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Testing sustainable mineral processing of Critical Raw Materials (CRM)

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Abstract

The sustainable processing of Critical Raw Materials (CRM) is essential for securing future resource supply while minimizing environmental impact. Titanium dioxide, a key industrial material primarily sourced from rutile, is classified as a critical raw material due to increasing demand and supply risks. Traditional rutile extraction methods pose challenges such as high energy consumption and environmental concerns. This study investigates the potential of eclogitic rocks from the Vara Valley, Italy, as an alternative TiO_2 source, emphasizing sustainable mineral processing techniques to enhance rutile recovery.

A comprehensive characterization of eclogite samples was done by X-ray fluorescence (XRF), X-ray diffraction (XRD), and optical microscopy to assess mineralogical composition and rutile distribution. The study explored dry separation techniques (magnetic and electrostatic separation) as environmentally friendly alternatives to conventional chemical processing. Wet and dry magnetic separation were applied at varying field intensities to isolate ferromagnetic and paramagnetic phases, while electrostatic separation was optimized through controlled voltage, temperature, and triboelectric charging. The results demonstrated that pre-heating samples (100°C) and triboelectric charging significantly improved rutile separation efficiency, highlighting their potential as green alternatives to flotation. Different separation techniques were tested, Test 2 (a combination of magnetic separation followed by electrostatic separation) can be considered a few more effective approach than Test 1 (a combination of electrostatic separation followed by magnetic separation).

The results revealed that a stepwise combination of magnetic and electrostatic separation can serve as an efficient, chemical-free alternative for rutile beneficiation, reducing the environmental impact compared to conventional flotation methods. The results demonstrate the potential of these approaches to enhance efficiency while simultaneously reducing costs associated with energy consumption, processing time, and environmental pollution . Future research should focus on different size separation (coarser grain size), using gravity separation in addition to the other used methods, industrial scalability, energy efficiency optimization, and life cycle assessments to validate the long-term sustainability and economic feasibility of this approach. Contribution of the study to the advancement of green mineral processing technologies, aligning with global efforts to secure sustainable CRM supply chains.

Keywords: Critical Raw Materials, Rutile, Sustainable Mineral Processing, Eclogite, Titanium Dioxide, Magnetic Separation, Electrostatic Separation, Green Processing

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1. Introduction

1.1 Background and Motivation

Critical raw materials (CRMs) are essential for modern industries, particularly in high performance applications such as aerospace, electronics, and renewable energy. Titanium dioxide sourced from rutile, is widely used in pigments, coatings, and metal alloys due to its high stability and optical properties. However, traditional extraction and beneficiation processes pose significant challenges, including high energy consumption, complex mineralogical associations, and environmental concerns.

The European Union and other international regulatory bodies have classified TiO_2 as a critical raw material due to its growing demand and strategic importance. Sustainable alternatives to conventional rutile sources are necessary to secure long-term supply chains while reducing the ecological footprint of extraction processes. Eclogites, high-pressure metamorphic rocks often found in subduction zones, offer a promising alternative due to their natural rutile enrichment.

This study focuses on eclogites from the Vara Valley in Voltri Massif, a region known for its complex geological history. These rocks occur as stratiform layers within serpentinites and have undergone multiple metamorphic transformations. Their mineralogical complexity and rutile-bearing nature make them ideal candidates for testing innovative separation techniques. This research explores