore sample, it was thought that such a low value was derived from the clay and carbonaceous minerals present in the ore sample. Therefore, the determined specific gravity value seems to be consistent with the mineralogical, XRD analyses of the ore sample. Based on all of these, the application of conventional gravity separation methods during the pre-concentration studies in order to obtain a pre-concentrate enriched in scandium bearing minerals, seems to be challenging due to both the low specific gravity of the ore and the closer specific gravity values of the ore minerals.

5.1.4 Sieve Analysis

As the last step of the characterization studies of the Eskişehir-Mihalıççık scandium ore, wet based sieve analyses, namely wet sieving analyses were performed. The main purposes of wet sieving were to evaluate the particle size distribution and the corresponding elemental distribution of the ore sample. Based on the conducted wet sieving, distribution of scandium, iron, aluminum and also the impurity content such as calcium, magnesium, and silicon with respect to the particle size distribution were evaluated. In addition to these, possible correlations between the aforementioned valuable and gangues were investigated in order to create the optimum pre-concentration study flowsheet and choosing the suitable operating parameters. This part of the study could be thought as the starting point of the following pre-concentration studies and also the pre-concentration studies were done based on the results obtained from the wet sieving analysis. In addition to these, XRD analyzes of the obtained size fractions were carried out in order to investigate the mineralogical structure of the sample in more detail. Previously prepared representative trench sample was used in wet sieving. Prior to wet sieving, the ore sample was steeped for the dispersion of the clay minerals covering the surfaces of the particles. Following this, the prepared slurry was sieved until minus 20 μm by using sieve sets. Based on the conducted wet sieving, obtained particle size distributions are given in Table 5.3 and Figure 5.4.

As it can be seen from the wet sieving results, the ore sample has a finer particle size distribution. This case is directly related with the clayey structure of the ore sample and also it confirms both the chemical and mineralogical analyses results. In this context, based on the wet sieving results, particle size of d_{80} and d_{50} were found to be 2033 μm and 119 μm , respectively. In addition to these, due to the fact that generally particles under 20 μm are called slimes and clay minerals in mineral processing operations, it was inferred that the clay minerals were dominating phases in the ore sample, because of approximately 35 % of the particles were under 20 μm .

Table 5.3 Particle size distribution of the representative trench sample

Fraction (mm)	Nominal Size (mm)	Weight (%)	Cum. Oversize (%)	Cum. Undersize (%)
+9.53	9.53	4.60	4.60	95.40
-9.53 + 6.35	6.35	4.53	9.13	90.87
-6.35 + 3.35	3.35	7.04	16.17	83.83
-3.35 + 2.14	2.14	3.17	19.34	80.66
-2.14 + 1.168	1.168	7.76	27.10	72.90
-1.168 + 0.85	0.85	3.28	30.38	69.62
-0.85 + 0.60	0.60	3.52	33.90	66.10
-0.60 + 0.417	0.417	1.89	35.79	64.21
-0.417 + 0.210	0.210	8.19	43.98	56.02
-0.210 + 0.106	0.106	7.26	51.24	48.76
-0.106 + 0.071	0.071	3.44	54.68	45.32
-0.071 + 0.053	0.053	4.17	58.86	41.14
-0.053 + 0.038	0.038	4.07	62.93	37.07
-0.038 + 0.020	0.020	2.51	65.44	34.56
-0.020	-	34.56	-	-
	<i>Total</i>	<i>100.00</i>		

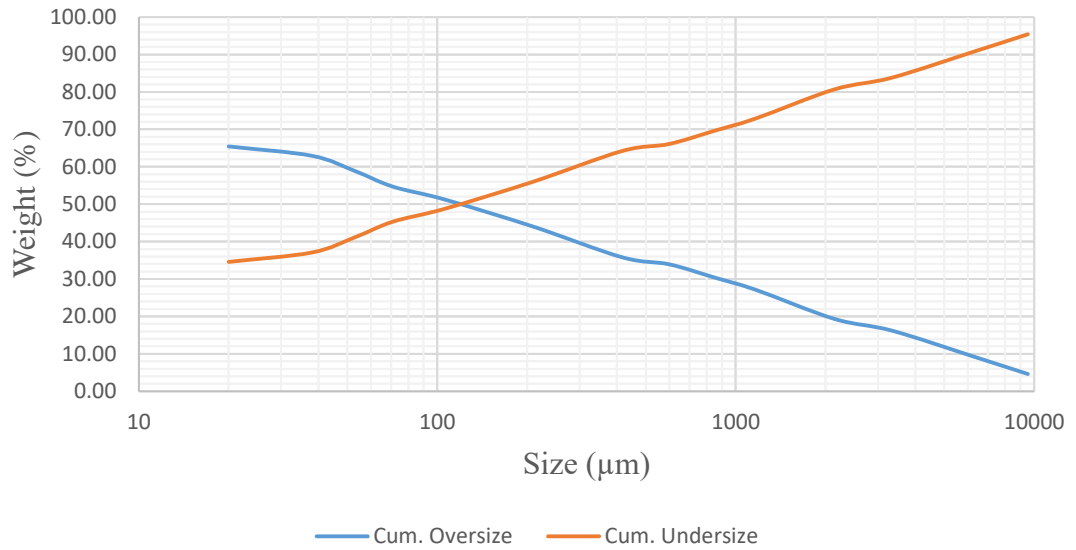


Figure 5.4 Cumulative particle size distributions of the representative trench sample

Following the examination of the particle size distribution of the ore sample, elemental grade values of the size fractions were investigated. Elemental analysis of the size fractions were determined by using ICP-OES method. Based on the elemental analysis results of the size fractions, metallurgical tables were constructed and recovery values were calculated. In this way, distributions of valuable elements and impurity elements of the ore sample with respect to the size fractions were observed and preliminary knowledge about the parameters of pre-concentration studies was obtained. In addition to these, cumulative recovery graphs for valuable elements and impurity elements were constructed in order to observe the possible relations between them. Also for the examination of the same situation, there was focused on the grade values and possible correlations were determined. Elemental distributions of the size fractions of the representative trench sample are given in Table 5.4.

Table 5.4 Elemental distributions of representative trench sample with respect to different particle sizes

Fraction (mm)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
+9.53	1.99	2.58	9.62	8.83	4.98	2.73	6.75	5.87	78	3.05	25.24	4.84
-9.53 + 6.35	2.06	2.62	10.59	9.56	4.76	2.56	7.24	6.20	87	3.37	25.87	4.87
-6.35 + 3.35	2.22	4.41	9.70	13.61	5.07	4.24	7.10	9.45	85	5.12	24.56	7.20
-3.35 + 2.14	2.13	1.90	9.10	5.76	4.96	1.87	6.89	4.13	81	2.21	25.88	3.42
-2.14 + 1.168	2.31	5.03	8.24	12.74	5.39	4.97	6.77	9.92	85	5.63	23.67	7.64
-1.168 + 0.85	2.14	1.97	7.49	4.90	5.32	2.08	6.35	3.94	84	2.36	26.80	3.66
-0.85 + 0.60	2.40	2.38	6.15	4.32	6.16	2.57	6.10	4.06	83	2.51	25.08	3.67
-0.60 + 0.417	2.16	1.15	5.91	2.23	5.69	1.28	5.60	2.00	83	1.18	26.85	2.11
-0.417 + 0.210	3.07	7.08	5.05	8.19	7.59	7.39	5.94	9.19	94	6.60	23.70	8.08
-0.210 + 0.106	3.21	6.56	4.76	6.88	8.09	6.98	5.70	7.82	98	6.09	23.06	6.97
-0.106 + 0.071	3.36	3.25	4.49	3.08	8.51	3.48	5.42	3.53	105	3.10	23.76	3.40
-0.071 + 0.053	3.44	4.04	3.80	3.17	8.77	4.35	5.13	4.05	111	3.95	23.71	4.12
-0.053 + 0.038	3.72	4.26	3.02	2.45	9.50	4.59	4.79	3.68	122	4.24	22.32	3.78
-0.038 + 0.020	3.80	2.69	2.91	1.46	9.59	2.86	4.61	2.18	125	2.67	23.13	2.41
-0.020	5.15	50.08	1.86	12.83	11.69	48.04	3.67	23.98	162	47.92	23.50	33.81
Total	3.55	100.00	5.02	100.00	8.41	100.00	5.29	100.00	117	100.00	24.02	100.00

Metallurgically calculated

As it can be seen from the elemental analysis results of the size fractions, impurities such as Ca, Mg, and Si enriched in relatively coarser size fractions, whereas elements such as Al, Fe, and Sc enriched in relatively finer size fractions. In terms of the impurity content of the ore, the highest Ca grade was observed at -9.53 + 6.35 mm fraction as 10.59 % and gradually decreased through finer size fractions and the lowest Ca grade was observed at - 0.020 mm fraction as 1.86 %. In this context, it was seen that recovery values of Ca was higher in the coarser fractions and approximately 60 % of Ca was concentrated in over 417 μm fractions. Apart from that, as well as Ca, highest grade of Mg was observed at -9.35 + 6.35 mm fraction as 7.24 % and gradually decreased through finer size fractions and the lowest Mg grade was observed at -0.020 mm fraction as 3.67 %. Also, recovery values of Mg indicated that it was mostly concentrated in coarser fractions and approximately 45 % of Mg was found in over 417 μm fractions. As another impurity, grades of Si did not show any big differences regarding particle size fractions. In conjunction with this situation, it was thought that the mono-dispersion of Si was based on the mineralogical structure of the ore sample and the silicon content of the ore was not only originated from the quartz mineral but also from clay and silicate minerals. The XRD analyses results of size fractions, which will be discussed in the following part of this section, provided given more detailed information about the presence of silicon, calcium and magnesium.

Based on the elemental analysis results of the size fractions, it was observed that elements such as Al, Fe, and Sc were enriched in finer fractions as mentioned before. In terms of scandium grade of the ore sample, the highest grade of Sc was observed at - 0.020 mm fraction as 162 ppm and the lowest Sc grade was observed at + 9.53 mm fraction as 78 ppm. In more details, under 417 μm , scandium grades were tended to increase from 83ppm to 162 ppm. As it was seen, Sc recovery increased drastically below 417 μm and approximately 48 % of scandium was concentrated below 20 μm . In this case, it was also determined that scandium must be recovered from the finer fractions of the ore. For the Fe content of the ore, the highest Fe grade was observed at - 20 μm fraction as 11.69 % and the lowest grade of Fe was observed at -9.53 + 6.35 mm fraction as 4.76 %. As well as Sc, Fe was already concentrated in finer

fractions and its grade tended to increase under 417 μm from 5.69 % to 11.69 %. Also, recovery values of Fe showed similarity with Sc recoveries that 48.04 % of the iron was concentrated below 20 μm , which also indicated the possibility of a correlation between scandium and iron. In the following parts of this section, a possible correlation of scandium and iron is investigated in more detail. Apart from these, in the case of Al distribution, the highest grade of Al was observed at - 0.020 mm as 5.15 % and the lowest grade of Al was observed at + 9.53 mm as 1.99 %. In terms of the recovery values of Al, it was observed that approximately 50 % of Al was concentrated below 20 μm , which seems to be similar with the distribution of Sc and Fe. In addition to these, based on the recovery values of valuable and impurity elements, constructed cumulative recovery graphs are given in Figure 5.5 and Figure 5.6.

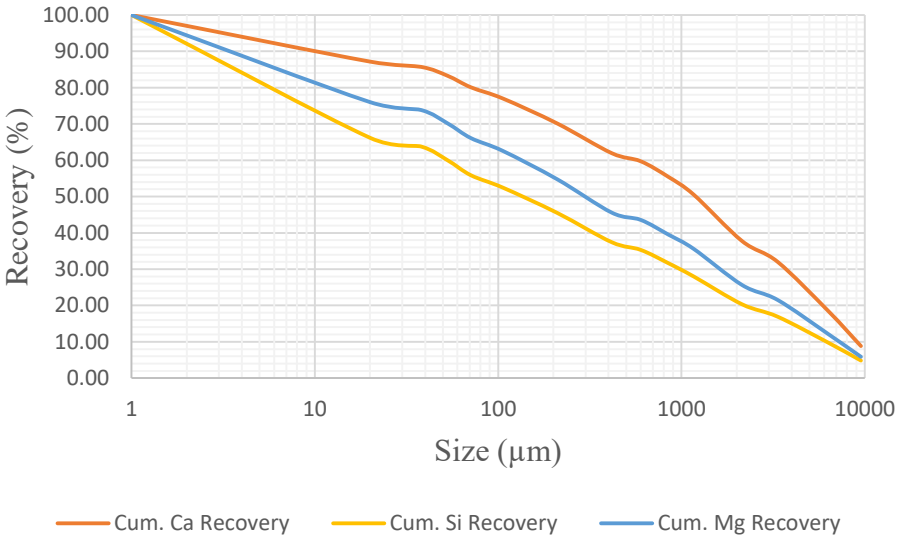


Figure 5.5 Cumulative recoveries of impurity elements with respect to particle sizes

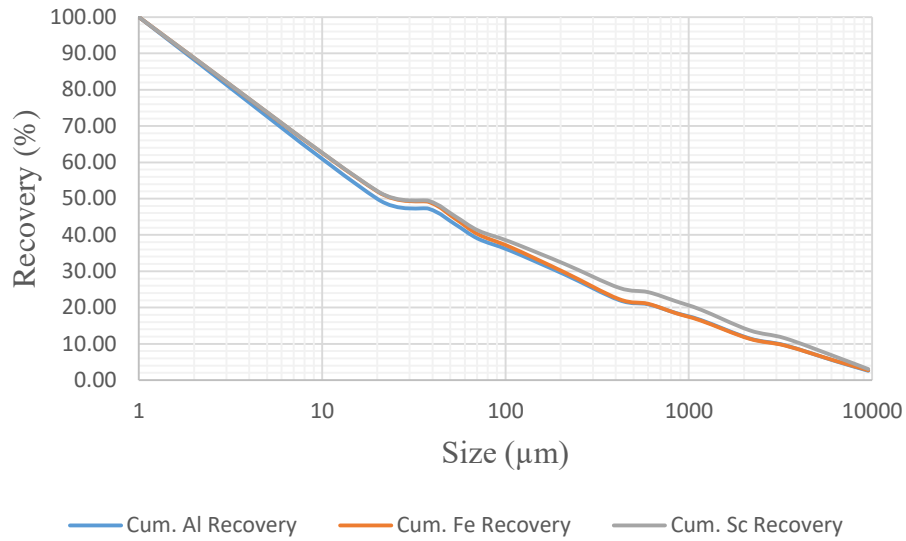


Figure 5.6 Cumulative recoveries of valuable elements with respect to particle sizes

According to the obtained grade values and cumulative recovery values of the elements from particle size distribution, once again it was indicated that impurity elements such as Ca, Mg, and Si, which were mostly concentrated in coarser fractions, behave completely different from their valuable counterparts namely Al, Fe, and Sc. In this context, a possibility of rough separation between valuable elements and impurity elements was observed by making size classification and also it was seen that the impurity elements of the ore could be removed in coarser fractions and thus enrichment of scandium in finer fractions could be provided. Impurities such as Ca and Mg, which make the subsequent hydrometallurgical processes challenging as consuming too much reagents, concentrated over 417 µm with the following recoveries as 64.94 % and 45.56 %, respectively. This case supported the pre-concentration studies in terms of the type of the method and the parameters of the method to be used. Although the mono-disperse structure of Si as another impurity element, 37.41 % of it was found over 417 µm, which was also seen as another advantage for the pre-concentration studies.

In addition to these, it was observed that valuable elements such as Al, Fe, and Sc behaves in similar manner with 77.96 %, 77.70 %, and 74.57 % recoveries, respectively, which can also be interpreted as a favorable condition for obtaining a pre-concentrate enriched in scandium as the result of the pre-concentration studies. At this stage, the relation between Sc and Fe was considered important and correlation plot of Sc and Fe grades regarding different size fractions was constructed in order to examine the relation in more details. The correlation plot of Sc and Fe grades is given in Figure 5.7.

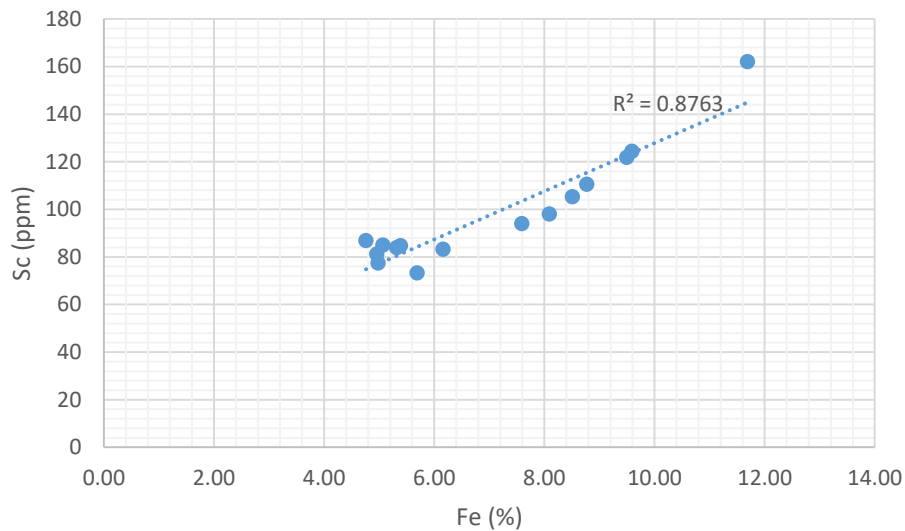


Figure 5.7 Correlation plot of Sc and Fe grades

Based on the constructed correlation plot of Sc and Fe grades, it was evident that there is a strong correlation between scandium and iron. According to the previous studies on correlation between Sc and Fe, this type of correlation was generally observed in clay minerals (Birgöl, 1981). Also, this finding seems to be consistent with the previous chemical and mineralogical analyses. This situation proved the clayey structure of the Ekişehir-Mihalıççık ore sample once again. As the result of the correlation plot, correlation coefficient of Sc and Fe was calculated as 0.8763, which indicated that there is a strong correlation between these two elements.

In addition to these, XRD analyses of the obtained size fractions were performed in order to investigate the mineralogical structure of the ore sample in more details. X-ray diffractograms of the size fractions are given in Appendix C (Figure C.1-C.15). According to the XRD analyses, the major components and their characteristic peak positions are as follows:

- **Dolomite:** 30.97°, 45.01°, 50.45°, 63.01°
- **Quartz:** 20.85°, 26.63°, 36.54°, 42.48°, 59.98°, 68.13°, 76.50°
- **Nontronite:** 19.94°, 20.85°, 25.55°, 26.63°, 39.48°, 50.17°, 61.29°, 68.13°, 71.51°
- **Montmorillonite:** 12.46°, 19.94°
- **Diopside:** 29.50°, 30.35°, 34.76°, 35.70°, 39.19°, 41.95°, 44.43°, 52.30°, 57.01°, 92.20°
- **Augite:** 27.96°, 29.50°, 39.19°, 41.95°, 57.01°, 59.16°
- **Pyroxene:** 27.96°, 66.15°
- **Calcite:** 65.90°
- **Sepiolite:** 26.63°

As it can be seen from the mineralogical analysis of the size fractions of the ore sample, presence of impurities such as Ca and Mg in the coarser fractions was based on the occurrence of calcite and dolomite minerals in these fractions. It was also observed that the intensity of the characteristic peaks of calcite and dolomite minerals tends to be decrease in finer size fractions. In this context, this situation seems to be consistent with the previous chemical analysis of the size fractions. Apart from these, for the presence of Si in the ore sample, two different occurrence mechanism were observed. The first occurrence state of Si was directly related with the occurrence of quartz and silicate minerals such as diopside and augite that were mostly concentrated in the coarser fractions. On the other hand, it was observed that the occurrence of Si in finer fractions was directly related with the clay minerals such as nontronite and montmorillonite, which were generally responsible for the most intense characteristic

peaks at finer size fractions. Also, it was thought that the monodispersed state of Si was based on these mentioned cases.

As the result of the mineralogical analysis, it was thought that the relative enrichment of elements such as Al, Fe and Sc in finer fractions was directly related with the presence of iron and aluminum rich clay minerals such as nontronite and montmorillonite in finer fractions. In addition, due to montmorillonite and nontronite minerals being members of smectite group clay minerals, conducted XRD analyses were seemed to be consistent with previously made XRD analysis. More than that, it was thought that the presence of scandium in the intermediate fractions was related with the presence of silicates such as diopside, pyroxene, and augite, but also there was strong evidence for a relation of scandium and clay minerals, namely enrichment of scandium under 20 μm as 162 ppm proved that hypothesis. In this context, mineralogically proven dominance of nontronite and montmorillonite under 20 μm and the enrichment of scandium in that fraction also indicated that scandium was enriched in clay minerals.

Considering the results of the characterization studies, major impurity minerals, containing Mg, Ca, and Si, to be removed during the pre-concentration studies were determined as calcite, dolomite and quartz. Besides that, due to the enrichment of scandium mostly in clay minerals, such as montmorillonite and nontronite, the recovery of the clay minerals during the pre-concentration studies in order to obtain a pre-concentrate enriched in scandium was considered. All the methods and operational parameters selected during the pre-concentration studies were based on the above mentioned main idea.

5.2 Pre-Concentration Studies

5.2.1 Attrition Scrubbing of the Scandium Ore

For the removal of major impurity minerals containing Ca, Mg and Si, namely calcite, dolomite, and quartz and to obtain a pre-concentrate enriched in scandium content, attrition scrubbing experiments were carried out according to the methodology given previously. Based on the examination of the particle size distribution and clayey structure of the ore obtained from the previously made wet sieving analysis, it was seen that the impurity minerals of the ore were concentrated in coarser fractions and also valuable element bearing clay minerals were concentrated in finer fractions. In this context, the necessity for the recovery of scandium concentrated in coarser fractions appeared to be crucial. It was also important to remove as much as possible gangue minerals during the recovery of scandium concentrated in coarser fractions, thereby it was observed that the concentrated scandium in coarser fractions was based on the clay minerals, which were covering the surfaces of coarser particles and agglomerated as the result of this. In this case, clay minerals present in the coarser fractions of the ore needed to be dispersed and liberated in order to recover the valuable content of the coarser fractions without fracturing or breaking the material. This is why attrition scrubbing was chosen as the initial pre-concentration method to be applied (Strazisar and Seselj, 1999; Petavy and V. Ruban, 2009). In addition to these, a size classification was required in order to separate the pre-concentrate and gangue fractions following the attrition scrubbing experiments. According to the wet sieving analysis results, it was found that scandium was mostly concentrated under 417 μm , on the contrary Ca, Mg, and Si containing impurity minerals were relatively concentrated above this size fraction. Thus, the size classification following the attrition scrubbing was decided to be made at 417 μm . The general flowsheet applied for the attrition scrubbing experiments is given in Figure 5.8. Prior to the second step of pre-concentration studies, namely enhanced gravity separation by using falcon concentrator, the extent of attrition scrubbing was first evaluated in terms of scandium

recovery in pre-concentrate and silica removal, magnesium removal, and calcium removal in tailing products.

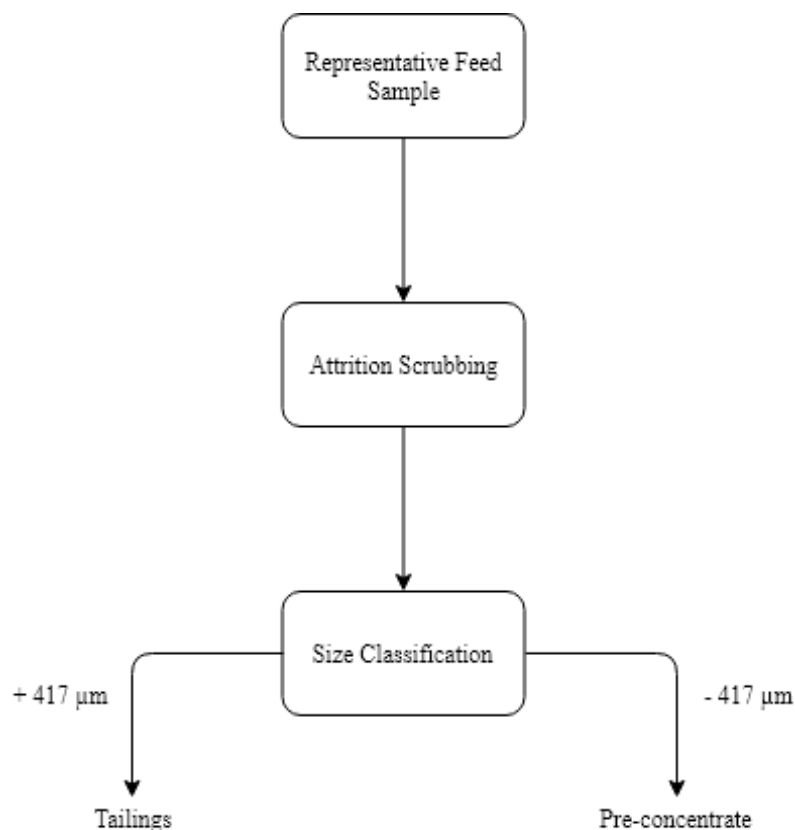


Figure 5.8 General flowsheet for the attrition scrubbing experiments

5.2.1.1 Preliminary Attrition Scrubbing Studies

As the initial step of the attrition scrubbing studies, preliminary tests of attrition scrubbing were carried out for the selection of the optimum parameter ranges. In this context, the effects of previously selected operational parameters on the scandium recovery, silicon removal, magnesium removal and calcium removal were investigated. Following the preliminary tests of attrition scrubbing, the optimum parameter range was determined and then these parameters were optimized in terms of grade and recovery of the products and tailings. For the preliminary attrition scrubbing tests, seven different experiments were conducted by changing the

operational parameters namely solid-liquid ratio and residence time. At this stage, rotational speed was kept fixed because of the narrow range of this parameter due to the limitations of the attrition scrubber. Experimental conditions of the preliminary attrition scrubbing tests are given in Table 5.5.

Table 5.5 Experimental conditions of preliminary attrition scrubbing tests

Exp. Code	Solid (%)	Rotation Speed (RPM)	Residence Time (Min)
YM-1	55.00	800	30
YM-2	40.00	800	30
YM-3	25.00	800	30
YM-4	25.00	800	90
YM-5	25.00	800	120
YM-6	25.00	800	150
YM-7	25.00	800	180

As it was examined from the previous attrition scrubbing studies on different types of ores, the solid-liquid ratio of the feed slurry was kept around 70 %, thus it was desired to start with a solid-liquid ratio of 70 % (Boundy *et al.*, 2017). Although it could have been applied for the ores with low amount of clay minerals, it was observed that 70 % solid-liquid ratio was not applicable for the Eskişehir-Mihalıççık scandium ore that contained too much clay minerals due to the formation of highly viscous slurry cake based on the pillowing effect of clay minerals at such a high solid content. In this case, previous attrition scrubbing studies on clayey ores were investigated and it was seen that the solid-liquid ratio of the slurry should be adjusted to around 50 % or below (Özbayoğlu and Atalay, 2000; Hassan and Abdel-Khalek, 1998). Also, it was considered that this situation was based on the clay minerals contained in the ore, which provided the sufficient viscosity of the slurry for the intensive inter-collisions between particles during attrition scrubbing. For these reasons, upper and lower limits of the solid-liquid ratio were tried as 55 % and 25 %, respectively.

Based on the previous studies on the residence time of attrition scrubbing, it was observed that relatively shorter times were applicable for ores without containing clay minerals (Birinci and Sarıkaya, 2004). In the presence of clay minerals, due to the sticky and surface covering characteristics of them, it was thought that relatively longer residence times could be beneficial in terms of recovery of clay minerals. In this context, residence times of 30, 60, 90, 120, 150, and 180 minutes, were tried in order to determine the upper and the lower limits of residence time for the optimization studies. Metallurgical results of the preliminary attrition scrubbing experiments are given in Table 5.6.

As the consequence of the metallurgical test results of the preliminary attrition scrubbing experiments, it was observed that both solid-liquid ratio and residence time directly affected the recovery and grade values of the pre-concentrate and tailings products. In this context, it was observed that increase in the solid-liquid ratio decreased the weight yield of the oversize products namely tailings, which means increased the inter-particle collisions and generated an intensive abrasion on the surface of particles. In addition to these, although higher solid-liquid ratios increased the scandium recovery, they also decreased removal rate of impurities such as Ca, Mg, and Si. As it can be seen from the metallurgical results of the experiment carried out at 55 % solid content, Ca, Mg, and Si removal rates were 30.15 %, 20.31 % and 22.23 %, respectively. This situation was considered to be based on the over-attrition of the ore, which was a result of the comminution of relatively coarse particles in highly viscous feed slurry and thereby impurity removal rates were slightly lowered in accordance. On the other hand, their removal rates tended to increase by the decreased solid-liquid ratio as 55.21 % Ca, 39.74 % Mg, and 32.59 % Si recovery in the tailings fraction when the solid-liquid ratio was adjusted to 40.00 %. In similar conditions by only changing the solid-liquid ratio to 25 %, no considerable difference in terms of impurity removal was observed but the scandium recovery relatively increased. Therefore, it was decided that the solid-liquid ratio between 25 and 55 % was applicable for the optimization of the attrition scrubbing experiments.

From a similar point of view, it was observed that the increase of residence time also increased the weight yield of the undersize material due to the exposure time of the sample to attrition scrubbing. In this context, four similar experiments with different residence times were carried out and it was seen that the weight yield of the undersize product namely pre-concentrate was increased from 68.70 % to 80.00%, while the residence time was increased from 30 to 180 minutes. Considering the effect of the change in residence time on the removal rate of impurities, it was observed that by the increase in of the residence time, removal rate was also decreased with related to the increased number of collisions between particles. On the contrary, again due to the increased number of collisions, scandium recovery was gradually increased by the increase of residence time. Within the scope of the preliminary attrition scrubbing experiments, different residence times up to 180 minutes were tried and it was observed that the residence time had no crucial effect on the scandium recovery after a certain optimum point. In addition to these, it was also seen that the removal rates of impurities such as Ca and Mg were relatively decreased after the optimum upper limit of residence time was reached. The upper limit for the residence time was selected as 90 minutes, which was thought as the boundary for the critical point of recovery of scandium and removal of impurities. The lower limit for the residence time was selected as 30 minutes, which was considered as the minimum time needed to disperse and liberate the clay minerals contained in the ore. Following the preliminary attrition scrubbing experiments, the optimization of the attrition scrubbing experiments was performed based on the parameter ranges that were indicated by the preliminary experiments, and as a result, the operational parameters that provided the optimum conditions in terms of both recovery and grade of the pre-concentrate and tailings products were determined.

Table 5.6 Metallurgical results of the preliminary attrition scrubbing experiments

Exp. Code	Fraction (µm)	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
YM-1	+417 µm	19.62	1.60	8.83	5.20	30.15	4.01	10.26	5.53	20.31	57	10.83	26.21	22.23
	-417 µm	80.38	4.03	91.17	2.94	69.85	8.56	89.74	5.30	79.69	115	89.17	22.38	77.77
	Total	100.00	3.56	100.00	3.38	100.00	7.66	100.00	5.35	100.00	104	100.00	23.13	100.00
YM-2	+417 µm	31.30	2.12	17.49	5.92	55.21	4.77	18.57	6.80	39.74	72	20.68	25.86	32.59
	-417 µm	68.70	4.55	82.51	2.19	44.79	9.54	81.43	4.70	60.26	126	79.32	24.37	67.41
	Total	100.00	3.79	100.00	3.36	100.00	8.05	100.00	5.36	100.00	109	100.00	24.84	100.00
YM-3	+417 µm	31.30	1.75	15.52	4.94	50.25	4.12	17.12	5.55	35.01	61	18.87	25.69	32.42
	-417 µm	68.70	4.34	84.48	2.23	49.75	9.09	82.88	4.70	64.99	119	81.13	24.40	67.58
	Total	100.00	3.53	100.00	3.08	100.00	7.53	100.00	4.96	100.00	101	100.00	24.80	100.00
YM-4	+417 µm	24.03	1.95	12.43	6.08	41.12	4.55	13.70	6.61	28.76	69	15.28	25.87	25.04
	-417 µm	75.97	4.35	87.57	2.75	58.88	9.07	86.30	5.18	71.24	121	84.72	24.50	74.96
	Total	100.00	3.77	100.00	3.55	100.00	7.98	100.00	5.52	100.00	109	100.00	24.83	100.00
YM-5	+417 µm	22.35	1.78	10.69	6.13	38.74	4.20	11.95	6.64	27.18	85	15.54	32.97	26.79
	-417 µm	77.65	4.28	89.31	2.79	61.26	8.91	88.05	5.12	72.82	133	84.46	25.93	73.21
	Total	100.00	3.72	100.00	3.54	100.00	7.86	100.00	5.46	100.00	122	100.00	27.50	100.00
YM-6	+417 µm	21.43	2.02	11.36	6.30	38.20	4.66	12.23	6.65	26.19	78	13.61	30.26	24.39
	-417 µm	78.57	4.30	88.64	2.78	61.80	9.12	87.77	5.11	73.81	135	86.39	25.58	75.61
	Total	100.00	3.81	100.00	3.53	100.00	8.16	100.00	5.44	100.00	123	100.00	26.58	100.00
YM-7	+417 µm	20.00	1.73	9.38	6.35	36.43	4.35	11.08	6.67	24.68	65	11.11	32.29	24.51
	-417 µm	80.00	4.18	90.62	2.77	63.57	8.73	88.92	5.09	75.32	130	88.89	24.86	75.49
	Total	100.00	3.69	100.00	3.49	100.00	7.85	100.00	5.41	100.00	117	100.00	26.35	100.00

Metallurgically calculated

Prior to the optimization of the attrition scrubbing experiments, another important operational parameter which is the feed particle size of the ore was examined. Based on the previously conducted wet sieving analysis, it was determined that approximately 90 % of the representative feed sample was under 6.35 mm. which was thought to be a suitable feed particle top size for the attrition scrubbing process (Hassan and Abdel-Khalek, 1998; Özbayoğlu and Atalay, 2000; Birinci and Sarıkaya, 2004). Therefore based on both the preliminary experimental results and wet sieving results, it was considered that there was no need for further particle size reduction. For these reasons, feed particle size was not studied during the optimization studies.

5.2.1.2 Optimization of the Attrition Scrubbing of Eskişehir-Mihalıççık Scandium Ore by Statistical Modeling

Optimization of the operational parameters of the attrition scrubbing of Eskişehir-Mihalıççık scandium ore was performed in order to obtain the maximum scandium recovery besides maximum removal of impurities such as Ca, Mg, and Si. The main purpose of the experiments is to obtain a pre-concentrate that was enriched in scandium content by rejecting impurities. In this context, experimental parameters were optimized based on the two level full factorial statistical design and types of the parameters and their ranges to be used in the experimental design were determined according to the results obtained from the preliminary attrition scrubbing experiments.

As aforementioned above, based on the results of the preliminary attrition scrubbing experiments, solid-liquid ratio, retention time and rotation speed were chosen as the three most important operational parameters that directly affected the results of the experiments. Within the scope of the experiments, these parameters were varied within a certain range and their effects on the results of the experiments were evaluated. Thereby, as the result of the statistical design, interactions between the parameters were also examined in details. For the optimization of the attrition scrubbing experiments, parameter codes, levels of parameters and assigned values to be used in the statistical design are given in Table 5.7. In addition to the optimization

of the attrition scrubbing experiments by statistical modeling for investigating the effects of each operational parameter on the recovery of scandium and removal of impurities, attrition scrubbing experiments were conducted while one parameter was varied and the other two parameters were fixed at the base levels.

Table 5.7 Parameter codes and assigned values for the attrition scrubbing experiments

Parameter / Code (%)	Lower Level	Base Level	Higher Level
Solid Ratio / A (%)	25 / -1	40 / 0	55 / 1
Retention Time / B (Min)	30 / -1	60 / 0	90 / 1
Rotation Speed / C (Rpm)	800 / -1	1000 / 0	1200 / 1

Based on the conducted attrition scrubbing experiments, experimental conditions and their metallurgical results for identifying the effect of solid-liquid ratio are given in Table 5.8. Retention time and rotation speed were kept same at all experiments as 1000 rpm and 60 min, respectively. As it can be seen from the metallurgical results, weight yield of the tailings namely the oversize material was decreased by increasing the solid-liquid ratio from 25 to 55 %. In more details, 14.64 % of the attrition scrubbed material was subjected to tailings at 55 % solid-liquid ratio, while 26.42 % and 31.58 % of the attrition scrubbed material were subjected to the tailings at solid-liquid ratios of 40 % and 25 %, respectively. It was thought that the decrease of the weight yield of tailings was directly related with the higher intensity of collisions between particles due to the increase of the solid-liquid ratio, namely increase of the viscosity of the slurry. Also, the change in the solid-liquid ratio directly affected the recovery of scandium and removal of impurities such as Ca, Mg, and Si. In this context, scandium recovery was 91.65 % for the experiment carried out at 55 % solid-liquid ratio. On the other hand, removal rates of Ca, Mg, and Si were 23.76 %, 16.03 %, and 17.99 %, respectively. For the experiment conducted at 40 % solid-liquid ratio, scandium recovery was decreased to 83.69 %, but also the removal rates of Ca, Mg, and Si were increased to 48.92 %, 32.39 %, and 26.19 %, respectively. Moreover, scandium recovery for the experiment of 25 % solid-liquid ratio was determined as 78.97 %, which showed that scandium recovery was tended to be decreased by decreasing solid-

liquid ratio. In addition to these, the removal rates of Ca, Mg, and Si were increased to 55.18 %, 38.29 %, and 30.58 %, respectively. Consequently, it was inferred that the solid-liquid ratio had a positive effect on the scandium recovery by increasing the intensity of collisions between particles, but decreased the removal rates of impurities related with the same reason. In this case, it was observed that the solid-liquid ratio was one of the important parameter that influenced the recovery and removal rates. In other words, scandium recovery was increased with increased solid-liquid ratio and removal rates of impurities were decreased at a relatively higher amount. In this case, it was considered that it might be feasible to reject relatively higher amount of impurities with a minimum loss of scandium at lower solid-liquid ratios.

Table 5.8 The Effect of solid-liquid ratio on attrition scrubbing of Eskişehir-Mihalıççık Scandium ore

Solid (%)	Rotation Speed (RPM)	Retention Time (min)	Fraction (µm)	Weight (%)	Ca Rec. (%)	Mg Rec. (%)	Sc Rec. (%)	Si Rec. (%)
55.00	1000	60.00	+417 µm	14.64	23.76	16.03	8.35	17.99
			-417 µm	85.36	76.24	83.97	91.65	82.01
			Total	100.00	100.00	100.00	100.00	100.00
40.00	1000	60.00	+417 µm	26.42	48.92	32.39	16.31	26.19
			-417 µm	73.58	51.08	67.61	83.69	73.81
			Total	100.00	100.00	100.00	100.00	100.00
25.00	1000	60.00	+417 µm	30.06	55.18	38.29	21.03	30.58
			-417 µm	69.94	44.82	61.71	78.97	69.42
			Total	100.00	100.00	100.00	100.00	100.00

For determining the effect of rotation speed on the scandium recovery and removal rates of impurities, experiments and their metallurgical results were investigated as summarized in Table 5.9. Solid-liquid ratio and retention time were kept same at all experiments as 40 % and 60 min., respectively. According to the metallurgical results of the experiments, weight yield of the tailings namely the oversize material was decreased by increasing the rotation speed from 800 rpm to 1200 rpm. In detail, 24.36 % of the attrition scrubbed material was subjected to tailings at 1200 rpm rotation speed, while 26.42 % and 27.03 % of the attrition scrubbed material were subjected to the tailings at rotation speeds of 1000 rpm and 800 rpm, respectively. Based on the

obtained results, it was thought that the decrease of the weight yield of the tailings with the increase of the rotation speed was directly related with the increased frequency of collisions between particles through the increase in rotation speed of the scrubber. In addition to these, it was observed that rotation speed did not have a crucial effect on scandium recovery, as the recoveries of scandium fluctuated between 81.08 % and 83.69 %. On the contrary, considerable effect of rotation speed on the removal of impurities such as Ca, Mg, and Si was observed. In this context, removal rates of Ca, Mg, Si were 41.08 %, 29.68 %, and 24.14 %, respectively for the experiment carried out at rotation speed of 1200 rpm. It was also observed that the removal rate of impurities were directly related with the frequency of collisions as the frequency of collisions increased when the removal rate of the impurities decreased. In conjunction with this, Ca, Mg, and Si removal rates were increased to 48.92 %, 32.39 % and 26.19 %, respectively for the experiment conducted at a rotation speed of 1000 rpm. Accordingly, removal rates of the experiment conducted at 800 rpm were 47.46 % for Ca, 32.99 % for Mg, and 29.12 % for Si, which also showed that the maximum removal of impurities was achieved at 800 rpm with a fair amount of scandium loss. Considering the above mentioned issues, it was inferred that rotation speed was not as important parameter as the solid-liquid ratio, but also it affected the removal rates to a certain extent. At that stage, it was considered that in order to determine the optimum value for the rotation speed, the results of the constructed statistical model about the interactions between the rotation speed, solid-liquid ratio and retention time, might be beneficial.

Table 5.9 The Effect of rotation speed on attrition scrubbing of Eskişehir-Mihalıççık Scandium ore

Solid (%)	Rotation Speed (RPM)	Retention Time (min)	Fraction (µm)	Weight (%)	Ca Rec. (%)	Mg Rec. (%)	Sc Rec. (%)	Si Rec. (%)
40.00	1200	60.00	+417 µm	24.36	41.08	29.68	17.15	24.14
			-417 µm	75.64	58.92	70.32	82.85	75.86
			Total	100.00	100.00	100.00	100.00	100.00
40.00	1000	60.00	+417 µm	26.42	48.92	32.39	16.31	26.19
			-417 µm	73.58	51.08	67.61	83.69	73.81
			Total	100.00	100.00	100.00	100.00	100.00
40.00	800	60.00	+417 µm	27.03	47.46	32.99	18.92	29.12
			-417 µm	72.97	52.54	67.01	81.08	70.88
			Total	100.00	100.00	100.00	100.00	100.00

In the context of determining the effect of the retention time on scandium recovery and removal rates of impurities, conducted experiments and their metallurgical results are given in Table 5.10. Solid-liquid ratio and rotation speed were kept constant at all experiments as 40 % and 1000 rpm, respectively. In the light of the obtained results, weight yield of tailings namely the oversize material was decreased by increasing the retention time from 30 min. to 90 min. Correspondingly, 24.04 % of the attrition scrubbed material was subjected to tailings at retention time 90 min., while 26.42 % and 30.91 % of the attrition scrubbed material were subjected to the tailings at retention times of 60 min. and 30 min., respectively. Considering the obtained results, it was observed that the decrease of the weight yields of the tailings with the increase of the retention time was directly related with the increased number of collisions of particles through increased retention time. The results of the experiments indicated that the scandium recovery was increased by increasing the retention time. Based on this, scandium recovery was determined as 85.52 % at retention time of 90 min. while 83.69 % and 75.78 % for retention times of 60 min. and 30 min., respectively. As it was previously observed for solid-liquid ratio and rotation speed, increase of retention time had also a negative effect on the removal of impurities. Removal rates of Ca, Mg, and Si were 39.56 %, 26.79 %, and 23.66 % for the experiment carried out at a retention time of 90 minutes. For the experiment conducted at retention time of 60 min., removal rates were 48.92 % for Ca, 32.39 % for Mg, and 26.19 % for Si. Also, removal rates

of impurities tended to increase with the decrease of retention time to 30 min., as 54.87 % of Ca, 39.88 % of Mg, and 33.20 % of Si were removed. On the other hand, it was observed that the removal rate of impurities was directly related with the number of collisions while the number of collisions increased, the removal rate of the impurities decreased. In connection with these results, it was inferred that retention time was the second important parameter following the solid-liquid ratio and it affected both the scandium recovery and removal of impurities at a certain amount. Optimization of these operational parameters was completed by the constructed statistical model which was given in forthcoming part.

Table 5.10 The Effect of retention time on attrition scrubbing of Eskişehir-Mihalıççık Scandium ore

Solid (%)	Rotation Speed (RPM)	Retention Time (min)	Fraction (μm)	Weight (%)	Ca Rec. (%)	Mg Rec. (%)	Sc Rec. (%)	Si Rec. (%)
40.00	1000	90.00	+417 μm	24.04	39.56	26.79	14.48	23.66
			-417 μm	75.96	60.44	73.21	85.52	76.34
			Total	100.00	100.00	100.00	100.00	100.00
40.00	1000	60.00	+417 μm	26.42	48.92	32.39	16.31	26.19
			-417 μm	73.58	51.08	67.61	83.69	73.81
			Total	100.00	100.00	100.00	100.00	100.00
40.00	1000	30.00	+417 μm	30.91	54.87	39.88	24.22	33.20
			-417 μm	69.09	45.13	60.12	75.78	66.80
			Total	100.00	100.00	100.00	100.00	100.00

For the optimization of the attrition scrubbing experiments, within the context of selected parameters and their ranges, design of experiments were made by using Stat Ease 9.0 software. In terms of the accuracy of the experimental results and the statistical model, each experiment was carried out in parallel. In addition to these, for the reliability of the constructed model, run orders of the experiments were randomized and three parallel experiments were conducted at the base level of the experimental design. In order to optimize the operational parameters of attrition scrubbing experiments, constructed design of experiments is given in Table 5-9. According to the design of experiments, the main purpose of the constructed model was to obtain a pre-concentrate enriched in scandium with maximum scandium recovery and

maximum impurity removal by keeping the operational parameters namely solid-liquid ratio, retention time, and rotation speed in the previously determined ranges. The detailed metallurgical results of the attrition scrubbing experiments in order to optimize the process are given in Appendix D (Table D.1-D.3).

Table 5.11 Design of attrition scrubbing experiments

Standard Order (Exp. Code)	Run Order	Solid Ratio / A	Retention Time / B	Rotation Speed / C
1	3	-1	-1	-1
2	7	-1	-1	-1
3	8	1	-1	-1
4	16	1	-1	-1
5	14	-1	1	-1
6	5	-1	1	-1
7	12	1	1	-1
8	1	1	1	-1
9	2	-1	-1	1
10	19	-1	-1	1
11	11	1	-1	1
12	10	1	-1	1
13	15	-1	1	1
14	17	-1	1	1
15	18	1	1	1
16	4	1	1	1
17	6	0	0	0
18	9	0	0	0
19	13	0	0	0

Within the scope of the optimization of the attrition scrubbing experiments, scandium recovery, calcium removal, magnesium removal, and silicon removal were taken as responses and the optimization study was conducted based on the results of these response values. Based on the design of experiments, average minimum and maximum values of responses obtained and the goals of the optimization study are given in Table 5-12. According to the results, scandium recovery changed between 78.72 % and 89.17 %. Also, removal rate of calcium fluctuated between 30.15 % and 58.87 %. During the experiments, silicon and magnesium behaved similarly as the removal rate for them were approximately differentiated from 18 % to 40 %. As it can be seen from the maximum and minimum response values, relatively higher rate of recovery of scandium was possible by removing the impurities at the maximum possible extent.

Table 5.12 Lower and upper response limits obtained from the attrition scrubbing experiments and goals of the statistical model

	Goal	Lower Limit	Upper Limit
A:Solid Ratio (%)	is in range	25	55
B:Retention Time (Min)	is in range	30	90
C:Rotation Speed (Rpm)	is in range	800	1200
Scandium Recovery (%)	maximize	78.72	89.17
Silica Rejection (%)	maximize	18.17	36.79
Magnesium Rejection (%)	maximize	20.31	39.98
Calcium Rejection (%)	maximize	30.15	58.87

Based on the metallurgical results obtained from the experiments, cubic representations of the statistical model were constructed by using the average response values. Cubic representations of the model are given in Figure 5.9 to Figure 5.12. In this context, the maximum scandium recovery was observed at 55 % solid-liquid ratio, 800 rpm rotation speed and 30 minutes of retention time, as 87.88 %. Maximum removal rates of impurities were generally observed at lower solid-liquid ratios, rotation speeds, and retention times. For the removal of calcium, maximum response

value was observed at 25 % solid-liquid ratio, 1200 rpm rotation speed, and 30 minutes of retention time, as 57.09 %. The peak value of magnesium removal was observed as 39.81 % at 25 % solid-liquid ratio, 1200 rpm rotation speed, and 30 minutes of retention time. Finally, maximum silica rejection was equal to 35.32 % at 25 % solid-liquid ratio, 800 rpm rotation speed, and 30 minutes of retention time.

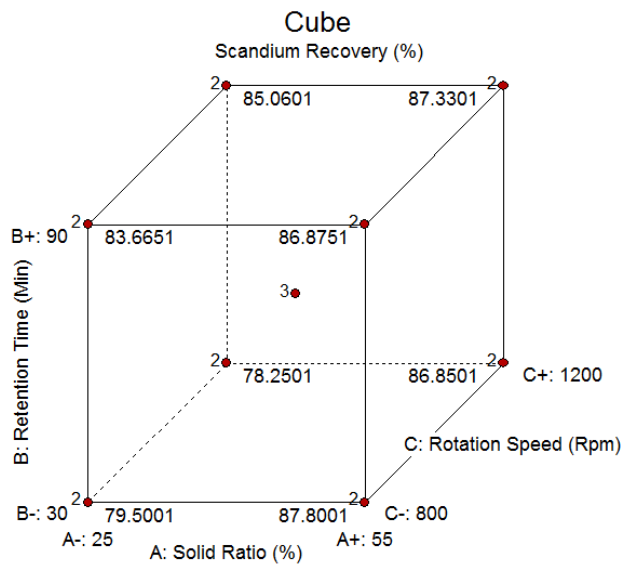


Figure 5.9 Cubic representation of the scandium recovery

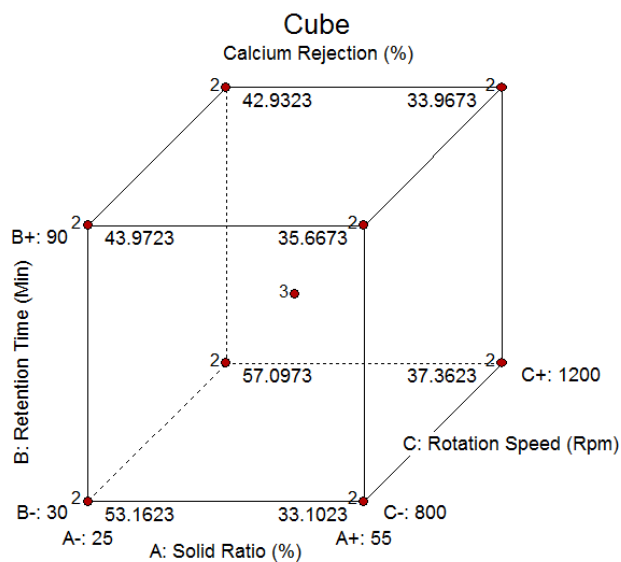


Figure 5.10 Cubic representation of the calcium removal

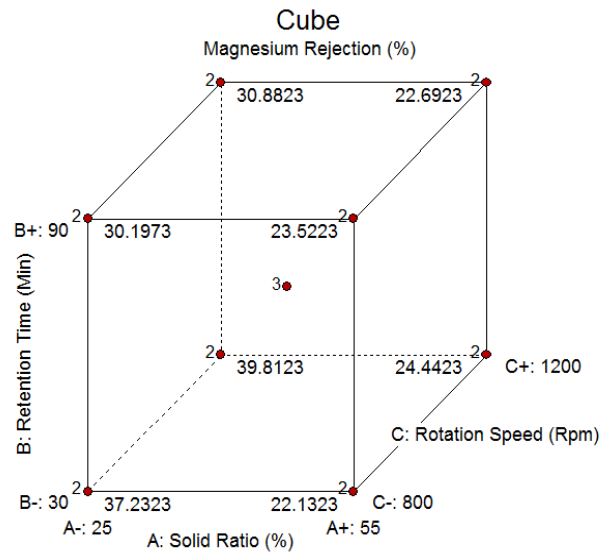


Figure 5.11 Cubic representation of the magnesium removal

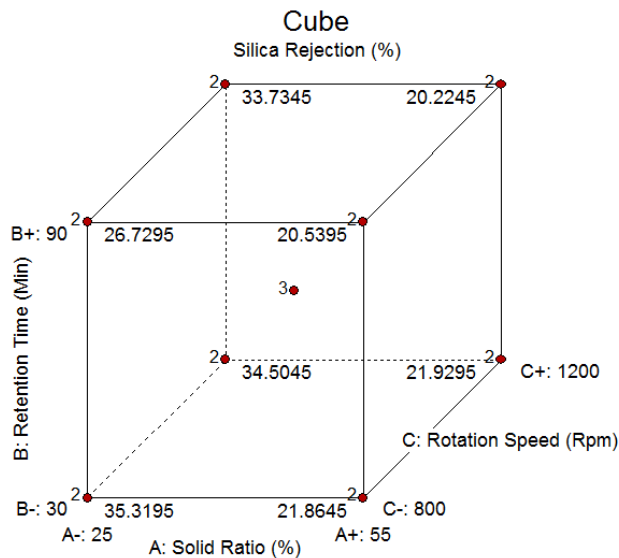


Figure 5.12 Cubic representation of the silicon removal

By evaluating the results according to the aforementioned four different responses, namely scandium recovery, calcium removal, magnesium removal, and silicon removal, direct effects of operating parameters and the effects of interactions between these parameters on the response values were determined. In this context, coefficient factor of each parameter on its own and interrelation coefficients between them were determined and regression model equations for each response were constructed by using these coefficients. Following these, significance order of parameters was

determined and each parameter was evaluated in terms of scandium recovery and also impurity removal. As the consequence of the determination of these coefficient factors and regression model equations, the optimum parameters that provides the maximum amount of removal of gangue minerals with as minimal scandium loss as possible were determined. Investigated effects of operating parameters on responses, namely scandium recovery, calcium removal, magnesium removal, and silicon removal, are given in Table 5.13. Correspondingly, constructed regression model equation for each response are given.

Table 5.13 Factors and corresponding effects on response values

Factor	Effect on Sc.	Effect on Si.	Effect on Mg.	Effect on Ca.
	Rec. (%)	Rej. (%)	Rej. (%)	Rej. (%)
Solid Ratio (A)	5.60	-11.43	-11.33	-14.27
Retention Time (B)	2.63	-3.10	-4.08	-6.05
Rotation Speed (C)	-0.087	1.48	1.19	1.36
AB	-2.86	1.58	3.90	5.63
AC	-0.16	-1.61	-0.45	-0.084
BC	1.01	1.86	-1.26	-2.73
ABC	-0.31	-2.05	-0.31	-0.25

$$\text{Scandium recovery (\%)} = 84.42 + 2.80* A + 1.32* B - 0.044* C - 1.43* AB - 0.080* AC + 0.51* BC - 0.15* ABC$$

$$\text{Silica rejection (\%)} = 26.86 - 5.72* A - 1.55* B + 0.74* C + 0.79* AB - 0.80* AC + 0.93* BC - 1.03* ABC$$

$$\text{Magnesium rejection (\%)} = 28.86 - 5.67* A - 2.04* B + 0.59* C + 1.95* AB - 0.22* AC - 0.63* BC - 0.16* ABC$$

$$\text{Calcium rejection (\%)} = 42.16 - 7.13 * A - 3.02 * B + 0.68 * C + 2.82 * AB - 0.042 * AC - 1.37 * BC - 0.12 * ABC$$

Based on the obtained effects of parameters and regression model equations for the response values, significance order of parameters in decreasing order was found as following;

- 1) Solid-liquid ratio
- 2) Retention time
- 3) Rotation speed

In these premises, it was observed that the solid-liquid ratio was the most influential factor on the attrition scrubbing process of Eskişehir-Mihalıççık Scandium Ore. Considering the attrition scrubbing as a washing and de-sliming process, the effect of abrasion is the most crucial process that influences the efficiency of the process, thereby increase of the solid-liquid ratio also intensified the collision and friction between particles, which led to a decrease of the removal rate of impurities, while increasing the recovery of scandium. Basically, this might be defined as an over-scrubbing effect or namely size reduction effect due to the intensive collisions. In more details, as it was seen previously, increase of the solid-liquid ratio increased the recovery of scandium, while it led to a drastically decrease in the removal rates of impurities such as Ca, Mg, and Si. As the second important parameter, retention time was also effective in terms of scandium recovery and impurity removal. Due to the increased number of collisions with the increasing retention time, scandium recovery was increased but also the rate of removal for the impurities decreased. Apart from these, effect of the rotation speed on the scandium recovery and the impurity removal was less than the parameters of solid-liquid ratio and retention time. At this stage, in order to optimize the parameters, investigation of the effect of the interrelations between parameters was considered as beneficial.

Based on the obtained effects of interrelations on response values, it was seen that the correlation between solid-liquid ratio and retention time had a considerable effect on the removal of impurities. On the other hand, the effect of the interrelation between solid-liquid ratio and rotation speed seems to be negligible similar to the effect of the relation between solid-liquid ratio, retention time and rotation speed. In addition to these, although the interrelation between retention time and rotation speed was considered as negligible, it might have a comparably smaller effect on the response values. Considering the above mentioned effects of parameters, the attrition scrubbing experiments were optimized by the help of the statistical model and the optimum parameters that provides the maximum amount of removal of gangue minerals together with the minimal scandium loss as possible.

According to the optimization study of the attrition scrubbing experiments, the combination of parameters that provided the most efficient results were as following;

- 25 % solid-liquid ratio
- 90 minutes of retention time
- 1200 rpm of rotation speed

As the result of the experiments carried out under the above mentioned optimum conditions, feed that initially contains 105 ppm Sc was enriched to 120 ppm at 85.06 % Sc recovery. In addition to these, 42.93 % of Ca, 30.88 % of Mg, and 33.73 % of Si were removed in the oversize material, namely tailings fraction. As the result of the conducted optimization study, it was seen that the removal of impurities such as Ca, Mg, and Si was possible by performing attrition scrubbing. In conjunction with this, rejection of gangue minerals that would cause negative impacts during the subsequent hydrometallurgical treatment of this type of the ore, at the highest possible extent, was achieved. The average chemical composition of the obtained pre-concentrate is given in Table 5.14.

Table 5.14 Average chemical composition of the obtained pre-concentrate from the attrition scrubbing experiments

Sample Code	Al (%)	Ca (%)	Fe (%)	Mg (%)	Sc (ppm)	Si (%)
Pre-Concentrate	4.35	3.45	9.35	4.99	120	23.04

At this stage, further attrition scrubbing experiments were carried out using optimum parameters in order to prepare a pre-concentrate and also for using as a feed material in the following pre-concentration studies. In addition to these, a detailed characterization study was performed to the obtained attrition scrubbed sample in order to evaluate the results of attrition scrubbing experiments in more detail.

5.2.1.3 Characterization of the Attrition Scrubbed Sample

The attrition scrubbed sample was characterized in detail following the optimization of the attrition scrubbing experiments, in order to evaluate the results of the attrition scrubbing experiments and also to provide insight for the subsequent pre-concentration studies. In the context of the characterization studies, mineralogical, XRD (X-Ray Diffraction) analysis of the pre-concentrate and tailings were performed to identify the surface characteristics and the compositional characteristics of the sample qualitatively. In that way, separation of the impurity minerals and the clay minerals were examined in more detail. Moreover, size classification of the attrition scrubbed sample was completed, by wet sieving for the purpose of identifying the weight distributions and also the elemental distributions with respect to the sizes of the particles. As the result of wet sieving, consistency of the results obtained from the attrition scrubbing experiments were checked and also the present weight and elemental distribution were revealed.

Metallurgical results obtained from the wet sieving of the attrition scrubbed sample are given in Table 5.15. As it can be seen from the wet sieving results, 50.50 % of the attrition scrubbed sample was under 20 μm , which indicated that the clay minerals previously concentrated at coarser sizes were successfully liberated and dispersed. According to the size classification of the sample, it was also observed that

approximately 23.50 % of the sample was removed as the tailings fraction, which was consistent with the results of the attrition scrubbing experiments. As it was previously determined, impurities such as Ca, Mg, and Si enriched in relatively coarser fractions whereas elements such as Al, Fe, and Sc enriched in relatively finer size fractions due to the clay minerals of the ore. Following the attrition scrubbing, as it was expected Al, Fe, and Sc content of the finer size fractions were increased. Approximately 85 % of scandium was concentrated under 417 μm , which was the classification size of the attrition scrubbing experiment. Also, Al and Fe behaved in the same way as Sc that proved the liberation and dispersion of clay minerals concentrated on coarser size fractions.

Table 5.15 Metallurgical results of the wet sieving of the attrition scrubbed ore sample

Fraction (mm)	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
+9,53	2.78	1.81	1.37	10.25	5.54	5.25	1.63	7.45	4.05	72	1.82	32.10	3.79
-9,53 + 6,35	3.17	1.81	1.56	10.04	6.18	4.87	1.72	7.40	4.58	67	1.93	31.59	4.26
-6,35 + 3,35	4.28	2.12	2.48	10.77	8.98	5.15	2.47	7.34	6.15	78	3.03	31.02	5.65
-3,35 + 2,14	2.76	2.09	1.58	10.28	5.53	4.89	1.51	7.15	3.86	75	1.89	31.55	3.71
-2,14 + 1,168	4.36	1.99	2.37	9.15	7.76	4.90	2.39	6.55	5.58	69	2.73	31.30	5.81
-1,168 + 0,85	1.65	1.67	0.75	7.62	2.45	4.58	0.85	5.95	1.92	59	0.89	32.49	2.28
-0,85 + 0,60	2.05	1.82	1.01	7.39	2.94	4.92	1.13	5.89	2.36	61	1.14	31.20	2.72
-0,60 + 0,417	2.08	1.85	1.05	7.45	3.01	5.00	1.16	5.85	2.37	62	1.17	28.88	2.55
-0,417 + 0,210	5.78	2.32	3.65	7.50	8.44	6.18	3.99	5.84	6.60	74	3.87	24.05	5.92
-0,210 + 0,106	7.75	2.54	5.36	7.05	10.63	6.51	5.63	5.75	8.71	80	5.60	23.65	7.80
-0,106 + 0,071	3.46	2.66	2.50	7.07	4.76	6.95	2.69	5.75	3.89	82	2.58	23.55	3.47
-0,071 + 0,053	3.92	2.86	3.05	7.80	5.94	7.37	3.23	5.70	4.36	88	3.14	22.75	3.79
-0,053 + 0,038	4.32	3.20	3.77	6.42	5.39	8.27	3.99	5.69	4.80	95	3.74	22.30	4.10
-0,038 + 0,020	1.14	2.97	0.93	5.97	1.33	7.80	1.00	5.67	1.27	86	0.89	22.25	1.08
-0,020	50.50	4.99	68.59	2.15	21.12	11.81	66.63	4.00	39.49	143	65.57	20.05	43.08
Total	100.00	3.67	100.00	5.14	100.00	8.95	100.00	5.12	100.00	110	100.00	23.51	100

Based on the metallurgical results of the wet sieving of the attrition scrubbed ore, it was observed that approximately 42 % of Ca, 31 % of Mg, and 31 % of Si were concentrated over 417 μm , which was also discarded as the tailings fraction. In addition to these, relative enrichment of impurities such as Ca, Mg, and Si in the coarser fractions indicated that impurity containing minerals were successfully removed as the result of the attrition scrubbing process. This case was also proven by the applied mineralogical analysis on the pre-concentrate and the tailings fractions obtained from the attrition scrubbing. X-ray diffractograms of the pre-concentrate and tailings fractions are given in Figure 5.13 and 5.14.

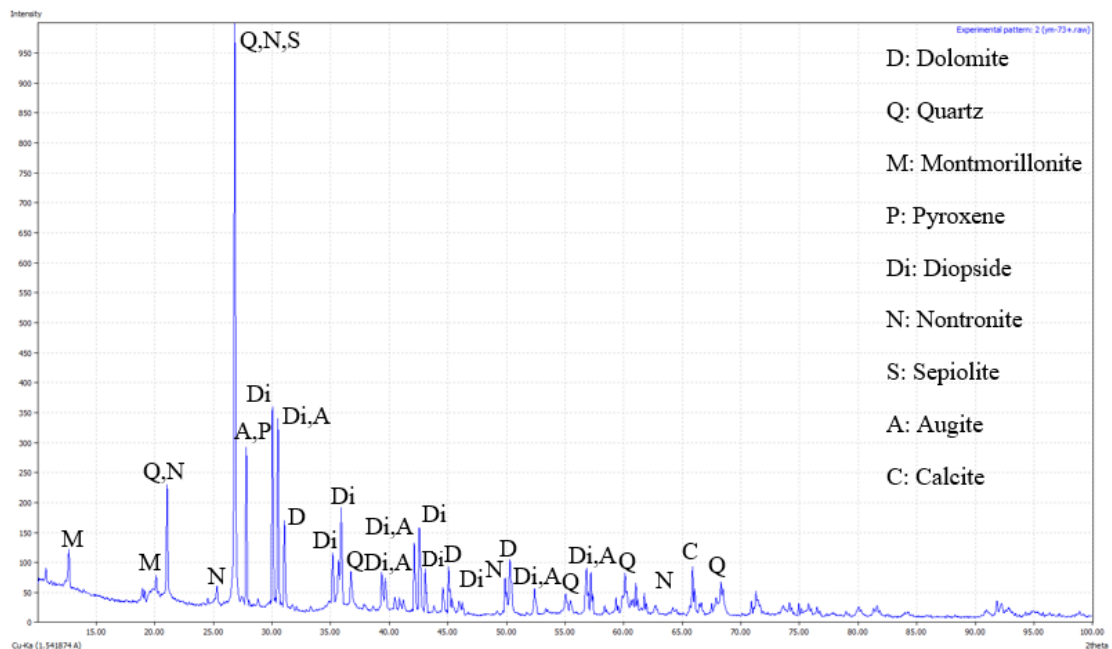


Figure 5.13 X-ray diffractogram of the tailings fraction obtained from attrition scrubbing

The X-ray diffractogram of the tailings fraction showed that quartz and nontronite were the dominating mineral phases. At that point, peaks of them were frequently overlapped and created the most intense peak at 26.63° . Relatively moderate intense characteristic peaks of quartz were observed at 20.85° , 36.54° , 54.90° , 59.98° , and 68.13° . Also, moderate intense characteristic peaks of nontronite were observed at 20.85° , 25.55° , 50.17° , and 61.29° . In addition to these, characteristic peaks of

dolomite were observed at 30.97°, 45.01°, and 50.45°. Also, relatively moderate intense peaks of diopside were observed at 29.50°, 34.76°, 35.70°, 39.19°, 41.95°, 44.43°, and 57.01°. Moreover, characteristic peaks of augite were observed at 27.96°, 29.50°, 39.19°, 41.95°, and 57.01°. Moderate intense characteristic peak of calcite was observed at 65.90°. In addition, the characteristic peak of pyroxene was overlapped with the characteristic peak of augite at 27.96°. Furthermore, lower intensity characteristic peaks of sepiolite and montmorillonite were observed.

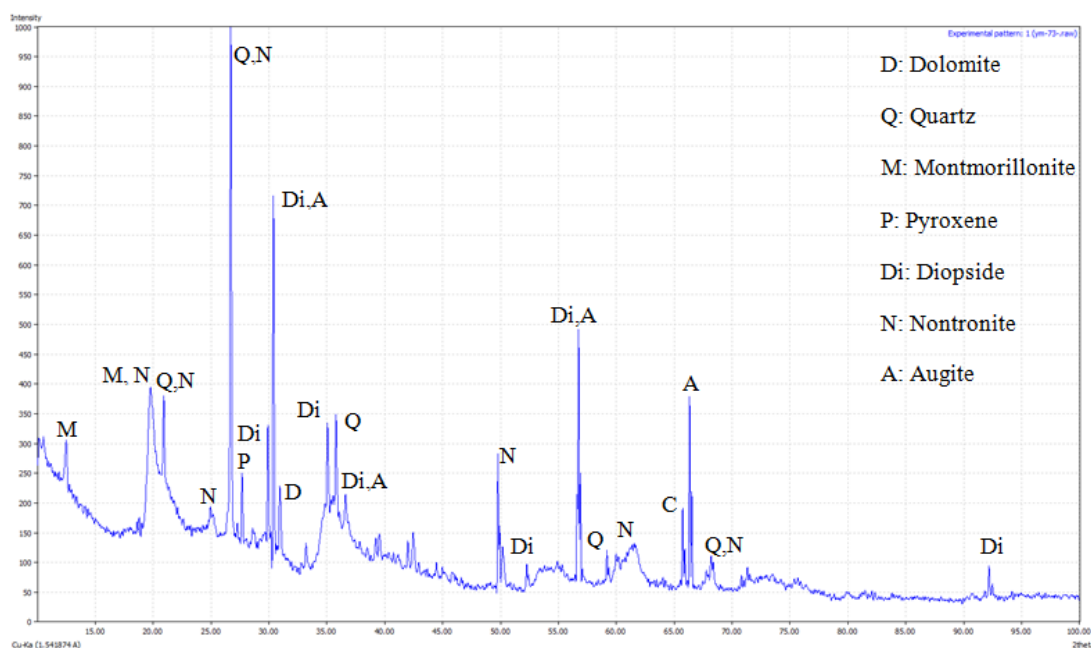


Figure 5.14 X-ray diffractogram of the pre-concentrate fraction obtained from attrition scrubbing

The X-ray diffractogram of the pre-concentrate fraction showed that quartz, nontronite and montmorillonite were the dominating mineral phases. At that point, peaks of quartz and nontronite were frequently overlapped and created the most intense peak at 26.63°. Relatively moderate intense characteristic peaks of quartz were observed at 20.85°, 36.54°, 59.98°, and 68.13°. Also, moderate intense characteristic peaks of nontronite were observed at 20.85°, 25.55°, 50.17°, 61.29°, and 68.13°. In addition to these, characteristic peaks of motmorillonite were observed at 12.46° and 19.94°. Also, relatively moderate intense peaks of diopside were observed at 29.50°, 34.76°, 35.70°,

39.19°, 41.95°, 44.43°, and 57.01°. Moreover, characteristic peaks of augite were observed at 27.96°, 29.50°, 39.19°, 41.95°, and 57.01°. In addition, the characteristic peak of pyroxene was observed at 27.96°. Less intense characteristic peaks of calcite and dolomite were observed at 65.90° and 30.97°, respectively.

Comparing the results of the mineralogical, XRD analyses, it was inferred that Ca and Mg containing impurity minerals such as calcite and dolomite were enriched in the tailings fraction. Furthermore, due to the liberation and dispersion of the clay minerals as a result of attrition scrubbing, smectite group clay minerals, as nontronite and montmorillonite, were enriched in the pre-concentrate fraction. Based on these, it was seen that the results of the mineralogical analysis were consistent with the wet sieving analysis of the attrition scrubbed ore. Both characterization studies, including wet sieving and XRD analyses, proved that the impurity containing minerals were successfully removed as the result of the performed attrition scrubbing and also a pre-concentrate enriched in scandium bearing clay minerals was obtained. As the result of the attrition scrubbing study, it was seen that the process successfully achieved its goal. Moreover, obtained pre-concentrate from the attrition scrubbing study was used as a feed material for the further pre-concentration studies in order to remove impurities still contained in it and to obtain a further pre-concentrate relatively enriched in scandium content.

5.2.2 Enhanced Gravity Separation of the Scandium Ore by Using Falcon Concentrator

As the second step of the pre-concentration studies, enhanced gravity separation of Eskişehir-Mihalıççık Scandium ore was carried out by using falcon concentrator in order to remove the impurities containing in the previously obtained pre-concentrate such as Ca, Mg, and Si and also to obtain a further pre-concentrate enriched in scandium content. Falcon concentrator experiments were carried out according to the methodology given before. Based on the characterization of the run of mine and the pre-concentrate obtained from the attrition scrubbing experiments, it was observed that scandium was mainly concentrated in the finer size fractions and the impurity minerals were mainly concentrated in the coarser fractions. In this context, some part of the impurity mineral content of the run of mine was rejected by the attrition scrubbing experiments previously. Nevertheless, it was seen that the pre-concentrate obtained from the attrition scrubbing also contained impurity elements such as Ca, Mg, and Si, so the reason for the application of the falcon concentrator was the removal of these impurities as well as obtaining a pre-concentrate enriched in scandium. Considering that the enhanced gravity separation, namely falcon concentrator, was mainly used for the recovery of fine and ultrafine particles under the influence of ultrahigh gravitational forces (El-Midany and Ibrahim, 2011), application of the falcon concentrator on the previously obtained pre-concentrate was considered as suitable. In addition to these, it was stated that the falcon concentrator can provide enhanced gravitational force up to 300 g that leads to successful separation at both lower gravity points and finer sizes typically lower than 100 μm (El-Midany and Ibrahim, 2011), therefore it was thought that previously obtained pre-concentrate could be further concentrated by using falcon concentrator. Based on the lower specific gravity of the ore mainly due to the clay content and particle size distribution of the pre-concentrate, approximately 80 % of it was under 106 μm , it was thought that there was no need to make any further size reduction prior to the falcon concentration.

Falcon concentrator experiments were performed in order to concentrate the Sc-bearing clay minerals, which had lower specific gravity and finer particle size distribution, in the lighter fraction and to concentrate Ca, Mg, and Si containing impurity minerals in the heavier fraction. In this context, the extent of the falcon concentrator experiments were first evaluated in terms of scandium recovery in lighter fraction and silica removal, magnesium removal and calcium removal in heavier fraction. Therefore, lighter fraction was called the pre-concentrate, while the heavier fraction was defined as the tailing products. General flowsheet for the carried out falcon concentrator experiments is given in Figure 5.15.

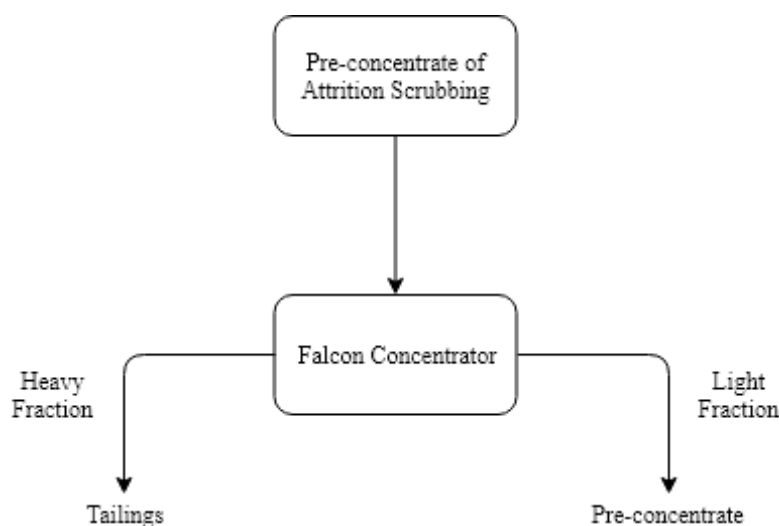


Figure 5.15 General flowsheet for the falcon concentrator experiments

Within the scope of the falcon concentrator experiments, the effects of previously selected operational parameters, namely centrifugal force and fluidization water pressure, on scandium recovery, silicon removal, magnesium removal and calcium removal were investigated. At this point, due to the limitations of the falcon concentrator, feeding rate was manually adjusted and tried to be kept constant for all experiments. In addition to these, the solid content of the feed slurry was adjusted to 20 % and kept constant during the experiments, thus it was thought that 20 % solid content was enough to constitute the hindered settling conditions in the presence of ultrafine and clayey particles, which was required for an efficient separation (Zhang,

et al., 2011; Boylu, 2013). Following the falcon concentrator experiments, optimum parameters were determined in terms of grade and recovery of the products and tailings. In order to determine the effects of parameters on the falcon concentrator process, falcon concentrator experiments were conducted by varying one parameter at a time while keeping the other parameter fixed.

Based on the carried out falcon concentrator experiments, experimental conditions and their metallurgical results for identifying the effects of centrifugal force and fluidized water pressure are given in Tables 5.16, 5.17, and 5.18. As it can be seen from the metallurgical results of the experiments performed at 2 psi water pressure, weight yield of the tailings namely the heavy fraction was increased by increasing the centrifugal force from 20 g to 300 g. In more details, 8.87 % of the feed material was subjected to tailings at 20 g centrifugal force, while 19.76 %, 27.52 %, and 25.71 % of the feed material were subjected to the tailings at centrifugal forces of 150 g, 275 g, and 300 g, respectively. Considering the finer size distribution and lower specific gravity of the feed material, it was seen that centrifugal force has a significant effect on the separation of the lighter and heavier fractions. It was observed that due to the increase in the centrifugal force, heavier particles were directly stratified without being exposed to the effect of the fluidization zone and thus the stratification layer became compact and the drag forces were not enough to remove the relatively lighter particles entrapped in these layers. Moreover, heavier particles of smaller sizes were substituted with lighter particles and thereby the relatively lighter particles were subjected to the central part of the spinning bowl, which was followed by the removal of them by the exerted drag forces on them. Also, the change in the centrifugal force directly affected the recovery of scandium and the removal of impurities such as Ca, Mg, and Si. In this context, scandium recovery was 94.30 % for the experiment carried out at 20 g centrifugal force. Scandium recoveries for the falcon concentrator experiments performed at 150 g, 275g, and 300 g were 90.18 %, 78.99 %, and 83.00 %, respectively. The metallurgical results for scandium indicated that the Sc-bearing clay minerals were concentrated in the lighter fraction but also the relatively heavier Sc-bearing diopside and pyroxene minerals were concentrated at heavier fraction with the increase of the

centrifugal force over a certain value. Therefore, scandium recovery tend to decrease with the increased centrifugal force from 150 g to 300 g. On the other hand, removal rates of Ca, Mg, and Si were 27.71 %, 12.47 %, and 9.42 % for the experiment performed at 20 g centrifugal force. Also, removal rates of impurities were perceptibly increased by increasing the centrifugal force from 20 g to 300 g. Calcium removal rates for the experiments performed at 150 g, 275 g, and 300 g were 50.24 %, 60.14 %, and 57.01 %, respectively. As well as Ca, removal rates of Mg were also increased with the increasing centrifugal force as 26.65 %, 36.26 %, and 33.45 %, respectively. Considering that most of the silicon content of the feed material originated from the clay mineral content of the material, it was thought that the silicon to be removed by the heavier fraction was not expected to be too much. In this context, although not as much as calcium removal, removal rates of silicon for the experiments performed at 150 g, 275 g, and 300 g were 20.14 %, 28.43 %, and 33.10 %, respectively. As the results of the experiments performed at 2 psi water pressure by varying the centrifugal forces from 20 to 300 g, it was seen that the removal rates of impurities such as Ca, Mg, and Si were increased by increasing the centrifugal force and also the increase of centrifugal force led to considerable changes especially in the removal rate of calcium. Contrary to that, it was also observed that the increase of the centrifugal force above a certain value, namely 150 g, led to decrease in the recovery of scandium in the lighter fraction. In this case, it was considered that it might be feasible to reject relatively heavier and coarser particles as impurity minerals with a minimum loss of scandium at mid centrifugal force values. As it can be seen from the metallurgical results of the experiments, removal of impurities such as Ca, Mg, and Si was considered as possible with enhanced gravity separation by using falcon concentrator.

Table 5.16 The effect of centrifugal force on the enhanced gravity separation of Eskişehir-Mihalıççık Scandium ore at 2 psi water pressure

Exp. Code	Fraction	Water Pressure (Psi)	Centrifugal Force (Hz/g)	Weight (%)	Ca Rec. (%)	Mg Rec. (%)	Sc Rec. (%)	Si Rec. (%)
1	Light	2.00	20 / 20	91.13	72.29	87.53	94.30	90.58
	Heavy			8.87	27.71	12.47	5.70	9.42
	Total			100.00	100.00	100.00	100.00	100.00
2	Light	2.00	55 / 150	80.24	49.76	73.35	90.18	79.86
	Heavy			19.76	50.24	26.65	9.82	20.14
	Total			100.00	100.00	100.00	100.00	100.00
3	Light	2.00	75 / 275	72.48	39.86	63.74	78.99	71.57
	Heavy			27.52	60.14	36.26	21.01	28.43
	Total			100.00	100.00	100.00	100.00	100.00
4	Light	2.00	78 / 300	74.29	42.99	66.55	83.00	66.90
	Heavy			25.71	57.01	33.45	17.00	33.10
	Total			100.00	100.00	100.00	100.00	100.00

As it can be seen from the metallurgical results of the experiments performed at 6 psi fluidization water pressure, weight yield of the tailings namely the heavy fraction was increased after centrifugal force of 20 g and almost fixed after centrifugal force of 150 g. For the falcon concentrator experiment performed at 20 g, where the drag forces originated from the fluidization water pressure were the dominating forces, whole material was subjected to the lighter fraction without being exposed to the stratification cycle. Therefore, an efficient separation was not observed for that case. On the contrary, weight yields of the tailings for the experiments performed at 150 g, 275 g, and 300 g were approximately 15 %, which also indicated that the applied centrifugal forces were enough to push the heavier particles through the stratification cycle occurred on the bowl. As it was observed in the weight yields of the tailings, scandium recoveries were not changed drastically between centrifugal forces of 150 g and 300 g. Scandium recoveries for the experiments performed at centrifugal forces of 150 g, 275 g, 300 g were 90.46 %, 91.10 %, and 88.38 %, respectively. As well as the scandium recovery, removal rates of Ca, Mg, and Si did not changed extremely between centrifugal forces of 150 g and 300 g. In this context Ca removal rates for the experiments performed at 150 g, 275 g and 300 g were 41.99 %, 41.38 % and 43.15 %, respectively. As in Ca removal, Mg removal rates also represented the same trend as 21.94 %, 21.05 % and 24.41 %. Comparing the Si removal rates of the experiments

performed at 6 psi and the experiments performed at 2 psi, it was inferred that due to the lower specific gravity of Si containing impurities, removal rates for the experiments performed at 6 psi was lower because of the exposure of Si containing minerals to the light fraction. Related to this, Si removal rates were approximately 16 %. Based on the obtained metallurgical results from the falcon concentrator experiments performed at 6 psi, it was observed that the increase of fluidization water pressure, namely the drag force, increased the scandium recovery at a certain amount but also decreased the impurity removal rate, which attracted the attention as the negative effect of increasing the fluidization water pressure.

Table 5.17 The effect of centrifugal force on the enhanced gravity separation of Eskişehir-Mihalıççık Scandium ore at 6 psi water pressure

Exp. Code	Fraction	Water Pressure (Psi)	Frequency (Hz/G)	Weight (%)	Ca Rec. (%)	Mg Rec. (%)	Sc Rec. (%)	Si Rec. (%)
5	Light	6.00	20 / 20	99.30	97.32	98.96	99.58	99.26
	Heavy			0.70	2.68	1.04	0.42	0.74
	Total			100.00	100.00	100.00	100.00	100.00
6	Light	6.00	55 / 150	84.18	58.01	78.06	90.46	83.57
	Heavy			15.82	41.99	21.94	9.54	16.43
	Total			100.00	100.00	100.00	100.00	100.00
7	Light	6.00	75 / 275	85.26	58.62	78.95	91.10	84.66
	Heavy			14.74	41.38	21.05	8.90	15.34
	Total			100.00	100.00	100.00	100.00	100.00
8	Light	6.00	78 / 300	82.29	56.85	75.59	88.38	81.63
	Heavy			17.71	43.15	24.41	11.62	18.37
	Total			100.00	100.00	100.00	100.00	100.00

Based on the metallurgical results of the experiments performed at 10 psi water pressure, it was observed that there was no dramatical change in both the weight yields and the removal rates of impurities. In this context, weight yield of the heavy fractions, namely tailings, fluctuated between 2.45 % and 4.59 %, which were also thought as lower yields for an efficient separation. Furthermore, it was indicated that fluidization water pressure was assisted the removal of the lighter material by means of the effect of drag force (Nayak and Pal, 2013) according to the previous studies on the effect of fluidization water pressure. In addition, in the case of the drag forces dominating rather than the centrifugal forces, relatively heavier particles were subjected to lighter

fraction besides the lighter particles. As it can be seen from the metallurgical results, due to the finer particle size distribution and the relatively lighter particle content of the feed material, drag forces directly removed both the heavier and lighter fraction without exposing them to the stratification cycle. Within this scope, although the scandium recovery was observed to be higher, removal rates of the impurities such as Ca, Mg, and Si were also lower for a properly conducted separation. As the consequence of the results obtained from the experiments performed at 10 psi fluidization water pressure, it was observed that the fluidization water pressure was too high to make an efficient separation and thus the whole feed material was subjected to the lighter fraction by the help of the drag forces. For these reasons, it was stated that further increase of the fluidization water pressure could not provide any positive effect on the separation process. Therefore, the separation was not performed with higher fluidization water pressure values. The detailed metallurgical results of the enhanced gravity separation of Eskişehir-Mihalıççık scandium ore by using falcon concentrator are given in Appendix E (Table E.1-E.3).

Table 5.18 The effect of centrifugal force on the enhanced gravity separation of Eskişehir-Mihalıççık Scandium ore at 10 psi water pressure

Exp. Code	Fraction	Water Pressure (Psi)	Frequency (Hz/G)	Weight (%)	Ca Rec. (%)	Mg Rec. (%)	Sc Rec. (%)	Si Rec. (%)
9	Light	10.00	20 / 20	97.55	91.60	96.43	98.45	98.06
	Heavy			2.45	8.40	3.57	1.55	1.94
	Total			100.00	100.00	100.00	100.00	100.00
10	Light	10.00	55 / 150	95.95	85.95	93.99	97.38	96.21
	Heavy			4.05	14.05	6.01	2.62	3.79
	Total			100.00	100.00	100.00	100.00	100.00
11	Light	10.00	75 / 275	95.41	84.97	93.34	97.13	95.52
	Heavy			4.59	15.03	6.66	2.87	4.48
	Total			100.00	100.00	100.00	100.00	100.00
12	Light	10.00	78 / 300	96.73	89.34	95.37	97.97	96.59
	Heavy			3.27	10.66	4.63	2.03	3.41
	Total			100.00	100.00	100.00	100.00	100.00

By evaluating the metallurgical results obtained from the falcon concentrator experiments in terms of scandium recovery, calcium removal, magnesium removal, and silicon removal, effects of the operating parameters, namely centrifugal force and fluidization water pressure were investigated. Based on the performed experiments, optimum parameters that provide the maximum amount of removal of gangue minerals with the minimum scandium loss as possible were determined. In these premises, it was inferred that the centrifugal force has a significant effect on the recovery of scandium and removal of impurities until a certain point of fluidization water pressure is reached. In the case of the fluidization water pressure exceeded a certain value, it became the dominating force on the separation process, which also prevented the occurrence of an efficient separation. As it can be seen from the metallurgical results obtained from the falcon concentrator experiments, operating at lower fluidization water pressures was beneficial in terms of the removal of the relatively lighter gangue particles. In the case of higher centrifugal forces although the impurity removal rate was increased, the scandium recovery was decreased due to the relatively heavier Sc-bearing minerals as diopside and pyroxene were entrapped on the bowl of the falcon concentrator and subjected to the heavier fraction. In addition to these, by adjusting the centrifugal force at mid-values as 150 g, as much impurity as possible was removed

with the minimal possible loss of scandium. According to the performed falcon concentrator experiments, the parameter combination that provided the most efficient results were as followings;

- Centrifugal force of 150 g
- Fluidization water pressure of 2 psi

As the results of the experiments carried out under the above mentioned optimum conditions, previously attrition scrubbed feed material that initially contains 120 ppm Sc, was enriched to 138 ppm at 90.18 % at Sc recovery. In addition to these, 50.24 % of Ca, 26.65 % of Mg, and 20.14 % Si were removed in the heavier fraction namely tailings. Based on these results, it was seen that the removal of impurities such as Ca, Mg, and Si was possible with the performed enhanced gravity separation by using falcon concentrator. In conjunction with this, rejection of gangue minerals that would cause negative impacts during the subsequent hydrometallurgical treatment of this type of the ore, at the highest possible extent, was achieved. Chemical composition of the obtained pre-concentrate is given in Table 5.19.

Table 5.19 Chemical composition of the obtained pre-concentrate from the falcon concentrator experiments

Sample Code	Al (%)	Ca (%)	Fe (%)	Mg (%)	Sc (ppm)	Si (%)
Pre-Concentrate	4.98	2.62	11.21	5.24	138	27.43

Furthermore, a detailed characterization study including mineralogical, XRD analyses were performed on the obtained pre-concentrate in order to evaluate the results of the falcon concentrator experiments in more details.

5.2.2.1 Characterization of the Pre-Concentrate and Tailings Obtained from Falcon Concentrator

Following the determination of the optimum conditions for the falcon concentrator experiments, in order to evaluate the results of the falcon concentrator in more detail, mineralogical characterization namely XRD analysis was performed. In the context of the characterization studies, mineralogical, XRD analysis of the obtained pre-concentrate and tailings were completed to identify the surface characteristics and the compositional characteristics of the samples qualitatively. In this way, separation efficiency of the impurity containing minerals and Sc-bearing clay minerals were examined in more details. X-ray diffractograms of the pre-concentrate and tailings fractions are given in Figure 5.16 and 5.17.

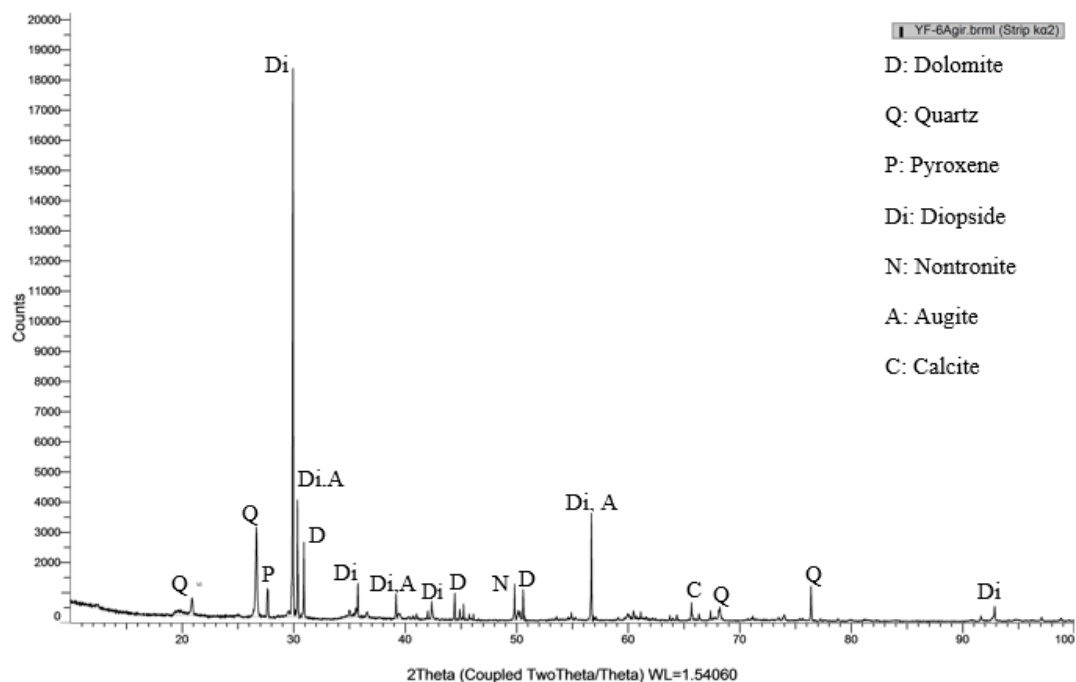


Figure 5.16 X-ray diffractogram of the tailings fraction obtained from the falcon concentrator

X-ray diffractogram of the tailings fraction showed that diopside was the dominating mineral phase and responsible for the most intense peak at 29.50 °. Relatively moderate intense characteristic peaks of diopside were observed at 35.70°, 39.19°, and 56.50°.

41.95°, 57.01°, and 92.50°. Also, moderate intense characteristic peaks of quartz were observed at 20.85°, 26.63°, 68.13°, and 76.50°. In addition to these, characteristic peaks of dolomite were observed at 30.97°, 45.01°, and 50.45°. By the way, moderate intense characteristic peak of calcite was observed at 65.90°. Moreover, characteristic peaks of augite were observed at 29.50°, 39.19°, and 57.01°. Furthermore, lower intensity characteristic peaks of nontronite and pyroxene were observed.

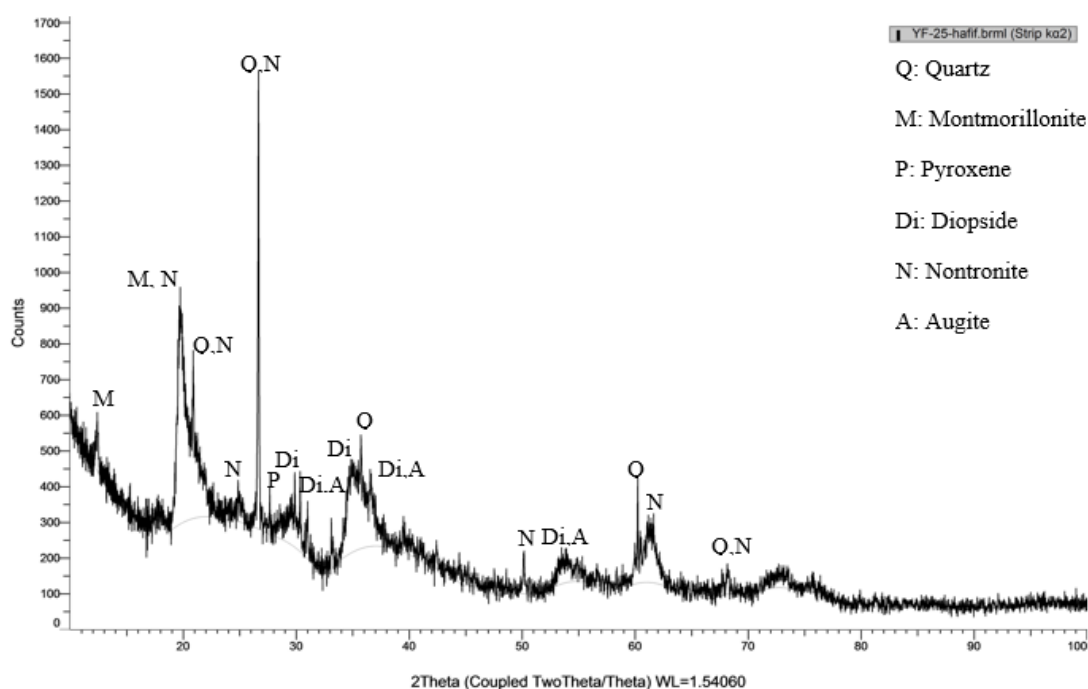


Figure 5.17 X-ray diffractogram of the pre-concentrate obtained from the falcon concentrator

X-ray diffractogram of the pre-concentrate obtained from the falcon concentrator showed that quartz, nontronite, and montmorillonite were the dominating mineral phases. In this case, peaks of quartz and nontronite were frequently overlapped and created the most intense peak at 26.63°. Relatively moderate intense characteristic peaks of quartz were observed at 20.85°, 36.54°, 59.98° and 68.13°. Also, moderate intense characteristic peaks of nontronite were observed at 20.85°, 25.55°, 50.17°, 61.29°, and 68.13°. In addition to these, characteristic peaks of montmorillonite were observed at 12.46° and 19.94°. Also, relatively moderate intense peaks of diopside

were observed at 29.50°, 34.76°, 35.70°, 39.19°, and 54.05°. Moreover, characteristic peaks of augite were observed at 29.50°, 41.95°, and 54.05°. Furthermore, lower intensity characteristic peak of pyroxene was observed. Apart from these, there were no observed characteristic peaks of calcite and dolomite.

As the result of the mineralogical characterization of the pre-concentrate and tailings obtained from the falcon concentrator, it was observed that Ca and Mg containing impurity minerals such as calcite and dolomite were concentrated in the tailings fraction. In addition to these, concentration of clay minerals such as montmorillonite and nontronite in the pre-concentrate material namely light fraction indicated that Sc-bearing clay minerals were successfully enriched in the pre-concentrate fraction as the result of the applied enhanced gravity separation by using falcon concentrator. Based on these, it was seen that the mineralogical analyses were seen to be consistent with the chemical analyses of the pre-concentrate and the tailings products and proved that the impurity containing minerals were successfully removed in the tailings fraction. As another important point, a pre-concentrate that was enriched in scandium was obtained as the result of the falcon concentrator experiments. So, it was seen that the falcon concentration process successfully achieved its goal. Following parts of the study consist of the methods that were thought to be an alternative path to enhanced gravity separation, by using falcon concentrator, in order to obtain a pre-concentrate that was enriched in scandium content.

5.2.3 Magnetic Separation of the Scandium Ore

As an alternative pre-concentration method to enhanced gravity separation by using falcon concentrator, magnetic separation of Eskişehir-Mihalıççık Scandium ore was performed by using high intensity wet magnetic separator in order to remove the diamagnetic impurity minerals namely calcite, dolomite, and quartz contained in the previously obtained pre-concentrate and also to obtain a further pre-concentrate enriched in scandium content. Magnetic separation experiments were performed according to the methodology given before. Based on the results obtained from both

the characterization study performed on the representative trench sample, including mineralogical analysis and wet sieving analysis, and also on the obtained pre-concentrate from attrition scrubbing experiments, it was determined that scandium was concentrated in the finer size fractions mainly including weakly paramagnetic clay minerals namely nontronite and montmorillonite besides the paramagnetic silicate minerals as diopside and pyroxene. In this context, it was thought that the separation of Sc-bearing weakly paramagnetic and paramagnetic minerals from their diamagnetic counterparts namely calcite, dolomite and quartz could be possible by using high intensity wet magnetic separator. In addition to these, it was also seen that the pre-concentrate obtained from attrition scrubbing also contained impurity elements such as Ca, Mg, and Si based on its calcite, dolomite, and quartz content, so the reason for the application of magnetic separation was the removal of these impurities as well as obtaining a pre-concentrate enriched in scandium. According to the fundamental principles of the magnetic separation process, diamagnetic minerals are not attracted by magnetic forces and thus they are concentrated in the non-magnetic fraction, while ferromagnetic, paramagnetic, and weakly paramagnetic minerals are concentrated in the magnetic fraction regarding the intensity of the magnetic field to be applied (Wills and Finch, 2016; Fuerstenau and Han, 2003). Furthermore, it was stated that weakly paramagnetic material contained in clayey ores can be recovered by using high intensity wet magnetic separation (Oberteuffer, 1974), so the application of the magnetic separation on the previously obtained pre-concentrate was seen to be consistent.

Magnetic separation experiments were performed in order to concentrate the Sc-bearing clay minerals, diopside and pyroxene, which were weakly paramagnetic and paramagnetic, respectively, in the magnetic concentrate. At the same time, concentration of impurity elements such as Ca, Mg and, Si by concentrating calcite, dolomite, and quartz in the non-magnetic fraction was also intended. In this context, the extent of the magnetic separation experiments were first evaluated in terms of scandium recovery in magnetic fraction and silica removal, magnesium removal and calcium removal in non-magnetic fraction. According to that, the magnetic fraction

was called pre-concentrate, while the non-magnetic fraction was called the tailing products. The general flowsheet for the performed magnetic separation experiments is given in Figure 5.18.

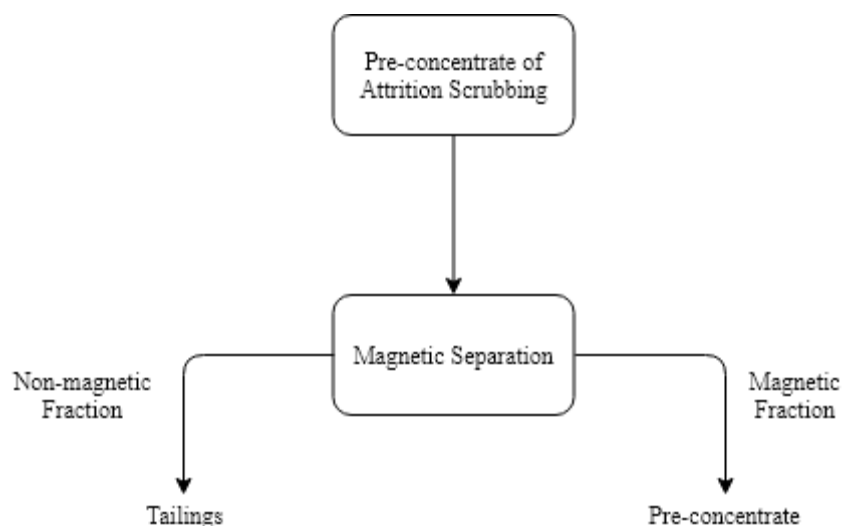


Figure 5.18 General flowsheet for the magnetic separation experiments

Within the scope of the magnetic separation experiments, the possibility of obtaining a pre-concentrate enriched in scandium by using high intensity magnetic separator and also the effects of the magnetic force on scandium recovery, silicon removal, magnesium removal, and calcium removal were investigated. Due to the limitations of the magnetic separator, the feeding rate and the volume of rinsing water was manually adjusted and tried to be kept fixed during the experiments. In addition to these, solid content of the feed slurry was adjusted to 20 % prior to the each magnetic separation experiment. Experimental design of the magnetic separation experiments were constructed by only varying the magnetic field intensity, while keeping the other parameters such as solid content, feeding rate of the feed slurry, and volume of rinsing water as fixed.

Based on the carried out magnetic separation experiments, experimental conditions and their metallurgical results are given in Table 5.20. As it can be seen from the metallurgical results of the magnetic separation experiments, weight yields of the magnetic fraction was decreased by decreasing the magnetic field intensity from 4000 G to 1000 G. In details, 24.62 % of the feed material was subjected to the magnetic fraction at magnetic field intensity of 4000 G, while 18.91 % and 7.59 % of the feed material were subjected to the magnetic fraction at magnetic field intensities of 2000 G and 1000 G, respectively. As it was expected, the weight yields of the magnetic fraction was decreased by decreasing the magnetic field intensity due to the relatively lower affection of low magnetic fields on the paramagnetic minerals. Thereby, weakly paramagnetic and paramagnetic minerals were not affected by lower magnetic field intensities and they behaved as diamagnetic minerals. Considering the obtained metallurgical results in terms of scandium recovery in the magnetic fraction, it was decreased with the decreasing magnetic field intensity due to that the weakly paramagnetic and paramagnetic Sc-bearing minerals were less affected in the presence of the relatively lower magnetic field intensities. In this context, scandium recovery for the experiments performed at 4000 G, 2000 G, and 1000 G were 21.91 %, 15.67 %, and 5.81 %, respectively. Based on these results, it was indicated that no favorable outcome from magnetic separation experiments in terms of scandium recovery was observed. By evaluating the removal rates of impurity elements, it was seen that they behaved in the same way as scandium. Although, they were mostly concentrated in the non-magnetic fraction due to the diamagnetic structure of them, scandium was also concentrated in the non-magnetic fraction. So, there was no observed successful separation of diamagnetic impurity minerals and paramagnetic Sc-bearing minerals. For the future works on the high intensity wet magnetic separation of this type of ore, it was thought that the application of higher magnetic field intensities might increase the scandium recovery in the magnetic fraction but also decrease the removal rates of impurities. The detailed metallurgical results of the magnetic separation of Eskişehir-Mihalıççık scandium ore by using high intensity wet magnetic separator are given in Appendix F (Table F.1).

Table 5.20 Experimental conditions and the metallurgical results of the magnetic separation of Eskişehir-Mihalıççık Scandium ore

Exp. Code	Fraction	Magnetic Intensity (G)	Weight (%)	Ca Rec. (%)	Mg Rec. (%)	Sc Rec. (%)	Si Rec. (%)
1	Mag.	4000	24.62	31.01	26.91	21.91	23.86
	Non. Mag.		75.38	68.99	73.09	78.09	76.14
	Total		100.00	100.00	100.00	100.00	100.00
2	Mag.	2000	18.91	23.66	20.13	15.67	18.04
	Non. Mag.		81.09	76.34	79.87	84.33	81.96
	Total		100.00	100.00	100.00	100.00	100.00
3	Mag.	1000	7.59	10.23	7.89	5.81	6.71
	Non. Mag.		92.41	89.77	92.11	94.19	93.29
	Total		100.00	100.00	100.00	100.00	100.00

As the result of the magnetic separation experiments, it was observed that the attrition scrubbed feed material could not be enriched by the performed magnetic separation under the present conditions. Moreover, attrition scrubbed Eskişehir-Mihalıççık scandium ore did not respond positively to the applied magnetic separation method. In this context, sc-recovery was increased with the increase of the magnetic field intensity but also the recoveries of impurity elements in the magnetic concentrate were increased. Therefore, it was thought that a sharp separation could not be obtained as the result of the applied magnetic separation. On the other hand, although it was thought that sc-recovery could be increased by increasing the magnetic field intensity above 4000 G, the removal rates of the impurity elements such as Ca, Mg, and Si were also decreased, which could prevent the required separation to obtain a pre-concentrate enriched in scandium. Considering all of the above mentioned results, a pre-concentrate that was enriched in scandium was not be able to be obtained as the result of the applied magnetic separation experiments. So, it was seen that the magnetic separation process could not achieve its goal. In this case, it was also thought that magnetic separation was not an alternative pre-concentration process to the enhanced gravity separation of Eskişehir-Mihalıççık Scandium ore by using falcon concentrator.

5.2.4 Flotation Concentration of the Scandium Ore

As the second alternative pre-concentration method to enhanced gravity separation by using falcon concentrator, flotation concentration of Eskişehir-Mihalıççık Scandium ore was performed in order to remove the impurity minerals namely calcite, dolomite and quartz contained in the previously obtained pre-concentrate and also to obtain a further pre-concentrate enriched in scandium content. Flotation concentration experiments were carried out according to the methodology given before. By examining the results obtained from both the characterization study performed on the representative trench sample, including mineralogical analysis, wet sieving analysis, and also on the obtained pre-concentrate from attrition scrubbing experiments, it was observed that the main impurities to be removed as the result of the pre-concentration studies were Ca, Mg, and Si. In this context, due to the similar flotation behaviors of Ca, Mg, and Si containing impurity minerals namely calcite, dolomite, and quartz, it was thought that Sc-bearing clay minerals could possibly be recovered by flotation concentration method. Therefore, it was thought that the Sc-bearing minerals could be recovered in the flotation concentrate while the gangue minerals could be removed in the tailings of the flotation. Even though the flotation concentration method was considered to be applicable for the Eskişehir-Mihalıççık Scandium ore, it could be challenging due to the clay content and the finer particle size of the attrition scrubbed ore. Moreover, because of the presence of excess slimes, viscosity of the pulp was increased and the mineral surfaces were coated by these slimes which prevented the interactions between mineral surfaces and collectors (Taner and Onen, 2016; Yu *et al.*, 2017). So, this situation could lead to decreased flotation recovery and selectivity. In addition to these, the dispersed state of scandium in the feed material namely concentrated in clay minerals and silicates like diopside and pyroxene, the flotation concentration of the ore was considered to be problematic due to the uncertainty of the desired mineral to be floated.

Flotation concentration experiments were performed in order to concentrate the Sc-bearing clay minerals, diopside and pyroxene in the flotation concentrate, while removing the impurity containing minerals such as calcite, dolomite, and quartz in the tailings fraction. In this context, following the examination of the mineralogical analysis and wet sieving analysis results, namely results of the characterization study of the obtained pre-concentrate from attrition scrubbing, two different flotation concentration flowsheets were designed and followed for the further pre-concentration of the ore. The flotation flowsheets were differentiated in terms of the target minerals to be floated and so the types of the collectors that were used. Applied flotation flowsheets and reagent consumptions are given in Figure 5.19 and Figure 5.20.

For the both flotation experiments, technical grade sulfuric acid was used in order to adjust the pH of the pulp to 6.0, which was considered to be suitable for the surface activation of the Sc-bearing minerals. Following this, based on the clay and slime content of the pulp 1500 g/t sodium silicate was added for both the dispersion of the agglomerates and the depression of the impurity minerals such as calcite, dolomite, and quartz. Within the scope of the first flotation experiment, Sc-bearing clay minerals were desired to be floated, thus Aero 6493 hydroxamate based collector was used as 200 g/t based on the previous studies for the flotation of Sc-bearing minerals (China Patent No. CN104263926A, 2014). Apart from these, in the context of the second flotation experiment, Aero 866 sulfonate based collector was used as 150 g/t in order to float the iron containing minerals. According to the previous characterization studies of the scandium ore, the strong correlation between scandium and iron was determined and thereby, it was thought that scandium could be concentrated in the flotation concentrate of the iron containing minerals. Following the conditioning of the pulp with the collector, Oreprep 533 alcohol based frother was used as 30 g/t in order to provide the formation of stable froths.

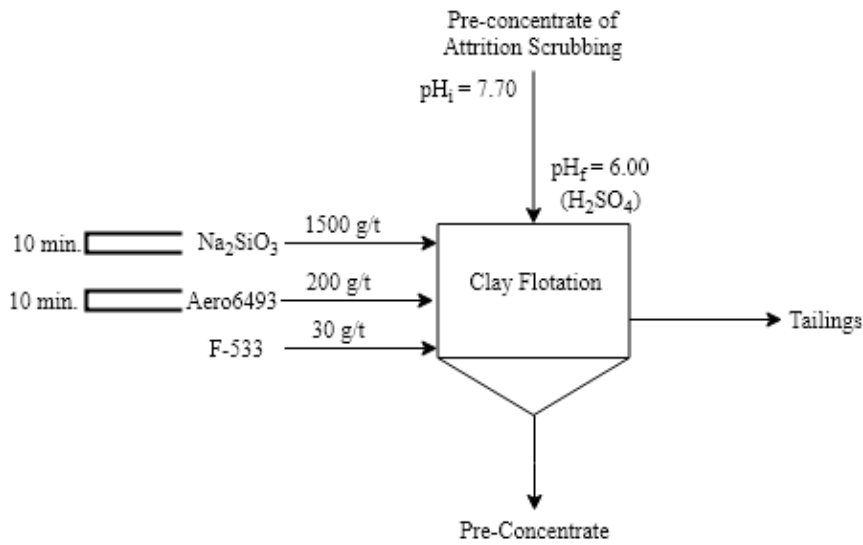


Figure 5.19 Proposed flowsheet and experimental conditions of the Sc-bearing clay minerals flotation

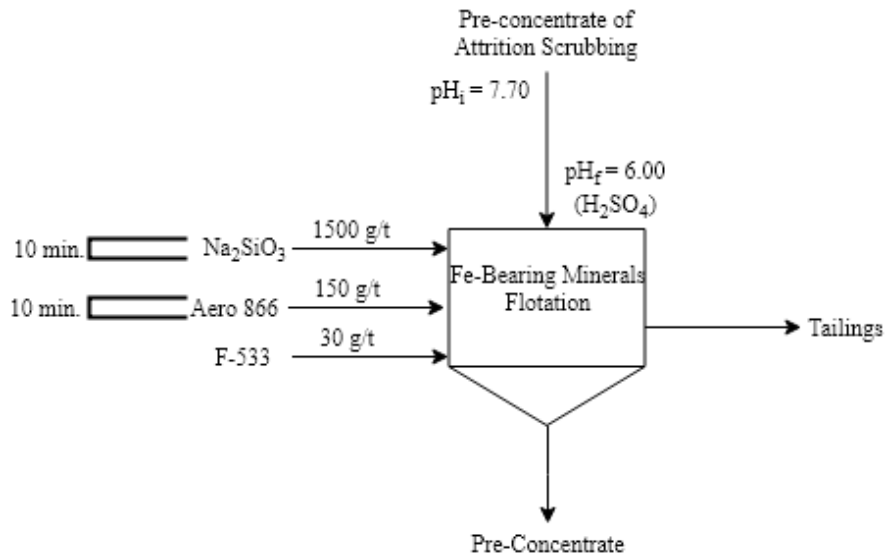


Figure 5.20 Proposed flowsheet and experimental conditions of the iron bearing minerals flotation

During the flotation concentration experiments of the Eskişehir-Mihalıççık scandium ore, it was observed that the attrition scrubbed ore did not show any observable respond to the flotation method. In this context, it was seen that the obtained froth as the result of the flotation experiments generally contained slimes and also the desired minerals to be floated were not observed in the obtained froth. As it was expected prior to the experiments, it was not possible to obtain a flotation concentrate due to the finer

particle size and clay content of the ore and also it was observed once more that the flotation concentration of these types of ores was problematic. On the other hand, the present study also confirmed that the results of the previous flotation studies on these types of clayey ores and flotation concentration of these ores were challenging. In addition to these, there was not seen any considerable difference between froth product and tailings. In this context, stable froth could not be obtained by the applied flotation concentration method due to the slimes problem. As the result of the flotation concentration experiments, it was observed that the attrition scrubbed feed material could not be enriched by the flotation concentration method under the present conditions. Moreover, attrition scrubbed Eskişehir-Mihalıççık scandium ore did not respond positively to the applied flotation concentration method. Thereby, a pre-concentrate that was enriched in scandium could not be obtained as the result of the applied flotation concentration experiments. Consequently, it was seen that the flotation concentration method could not achieve its goal. In this case, it was also thought that the flotation concentration method was not to an alternative to the pre-concentration process of the enhanced gravity separation of Eskişehir-Mihalıççık Scandium ore by using falcon concentrator.

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

Within the scope of this research, the possibility of obtaining a scandium (Sc) pre-concentrate from Eskişehir-Mihalıççık scandium bearing clayey ore was investigated through mineral processing methods namely attrition scrubbing, enhanced gravity separation by using falcon concentrator, magnetic separation, and flotation concentration. The main objective of the study was obtaining a pre-concentrate enriched in scandium at the highest possible extent by the removal of the gangue minerals including calcite, dolomite, and quartz.

In the context of the performed characterization studies, mineralogical analysis results indicated that the major Sc-bearing minerals are montmorillonite (Al, Mg containing, smectite group clay mineral), nontronite (iron rich, smectite group clay mineral), and also to lesser extent Sc-bearing diopside (Ca, Fe, Mg silicate mineral) and pyroxene (so-called pyroxenite, a rock-forming silicate mineral) . In addition to that, the major impurity minerals are calcite (CaCO_3), dolomite ($\text{CaMg}(\text{CO}_3)_2$), and quartz (SiO_2).

The ore was relatively enriched in scandium content, comparing with the crustal abundance of Earth's crust, as 105 ppm by mainly concentrating in the clay minerals such as montmorillonite and nontronite. According to the chemical analysis results of the ore, relatively higher Fe content of the ore, as 8.01 %, a considerable amount, and regardless of its grade, iron and also the aluminum were not seen as the main impurities due to the similar behaviors of them with scandium during the pre-concentration studies. Based on the occurrence of calcite, dolomite, and quartz, which have relatively lower scandium content, the main impurity elements were determined as Ca, Mg, and Si. Related with this, Ca, Mg, and Si grades of the ore were 4.44 %, 5.16 %, and 24.45 %, respectively. Due to higher reagent consumptions and problematic structure of

these elements during the subsequent hydrometallurgical processes, removal of them prior to these subsequent processes was seen to be crucial. In this context, higher impurity content of the ore appeared to be a challenge during the pre-concentration studies.

In the context of the pre-concentration studies of Eskişehir-Mihalıççık scandium ore by using mineral processing methods, two major challenges were confronted. First of all, due to the finer particle size distribution and the clay mineral content of the ore, the conventional mineral processing methods were not applicable. On the other hand, concentration of scandium in the relatively finer particle size fractions namely in clay minerals as in the dispersed state, made the beneficiation of this valuable element challenging.

For the solution of the problems caused by the clay minerals contained in the ore, it was proven that the attrition scrubbing was efficient. In order to cope with the problems occurring due to the clay mineral content and finer particle size distribution of the ore, attrition scrubbing played an important role and it was observed that the attrition scrubbing was seen to be an applicable method for this ore. Apart from that, the enhanced gravity separation by using falcon concentrator was performed to obtain a pre-concentrate enriched in scandium content even though scandium was concentrated in finer particle size fractions. So, the challenging structure with regard to the concentration of scandium in finer particle sizes was overcome by using falcon concentrator.

As it was mentioned previously in detail, performed pre-concentration methods and their objectives are as followings;

- Attrition scrubbing was performed for obtaining a pre-concentrate enriched in scandium content by the removal of impurities such as Ca, Mg, and Si. Briefly, attrition scrubbing was used for the dispersion and liberation of the clay minerals concentrated in the relatively coarser fractions.

- Enhanced gravity separation by using falcon concentrator was performed on the previously obtained pre-concentrate of attrition scrubbing in order to provide further pre-concentration and to enrich scandium concentrated in finer size fractions. Within the scope of the falcon concentrator experiments, relatively lower scandium containing impurities were removed by the help of the centrifugal force and also a relatively enriched scandium pre-concentrate was obtained.
- As an alternative method to the enhanced gravity separation by using falcon concentrator, magnetic separation experiments were carried out for the further concentration of the pre-concentrate obtained from the attrition scrubbing. In the context of the magnetic separation experiments, the main objective was the enrichment of the Sc-bearing weakly paramagnetic and paramagnetic minerals in the magnetic concentrate fraction.
- As the second alternative method to enhanced gravity and magnetic separation, flotation concentration of the previously attrition scrubbed ore was performed. The main objective of the flotation concentration experiments were the removal of the calcite, dolomite, and quartz minerals in the tailings, while obtaining a pre-concentrate enriched in scandium from the concentrate fraction.

As the result of the attrition scrubbing experiments carried out under the previously mentioned optimum conditions, feed that initially contains 105 ppm Sc., was enriched to 120 ppm at 85.06 % scandium recovery. . In addition to these, 42.93 % of Ca, 30.88 % of Mg, and 33.73 % of Si were removed in the oversize material namely tailings fraction. As the result of the conducted optimization study, it was seen that the removal of impurities such as Ca, Mg, and Si was possible by performing attrition scrubbing. In conjunction with this, rejection of gangue minerals that would cause negative

impacts during the subsequent hydrometallurgical treatment of this type of the ore at the highest possible extent, was achieved.

Enhanced gravity separation by using falcon concentrator was performed to the pre-concentrate of the attrition scrubbing experiments. In this context, pre-concentrate of the attrition scrubbing experiments that initially contains 120 ppm Sc, was enriched to 138 ppm at 90.17 % scandium recovery. In terms of the impurity removal, 50.24 % of Ca, 26.65 % of Mg, and 20.14 % Si were removed in the heavier fraction namely tailings. Based on these results, it was seen that the removal of impurities such as Ca, Mg, and Si was possible with the performed enhanced gravity separation by using falcon concentrator. In conjunction with this, rejection of the gangue minerals that would cause negative impacts during the subsequent hydrometallurgical treatment of this type of ore, was achieved at the highest possible extent.

Magnetic separation was performed to the pre-concentrate of the attrition scrubbing experiments and it was observed that the pre-concentrate could not be enriched by magnetic separation under the present conditions. In this context, a pre-concentrate that was enriched in scandium could not be obtained as the result of the applied magnetic separation experiments. So, it was seen that the magnetic separation process could not achieve its goal. In this case, it was also thought that the magnetic separation was not an alternative to the pre-concentration process of enhanced gravity separation by using falcon concentrator.

As it was observed in the magnetic separation experiments, pre-concentrate of the attrition scrubbing experiments could not be enriched by the flotation concentration due to the finer particle size distribution and the clay mineral content of the ore. This study also confirmed the results of the previous flotation studies on these types of clayey ores and showed that the flotation concentration of these ores was challenging. So, attrition scrubbed Eskişehir-Mihalıççık scandium ore did not respond positively to the applied flotation concentration method. Thereby, a pre-concentrate that was enriched in scandium was not able to obtained as the result of the applied flotation

concentration experiments. Consequently, it was seen that the flotation concentration method could not achieve its goal. In this case, it was also thought that the flotation concentration method was not an alternative pre-concentration process to the enhanced gravity separation of Eskişehir-Mihalıççık Scandium ore by using falcon concentrator.

Considering all of the results obtained from the pre-concentration studies, proposed pre-concentration flowsheet for the Eskişehir-Mihalıççık Scandium ore is given in Figure 6.1.

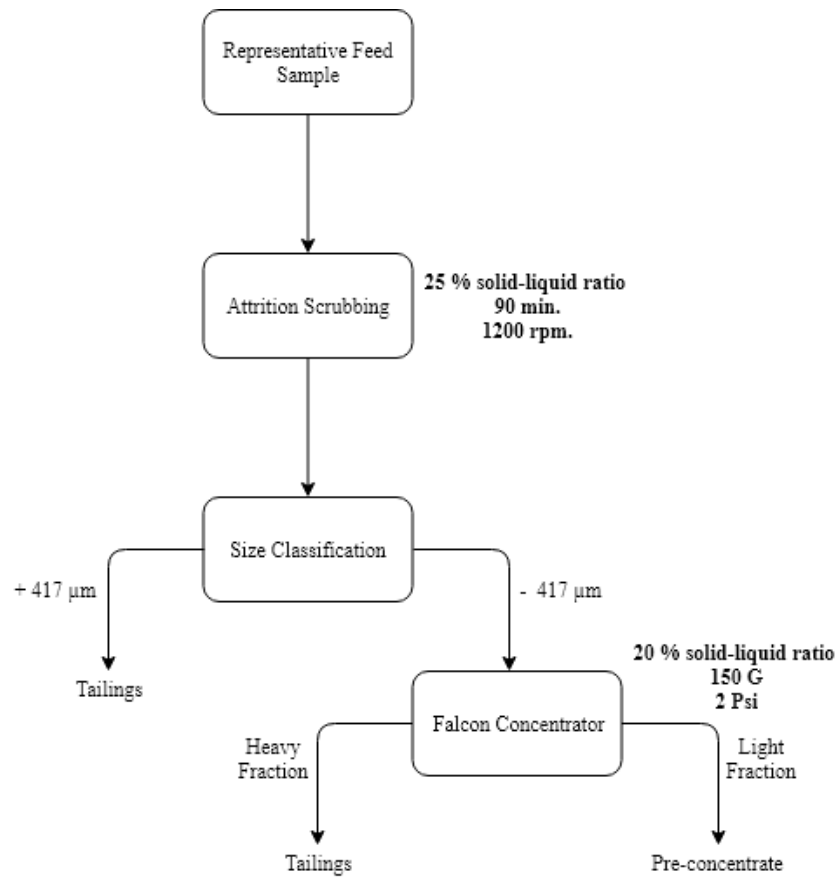


Figure 6.1 Proposed flowsheet for the pre-concentration of the Eskişehir-Mihalıççık Scandium ore

Based on the obtained results and inferences, it was considered that the applied pre-concentration study could facilitate the subsequent hydrometallurgical treatment of the ore by reducing the reagent consumptions and the process bottlenecks. As it was indicated, the present pre-concentration study performed on Eskişehir-Mihalıççık Scandium ore is unique in terms of filling the gap in the literature on the pre-concentration of scandium containing clayey ores. In this context, following recommendations are made for the future works:

1. Attrition scrubbing of the Eskişehir-Mihalıççık Scandium ore is promising but the process has already some deficits that should be improved. In this context, pre-leaching of the ore during the attrition scrubbing by the usage of organic acids can be performed for further removal of carbonaceous minerals such as calcite and dolomite. In this way, a further enriched scandium pre-concentrate can be obtained.
2. Another point that can be improved is related with the type of the falcon concentrator. In the present study, laboratory type falcon semi batch (SB) concentrator was used during the experiments. These type of concentrators are also used for the beneficiation of the relatively coarser materials. On the contrary, falcon ultra-fine (UF) concentrators were specifically designed for the beneficiation of minerals in the presence of ultra-fine particles. In this context, considering the finer particle size distribution of the ore, the process efficiency of the enhanced gravity separation can be increased by using Falcon UF type concentrator.
3. As it was previously mentioned, a pre-concentrate enriched in scandium could not be obtained by using magnetic separation. It was thought that this situation may be due to the fact that the provided magnetic field intensity was low. Therefore, it may be useful to subject the pre-concentrate of attrition scrubbing to the high gradient magnetic separator, namely Slon type, in terms of increasing the recovery of the scandium. Following these,

in order to increase the grade of the magnetic concentrate, further processing of it by using falcon concentrator may be useful.

4. Based on the observations during the flotation tests, it was seen that the concentrate could not be obtained because of the presence of excess slimes. Therefore, size classification of the attrition scrubbed material by discarding the ultra-fine size particles may be beneficial in terms of the flotation concentrate efficiency. Following the removal of slimes, carbonate flotation can also be done in order to remove the carbonaceous minerals. Furthermore, a systematically conducted flotation process for the removal of quartz by depressing the Sc-bearing clay minerals, diopside, and pyroxene, can be useful in terms of both the grade and the recovery of the obtained concentrate. At the end of the flotation experiments, slime fraction and the flotation tailings can be combined and evaluated together for the metallurgical results and calculations. As a consequence of this, a pre-concentrate that will be enriched in scandium content may be produced by removing the impurity minerals.

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APPENDICES

APPENDIX A

X-RAY DIFFRACTOGRAMS OF DRILL CORE SAMPLES

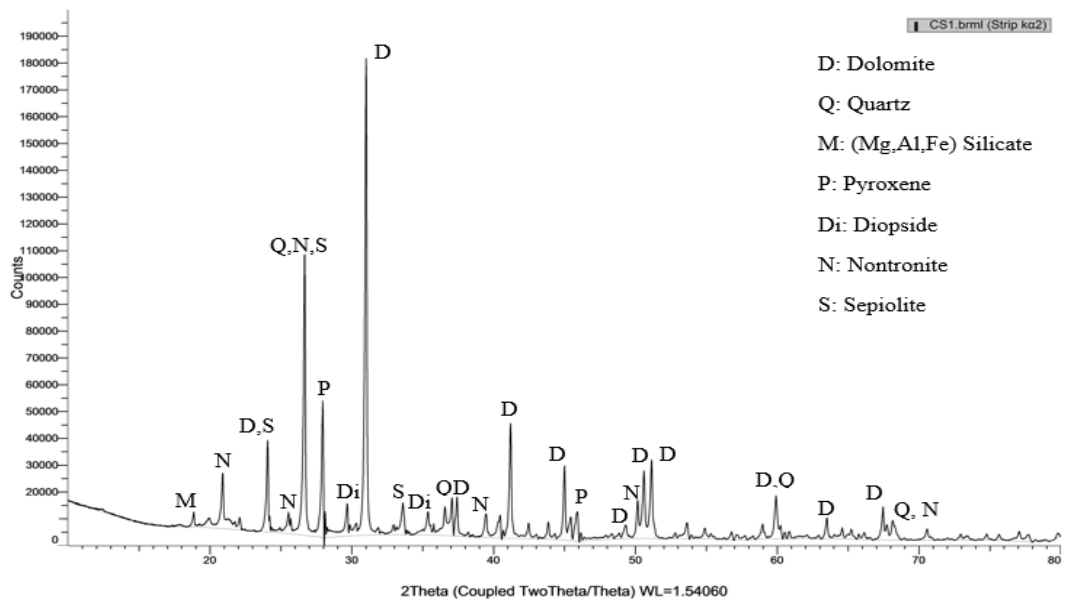


Figure A. 1 X-ray diffractogram of drill core sample 1

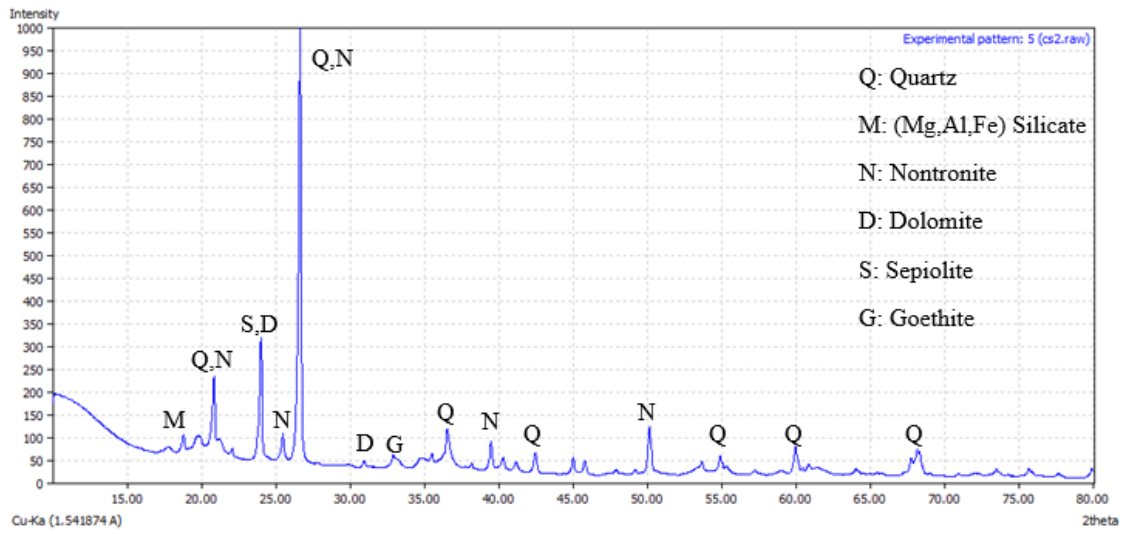


Figure A. 2 X-ray diffractogram of drill core sample 2

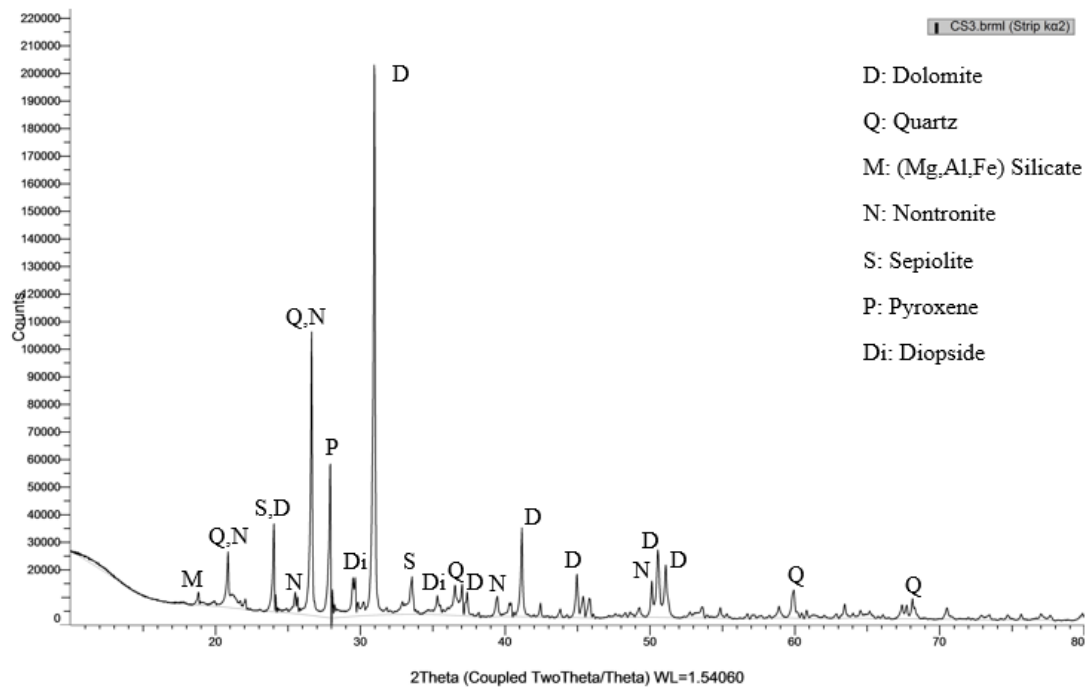


Figure A. 3 X-ray diffractogram of drill core sample 3

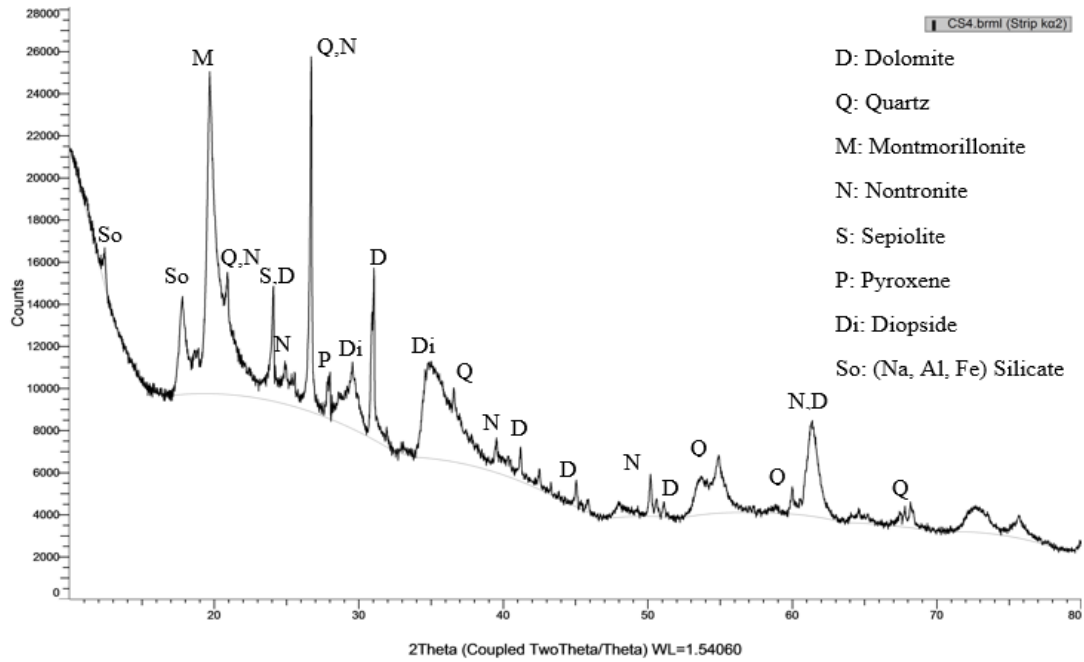


Figure A. 4 X-ray diffractogram of drill core sample 4

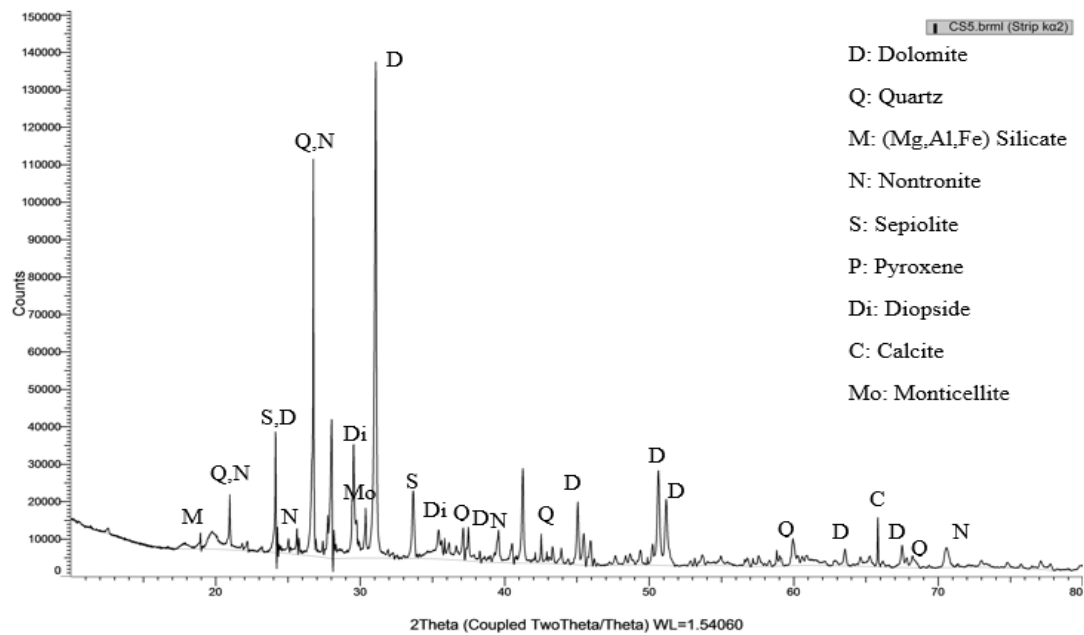


Figure A. 5 X-ray diffractogram of drill core sample 5

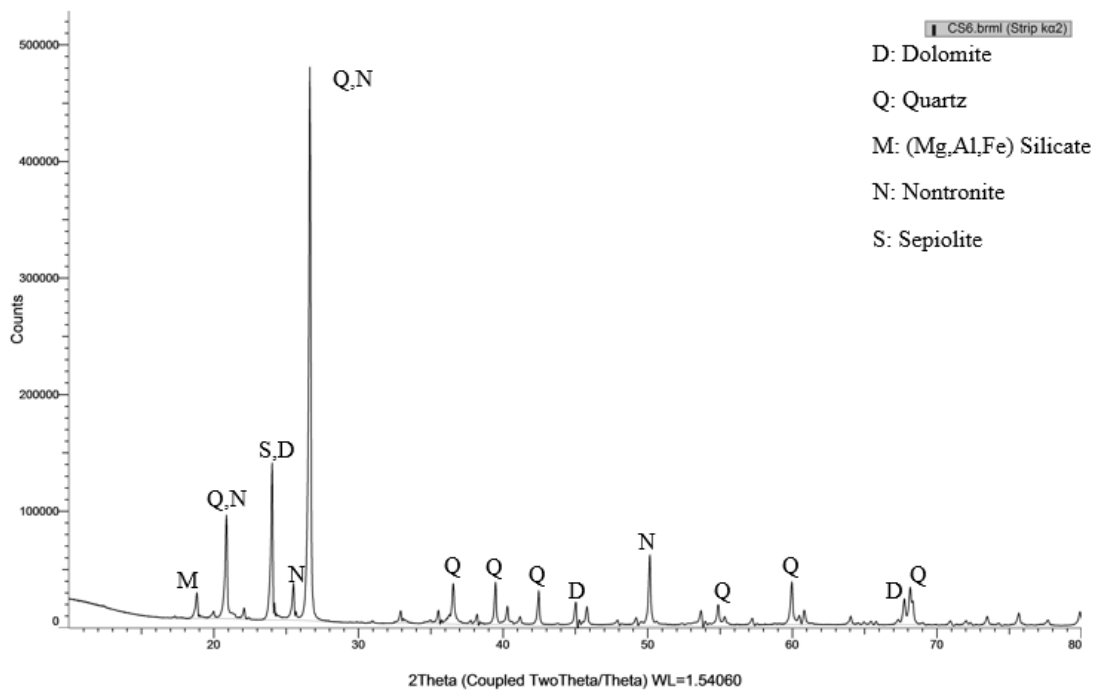


Figure A. 6 X-ray diffractogram of drill core sample 6

DETAILED CHEMICAL ANALYSIS RESULTS FOR THE DRILL
HOLE CORE SAMPLES

Table B 1 Elemental analysis results of drill hole core samples

Drill Core Sample No	Start (m)	End (m)	Sc (ppm)	Fe (%)	Al (%)	Ca (%)	Co (ppm)	Cr (ppm)	Mg (%)	Mn (ppm)	Ni (ppm)
<i>1</i>	1.00	16.40	90	6.07	2.53	7.79	90	1810	5.29	1095	574
<i>2</i>	16.40	19.40	58	3.84	1.57	13.65	62	1325	6.74	903	361
<i>3</i>	19.40	23.60	87	5.31	2.32	6.76	119	2930	4.54	1160	634
<i>4</i>	27.00	35.00	127	10.90	2.62	0.72	169	4590	1.12	2260	1070
<i>5</i>	37.00	53.70	76	6.01	1.95	8.46	89	2250	5.21	1470	513
<i>6</i>	58.00	67.00	44	4.09	1.11	8.25	61	2960	5.43	920	434

APPENDIX C

X-RAY DIFFRACTOGRAMS OF THE SIZE FRACTIONS OF THE ORE SAMPLE

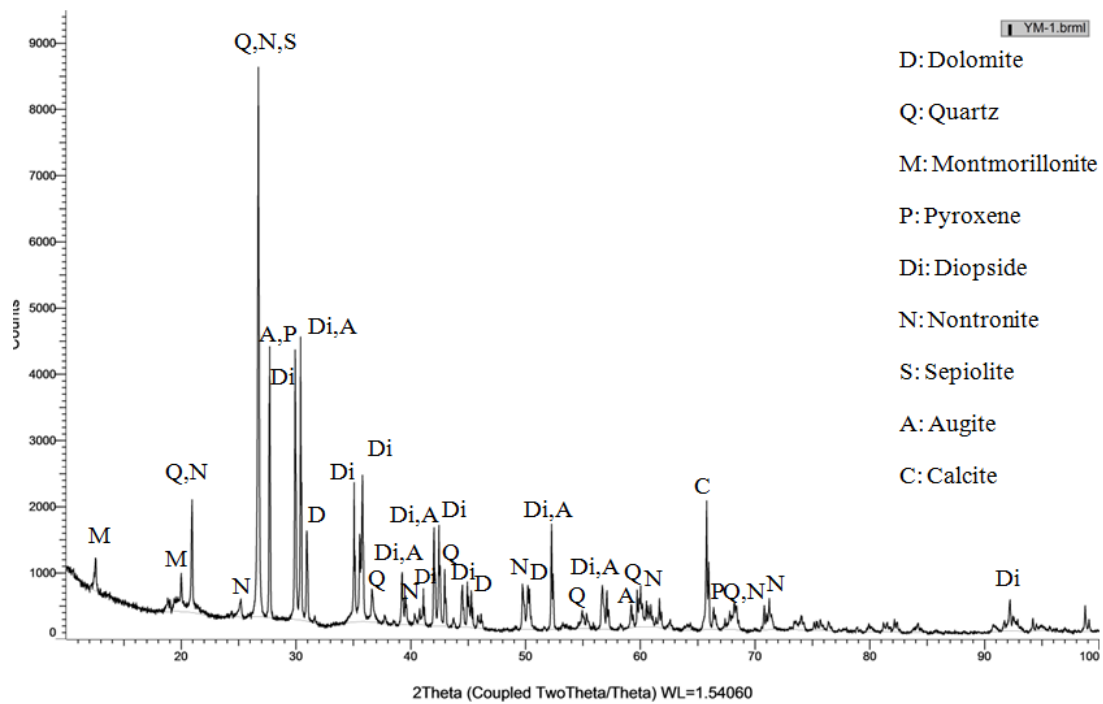


Figure C. 1 X-ray diffractogram of + 9.53 mm fraction

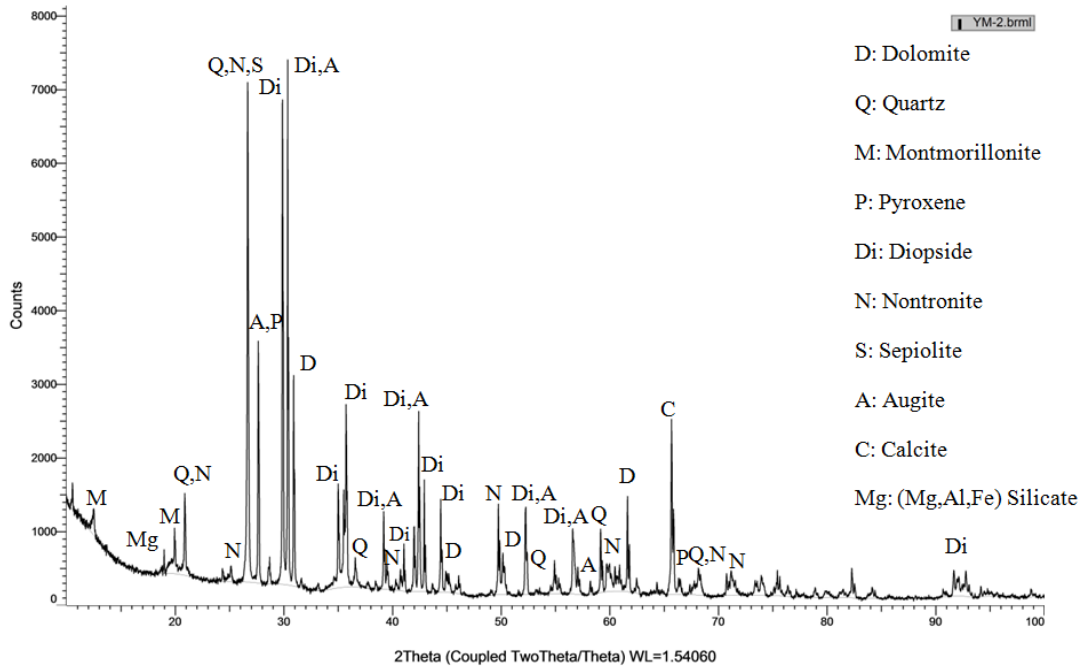


Figure C. 2 X-ray diffractogram of - 9.53 + 6.35 mm fraction

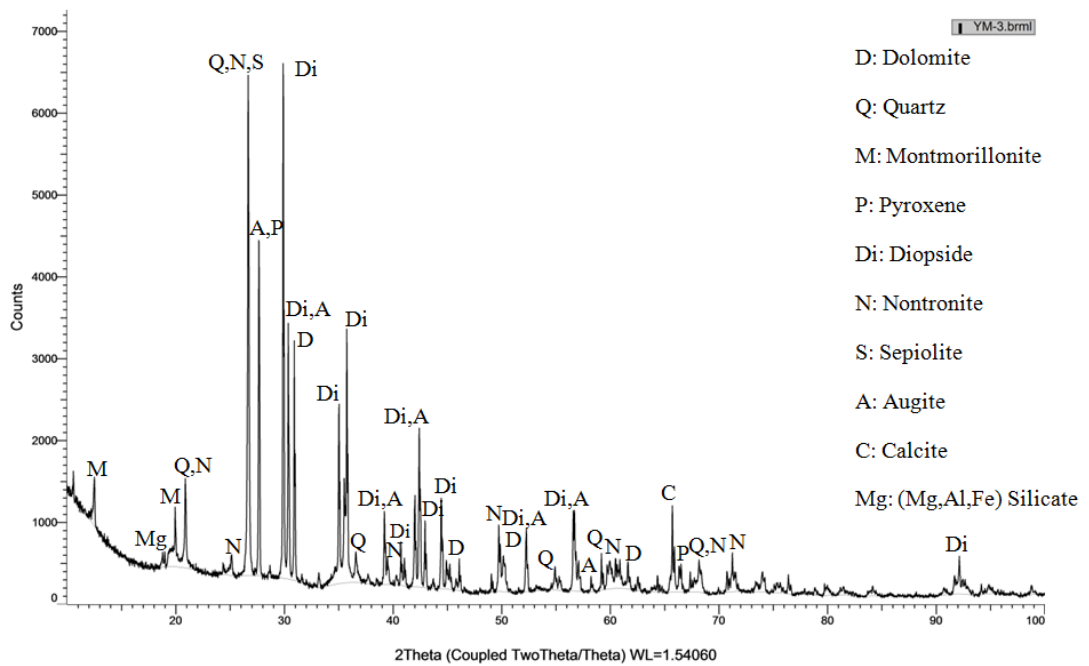


Figure C. 3 X-ray diffractogram of - 6.35 + 3.35 mm fraction

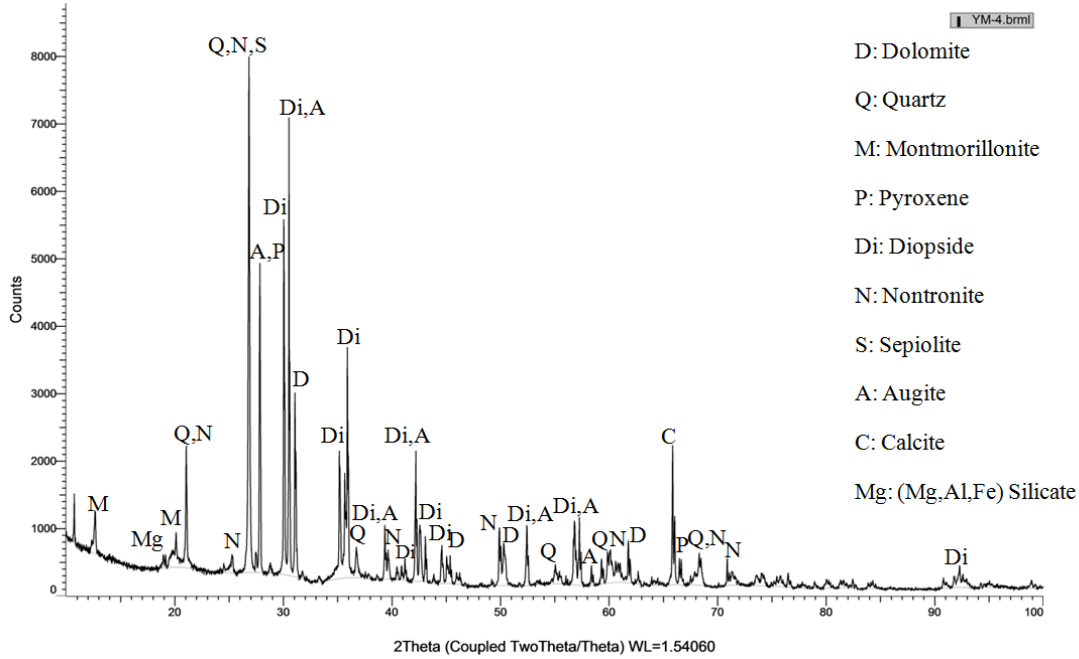


Figure C. 4 X-ray diffractogram of - 3.35 + 2.14 mm fraction

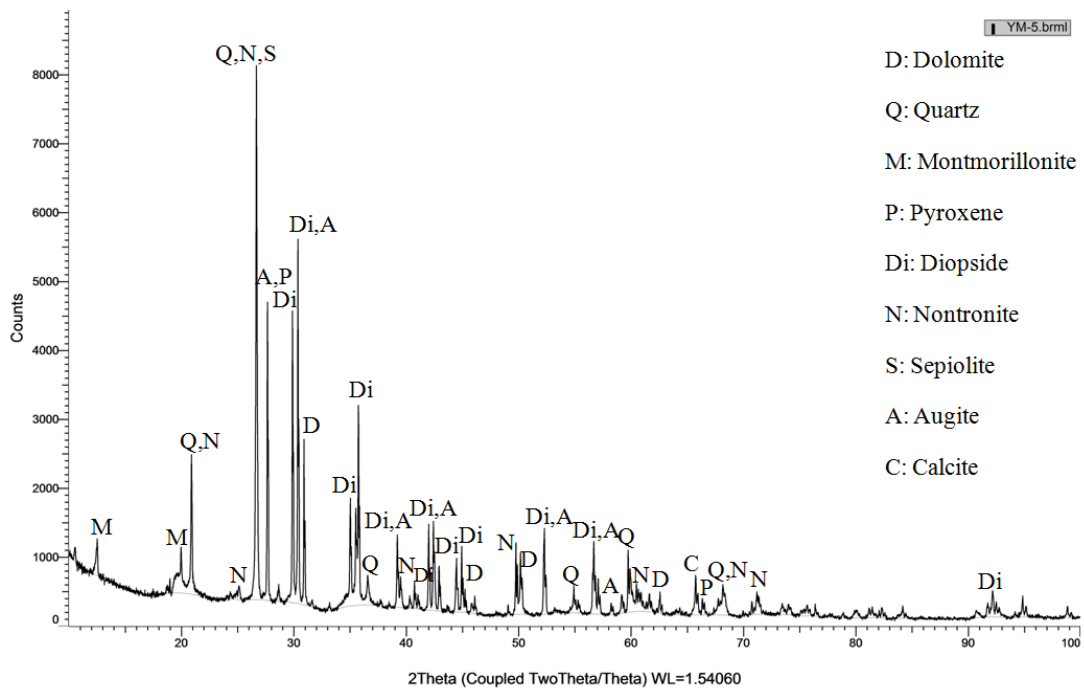


Figure C. 5 X-ray diffractogram of - 2.14 + 1.168 mm fraction

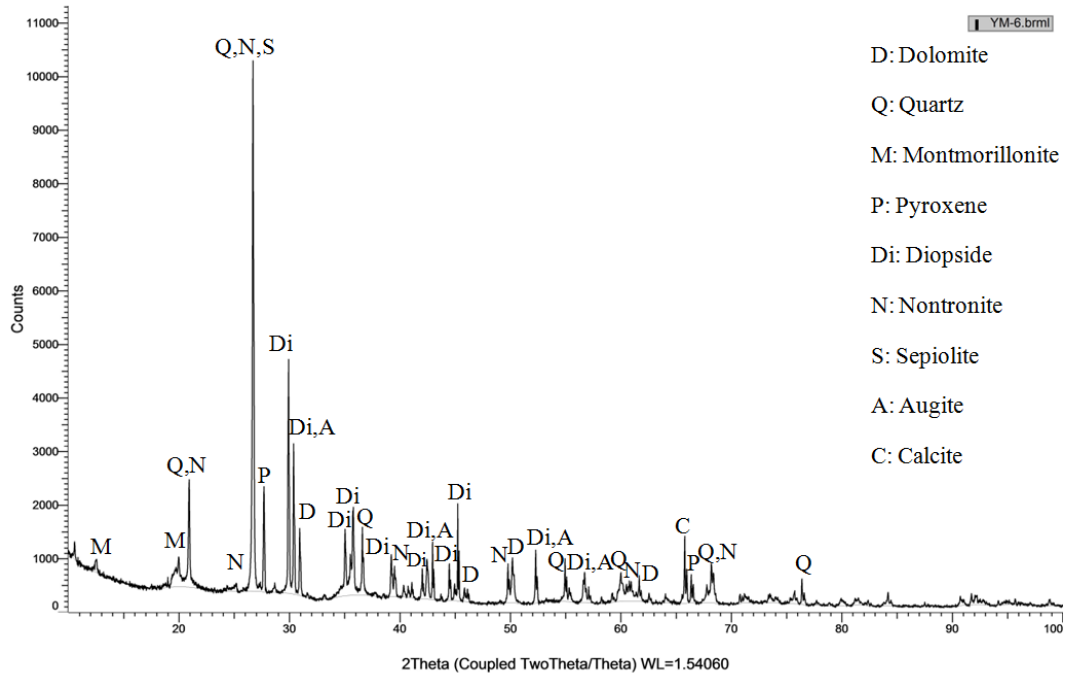


Figure C. 6 X-ray diffractogram of - 1.168 + 0.850 mm fraction

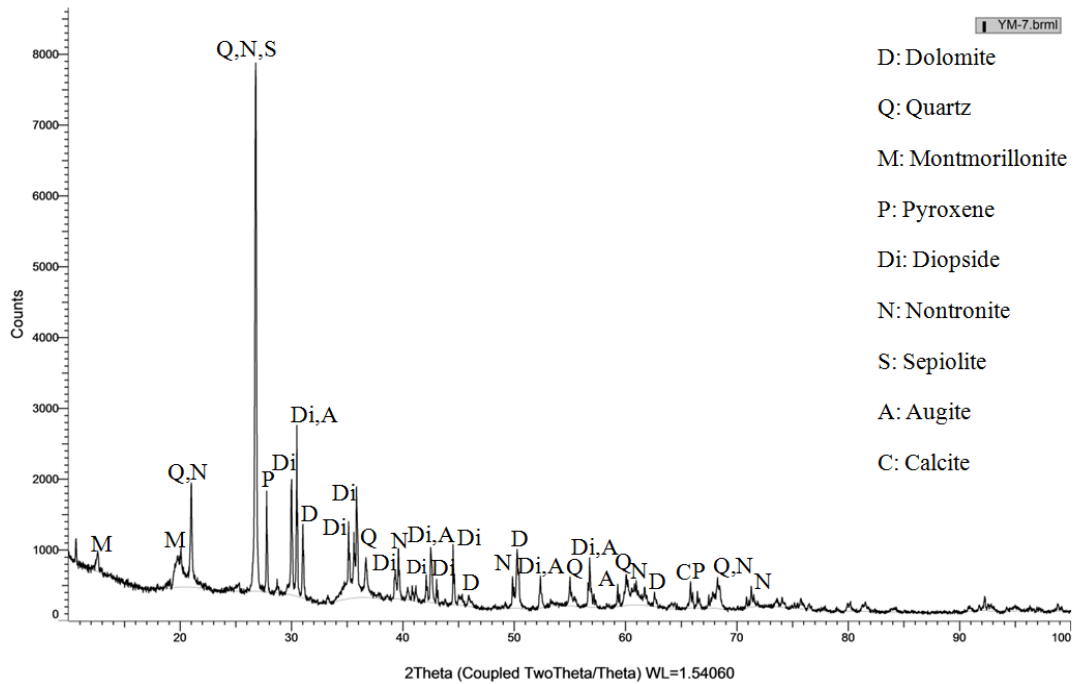


Figure C. 7 X-ray diffractogram of - 0.850 + 0.600 mm fraction

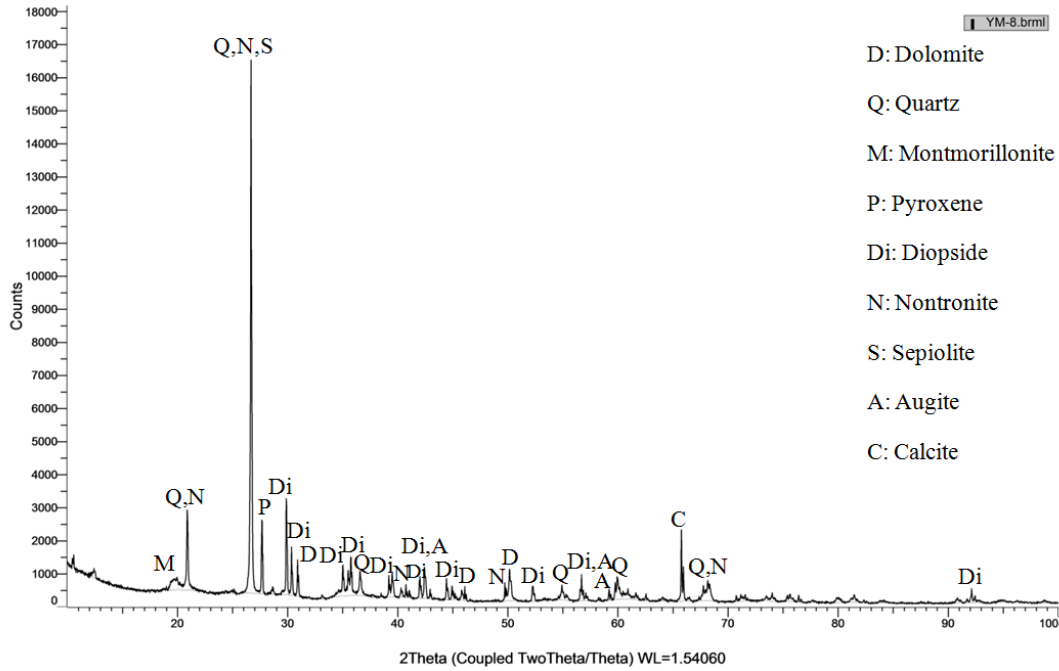


Figure C. 8 X-ray diffractogram of - 0.600 + 0.417 mm fraction

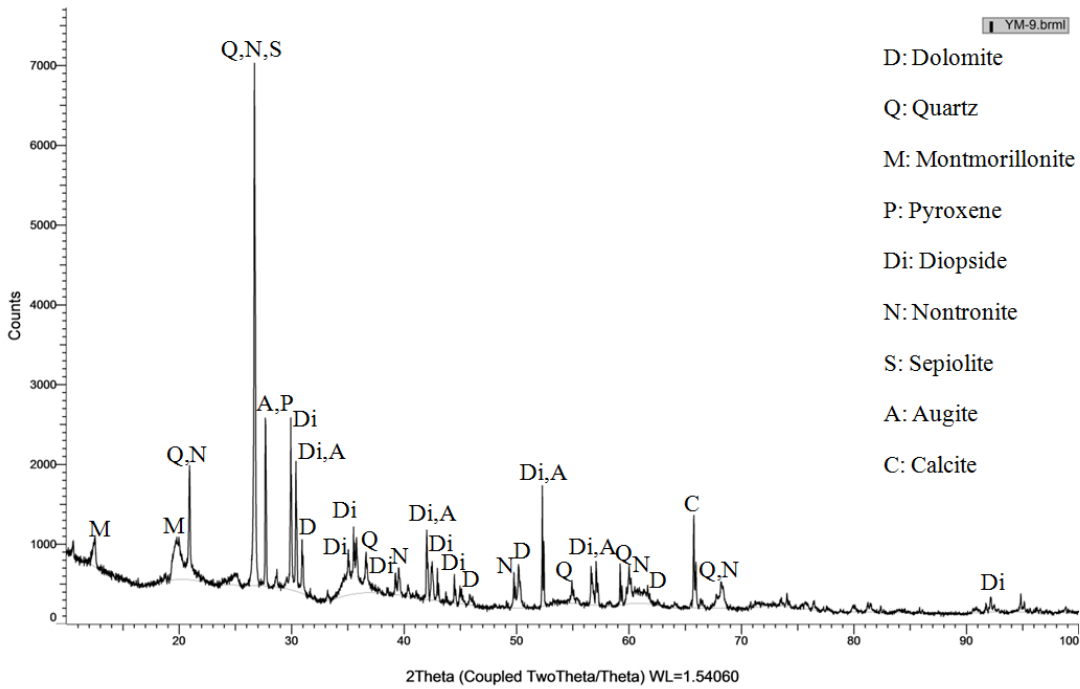


Figure C. 9 X-ray diffractogram of - 0.417 + 0.210 mm fraction

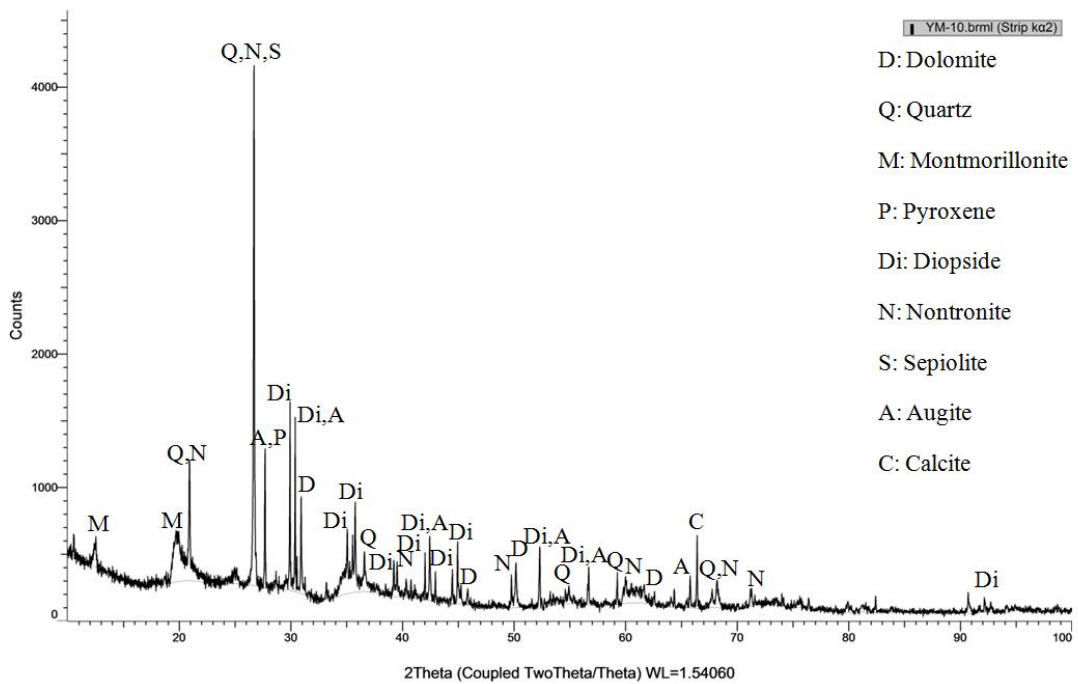


Figure C. 10 X-ray diffractogram of - 0.210 + 0.106 mm fraction

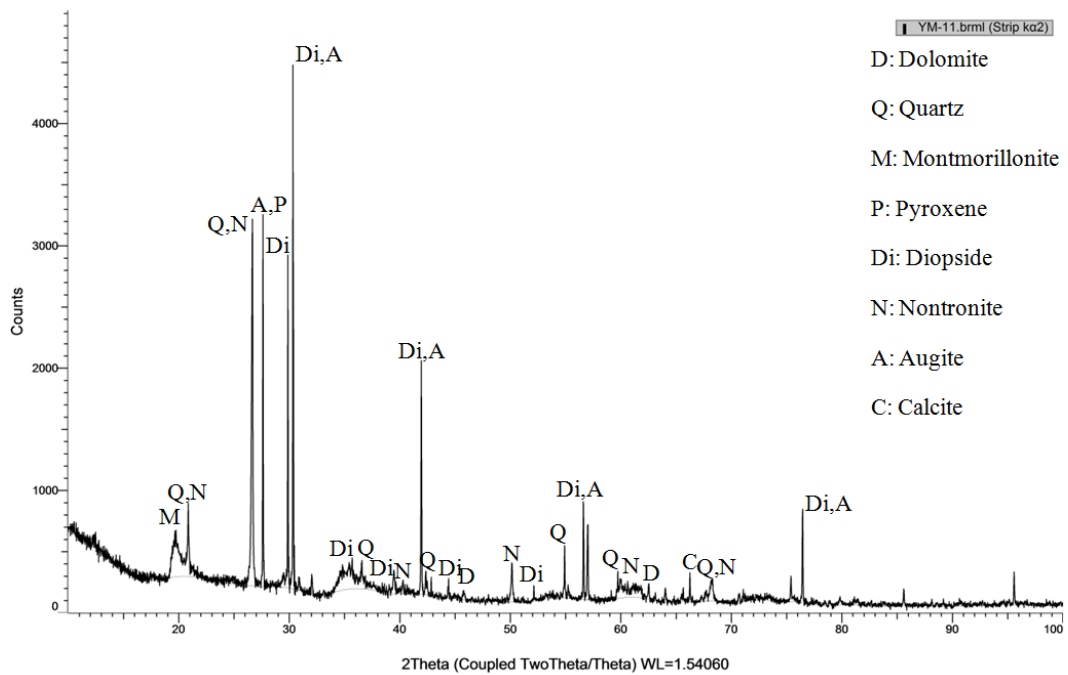


Figure C. 11 X-ray diffractogram of - 0.106 + 0.071 mm fraction

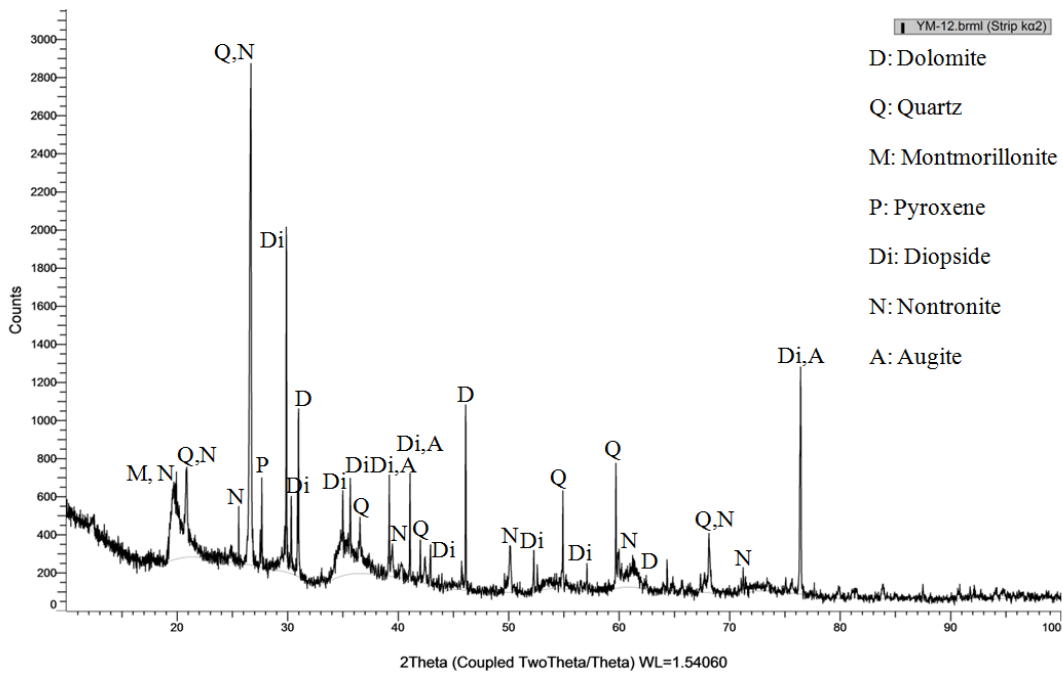


Figure C. 12 X-ray diffractogram of - 0.071 + 0.053 mm fraction

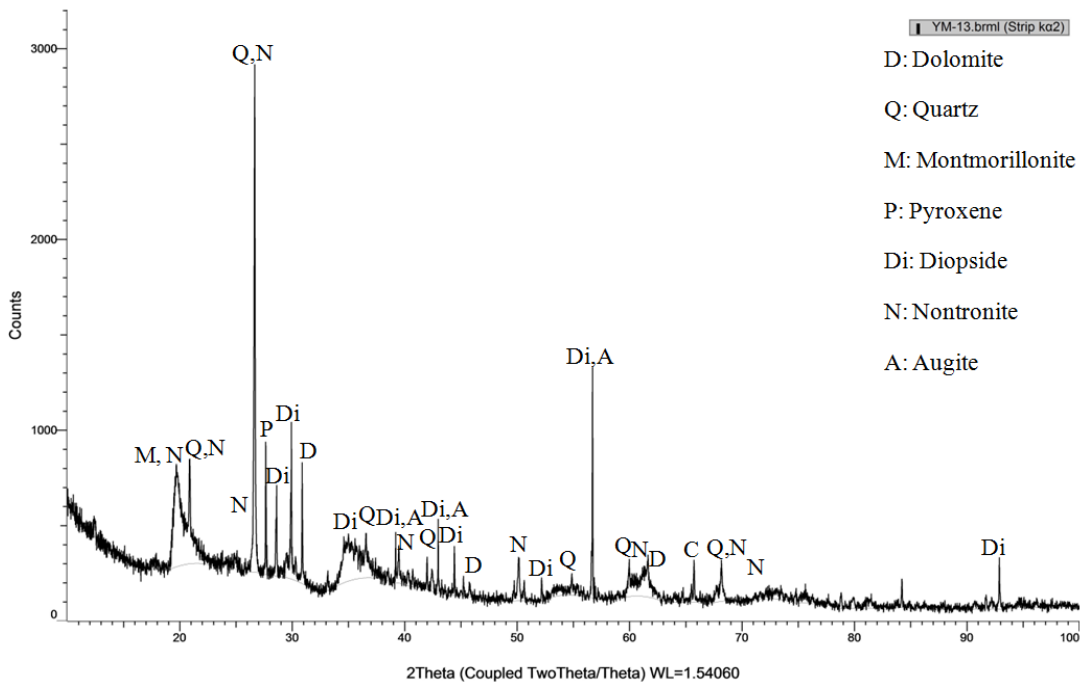


Figure C. 13 X-ray diffractogram of - 0.053 + 0.038 mm fraction

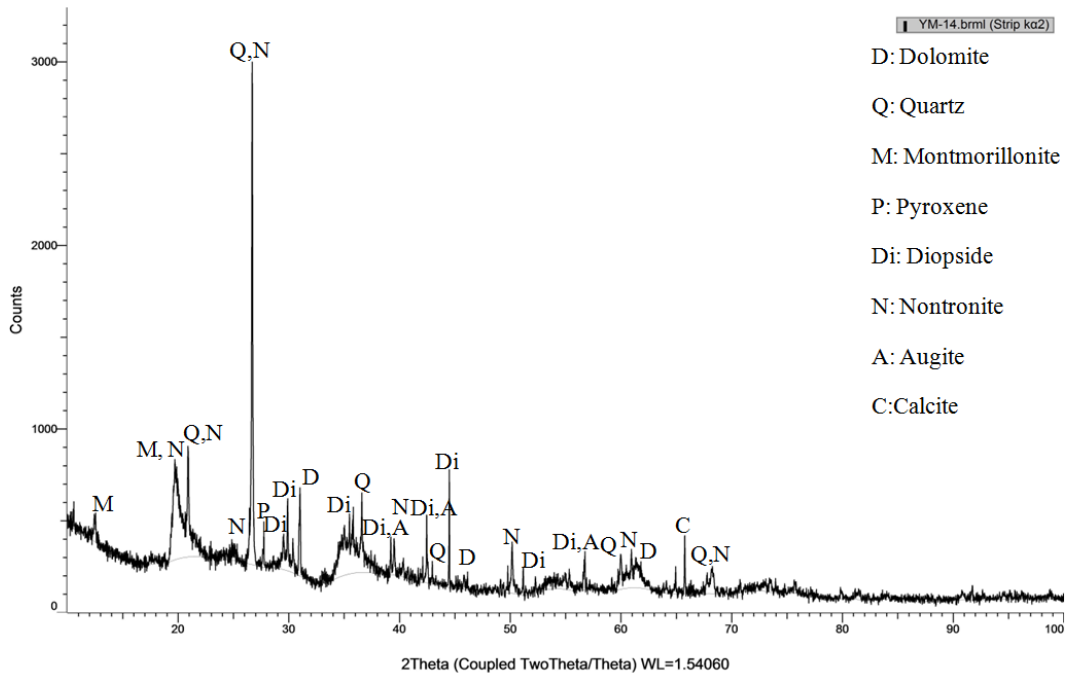


Figure C. 14 X-ray diffractogram of - 0.038 + 0.020 mm fraction

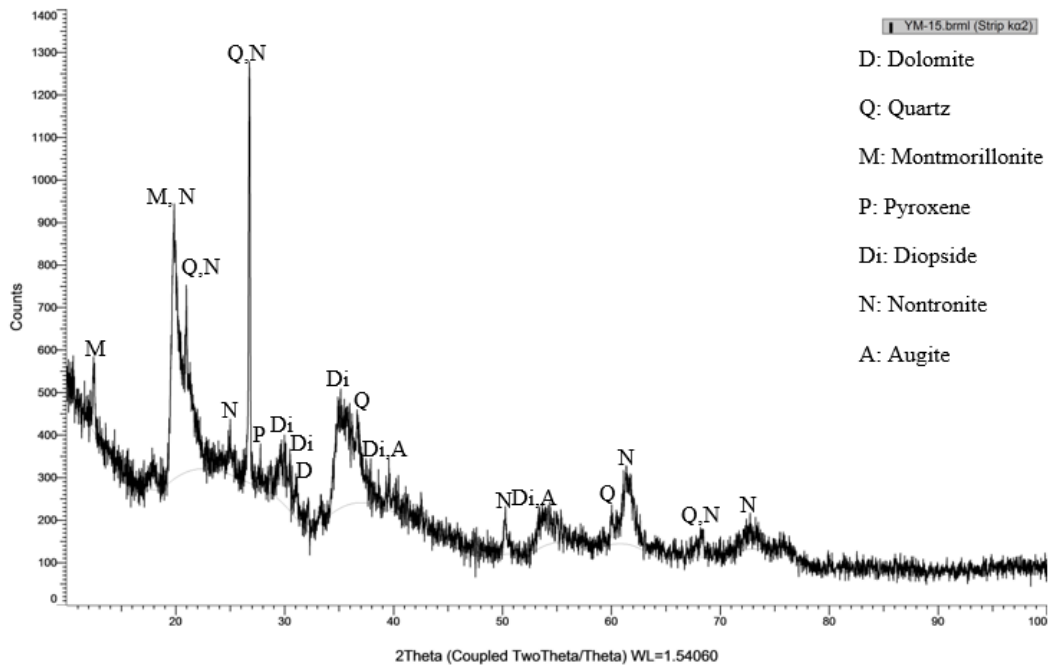


Figure C. 15 X-ray diffractogram of - 0.020 mm fraction

DETAILED METALLURGICAL RESULTS OF THE ATTRITION
SCRUBBING EXPERIMENTS

Table D. 1 Detailed metallurgical results of the attrition scrubbing experiments carried out at base levels

Exp Code	Fraction (μm)	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
17	+417 μm	26.42	2.01	14.00	6.05	48.92	4.42	14.53	6.41	32.39	67	16.31	24.21	26.19
	-417 μm	73.58	4.43	86.00	2.27	51.08	9.33	85.47	4.80	67.61	123	83.69	24.50	73.81
	Total	100.00	3.79	100.00	3.27	100.00	8.03	100.00	5.23	100.00	108	100.00	24.42	100.00
18	+417 μm	27.51	2.04	15.49	9.36	48.92	4.58	15.85	7.55	33.67	74	18.91	26.54	28.75
	-417 μm	72.49	4.23	84.51	3.71	51.08	9.22	84.15	5.64	66.33	120	81.09	24.96	71.25
	Total	100.00	3.63	100.00	5.26	100.00	7.94	100.00	6.16	100.00	108	100.00	25.40	100.00
19	+417 μm	25.89	2.06	15.02	10.21	48.19	4.85	16.09	7.66	32.65	90	20.97	26.91	27.46
	-417 μm	74.11	4.06	84.98	3.84	51.81	8.83	83.91	5.53	67.35	118	79.03	24.84	72.54
	Total	100.00	3.54	100.00	5.49	100.00	7.80	100.00	6.08	100.00	111	100.00	25.38	100.00

Metallurgically calculated

Table D. 2 Detailed metallurgical results of the attrition scrubbing experiments

Exp Code	Fraction (μm)	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
1	+417 μm	31.58	2.09	18.32	8.08	53.63	4.43	17.85	6.80	37.94	71	20.95	29.56	33.75
	-417 μm	68.42	4.30	81.68	3.23	46.37	9.41	82.15	5.14	62.06	124	79.05	26.78	66.25
	Total	100.00	3.60	100.00	4.76	100.00	7.84	100.00	5.66	100.00	108	100.00	27.66	100.00
3	+417 μm	19.59	1.81	10.08	8.67	33.61	4.29	10.49	6.91	22.44	73	12.39	29.82	21.27
	-417 μm	80.41	3.94	89.92	4.17	66.39	8.93	89.51	5.82	77.56	126	87.61	26.89	78.73
	Total	100.00	3.53	100.00	5.06	100.00	8.02	100.00	6.04	100.00	115	100.00	27.47	100.00
5	+417 μm	24.99	2.01	14.02	9.29	44.38	4.21	13.32	7.32	30.12	73	16.21	29.39	26.98
	-417 μm	75.01	4.11	85.98	3.88	55.62	9.13	86.68	5.66	69.88	126	83.79	26.50	73.02
	Total	100.00	3.59	100.00	5.23	100.00	7.90	100.00	6.07	100.00	113	100.00	27.22	100.00
7	+417 μm	19.53	1.80	10.13	8.59	31.96	4.31	10.99	6.85	22.01	77	13.53	26.19	19.31
	-417 μm	80.47	3.89	89.87	4.44	68.04	8.49	89.01	5.89	77.99	120	86.47	26.55	80.69
	Total	100.00	3.48	100.00	5.25	100.00	7.67	100.00	6.08	100.00	112	100.00	26.48	100.00
9	+417 μm	31.16	2.29	18.35	8.94	58.87	4.60	17.49	7.39	39.98	78	21.28	27.45	31.99
	-417 μm	68.84	4.62	81.65	2.83	41.13	9.83	82.51	5.02	60.02	131	78.72	26.42	68.01
	Total	100.00	3.89	100.00	4.73	100.00	8.20	100.00	5.76	100.00	114	100.00	26.74	100.00
11	+417 μm	19.87	1.82	10.74	9.45	34.50	3.76	9.91	7.01	22.76	69	12.74	22.90	18.29
	-417 μm	80.13	3.75	89.26	4.45	65.50	8.47	90.09	5.90	77.24	117	87.26	25.36	81.71
	Total	100.00	3.36	100.00	5.44	100.00	7.53	100.00	6.12	100.00	108	100.00	24.87	100.00
13	+417 μm	25.04	1.95	13.04	9.75	47.43	4.66	13.74	6.87	31.16	77	15.86	25.04	26.29
	-417 μm	74.96	4.33	86.96	3.61	52.57	9.76	86.26	5.07	68.84	136	84.14	23.44	73.71
	Total	100.00	3.74	100.00	5.15	100.00	8.48	100.00	5.52	100.00	121	100.00	23.84	100.00
15	+417 μm	20.13	1.86	9.77	8.36	35.17	4.46	10.44	6.82	22.68	74	12.49	29.90	22.05
	-417 μm	79.87	4.34	90.23	3.89	64.83	9.65	89.56	5.87	77.32	131	87.51	26.64	77.95
	Total	100.00	3.84	100.00	4.79	100.00	8.60	100.00	6.06	100.00	119	100.00	27.29	100.00

Table D. 3 Detailed metallurgical results of the parallel attrition scrubbing experiments

Exp Code	Fraction (μm)	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
2	+417 μm	31.30	1.75	15.52	4.94	50.25	4.12	17.12	5.55	35.01	61	18.87	29.35	32.97
	-417 μm	68.70	4.34	84.48	2.23	49.75	9.09	82.88	4.70	64.99	119	81.13	27.18	67.03
	Total	100.00	3.53	100.00	3.08	100.00	7.53	100.00	4.96	100.00	101	100.00	27.86	100.00
4	+417 μm	19.62	1.60	8.83	5.20	30.15	4.01	10.26	5.53	20.31	57	10.83	26.21	22.23
	-417 μm	80.38	4.03	91.17	2.94	69.85	8.56	89.74	5.30	79.69	115	89.17	22.38	77.77
	Total	100.00	3.56	100.00	3.38	100.00	7.66	100.00	5.35	100.00	104	100.00	23.13	100.00
6	+417 μm	24.03	1.95	12.43	6.08	41.12	4.55	13.70	6.61	28.76	69	15.28	29.59	26.13
	-417 μm	75.97	4.35	87.57	2.75	58.88	9.07	86.30	5.18	71.24	121	84.72	26.47	73.87
	Total	100.00	3.77	100.00	3.55	100.00	7.98	100.00	5.52	100.00	109	100.00	27.22	100.00
8	+417 μm	19.43	1.85	9.41	6.16	36.93	4.21	9.90	6.45	23.52	65	11.54	28.32	21.54
	-417 μm	80.57	4.29	90.59	2.54	63.07	9.23	90.10	5.06	76.48	121	88.46	24.87	78.46
	Total	100.00	3.82	100.00	3.24	100.00	8.25	100.00	5.33	100.00	110	100.00	25.54	100.00
10	+417 μm	33.75	1.92	17.48	5.53	52.88	4.45	18.98	6.14	38.13	65	21.04	29.12	36.79
	-417 μm	66.25	4.61	82.52	2.51	47.12	9.67	81.02	5.08	61.87	124	78.96	25.49	63.21
	Total	100.00	3.70	100.00	3.53	100.00	7.91	100.00	5.44	100.00	104	100.00	26.72	100.00
12	+417 μm	21.48	1.83	10.41	5.49	37.78	4.76	12.24	5.97	24.61	62	12.38	28.80	25.34
	-417 μm	78.52	4.31	89.59	2.47	62.22	9.33	87.76	5.00	75.39	121	87.62	23.21	74.66
	Total	100.00	3.78	100.00	3.12	100.00	8.35	100.00	5.21	100.00	108	100.00	24.41	100.00
14	+417 μm	27.33	1.20	9.73	3.83	33.92	5.62	19.21	4.31	23.75	51	14.10	33.57	33.74
	-417 μm	72.67	4.20	90.27	2.81	66.08	8.89	80.79	5.20	76.25	116	85.90	24.79	66.26
	Total	100.00	3.38	100.00	3.09	100.00	7.99	100.00	4.96	100.00	98	100.00	27.19	100.00
16	+417 μm	18.47	1.88	9.56	5.90	30.32	4.34	10.52	6.29	21.19	66	11.67	23.03	18.17
	-417 μm	81.53	4.03	90.44	3.07	69.68	8.36	89.48	5.30	78.81	113	88.33	23.48	81.83
	Total	100.00	3.64	100.00	3.59	100.00	7.62	100.00	5.48	100.00	104	100.00	23.40	100.00

DETAILED METALLURGICAL RESULTS OF THE FALCON
CONCENTRATOR EXPERIMENTS

Table E. 1 Detailed metallurgical results of the falcon concentrator experiments carried at 2 psi. fluidization water pressure

Exp. Code	Fraction	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
1	Light	91.13	4.51	64.71	2.61	72.29	9.32	94.92	4.81	87.53	131	94.30	24.45	90.58
	Heavy	8.87	2.46	35.29	10.30	27.71	5.12	5.08	7.04	12.47	81	5.70	26.13	9.42
	Total	100.00	4.33	100.00	3.30	100.00	8.95	100.00	5.01	100.00	126	100.00	24.60	100.00
2	Light	80.24	4.98	88.57	2.62	49.76	11.21	89.27	5.24	73.35	138	90.18	27.43	79.86
	Heavy	19.76	2.61	11.43	10.74	50.24	5.47	10.73	7.73	26.65	61	9.82	28.08	20.14
	Total	100.00	4.51	100.00	4.22	100.00	10.08	100.00	5.73	100.00	123	100.00	27.56	100.00
3	Light	72.48	4.61	62.54	1.95	39.86	9.54	81.30	4.46	63.74	131	78.99	25.16	71.57
	Heavy	27.52	2.76	37.46	7.74	60.14	5.78	18.70	6.67	36.26	92	21.01	26.32	28.43
	Total	100.00	4.10	100.00	3.54	100.00	8.50	100.00	5.07	100.00	120	100.00	25.48	100.00
4	Light	74.29	4.65	63.30	2.01	42.99	9.70	83.27	4.52	66.55	133	83.00	23.56	66.90
	Heavy	25.71	2.69	36.70	7.72	57.01	5.63	16.73	6.56	33.45	79	17.00	33.68	33.10
	Total	100.00	4.15	100.00	3.48	100.00	8.65	100.00	5.04	100.00	119	100.00	26.16	100.00

Metallurgically calculated

Table E. 2 Detailed metallurgical results of the falcon concentrator experiments carried at 6 psi. fluidization water pressure

Exp. Code	Fraction	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
5	Light	99.30	4.29	69.07	3.40	97.32	8.80	99.59	5.06	98.96	121	99.58	23.79	99.26
	Heavy	0.70	2.16	30.93	13.22	2.68	5.07	0.41	7.51	1.04	73	0.42	24.94	0.74
	Total	100.00	4.28	100.00	3.47	100.00	8.77	100.00	5.08	100.00	121	100.00	23.80	100.00
6	Light	84.18	4.50	66.34	2.59	58.01	9.37	91.87	4.77	78.06	127	90.46	23.96	83.57
	Heavy	15.82	2.28	33.66	9.99	41.99	4.41	8.13	7.13	21.94	71	9.54	25.07	16.43
	Total	100.00	4.15	100.00	3.76	100.00	8.58	100.00	5.14	100.00	118	100.00	24.14	100.00
7	Light	85.26	4.54	66.82	2.41	58.62	9.35	92.59	4.56	78.95	131	91.10	23.85	84.66
	Heavy	14.74	2.26	33.18	9.86	41.38	4.33	7.41	7.04	21.05	74	8.90	25.01	15.34
	Total	100.00	4.20	100.00	3.51	100.00	8.61	100.00	4.93	100.00	123	100.00	24.02	100.00
8	Light	82.29	4.50	63.83	2.72	56.85	9.41	89.38	4.71	75.59	128	88.38	23.89	81.63
	Heavy	17.71	2.55	36.17	9.61	43.15	5.20	10.62	7.06	24.41	78	11.62	24.99	18.37
	Total	100.00	4.15	100.00	3.94	100.00	8.67	100.00	5.12	100.00	119	100.00	24.08	100.00

Metallurgically calculated

Table E. 3 Detailed metallurgical results of the falcon concentrator experiments carried at 10 psi. fluidization water pressure

Exp. Code	Fraction	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
9	Light	97.55	4.22	66.14	3.28	91.60	8.77	98.70	5.04	96.43	120	98.45	20.64	98.06
	Heavy	2.45	2.16	33.86	11.99	8.40	4.61	1.30	7.43	3.57	75	1.55	16.31	1.94
	Total	100.00	4.17	100.00	3.49	100.00	8.67	100.00	5.09	100.00	119	100.00	20.53	100.00
10	Light	95.95	4.30	66.92	3.25	85.95	8.97	98.06	5.07	93.99	122	97.38	25.35	96.21
	Heavy	4.05	2.12	33.08	12.60	14.05	4.22	1.94	7.70	6.01	78	2.62	23.70	3.79
	Total	100.00	4.21	100.00	3.63	100.00	8.78	100.00	5.18	100.00	120	100.00	25.29	100.00
11	Light	95.41	4.29	66.49	3.13	84.97	8.89	97.69	4.98	93.34	123	97.13	26.67	95.52
	Heavy	4.59	2.16	33.51	11.50	15.03	4.37	2.31	7.38	6.66	75	2.87	25.97	4.48
	Total	100.00	4.19	100.00	3.51	100.00	8.68	100.00	5.09	100.00	120	100.00	26.64	100.00
12	Light	96.73	4.29	66.50	3.30	89.34	8.89	98.29	5.09	95.37	123	97.97	25.57	96.59
	Heavy	3.27	2.16	33.50	11.67	10.66	4.57	1.71	7.32	4.63	75	2.03	26.73	3.41
	Total	100.00	4.22	100.00	3.57	100.00	8.74	100.00	5.16	100.00	121	100.00	25.61	100.00

Metallurgically calculated

DETAILED METALLURGICAL RESULTS OF THE MAGNETIC
SEPARATION EXPERIMENTS

Table F. 1 Detailed metallurgical results of the magnetic separation experiments

Exp Code	Fraction (μm)	Weight (%)	Al (%)	Al Rec. (%)	Ca (%)	Ca Rec. (%)	Fe (%)	Fe Rec. (%)	Mg (%)	Mg Rec. (%)	Sc (ppm)	Sc Rec. (%)	Si (%)	Si Rec. (%)
1	Mag.	24.62	3.31	20.31	5.01	31.01	9.92	26.95	5.83	26.91	110	21.91	23.77	23.86
	Non. Mag.	75.38	4.24	79.69	3.64	68.99	8.78	73.05	5.17	73.09	128	78.09	24.77	76.14
	Total	100.00	4.01	100.00	3.98	100.00	9.06	100.00	5.33	100.00	124	100.00	24.52	100.00
2	Mag.	18.91	2.97	14.45	5.45	23.66	9.39	19.99	5.85	20.13	98	15.67	23.27	18.04
	Non. Mag.	81.09	4.10	85.55	4.10	76.34	8.76	80.01	5.41	79.87	123	84.33	24.65	81.96
	Total	100.00	3.89	100.00	4.36	100.00	8.88	100.00	5.49	100.00	118	100.00	24.39	100.00
3	Mag.	7.59	2.82	5.32	5.26	10.23	10.71	8.92	5.46	7.89	94	5.81	22.08	6.71
	Non. Mag.	92.41	4.12	94.68	3.79	89.77	8.98	91.08	5.23	92.11	125	94.19	25.22	93.29
	Total	100.00	4.02	100.00	3.90	100.00	9.11	100.00	5.25	100.00	123	100.00	24.98	100.00
<i>Metallurgically calculated</i>														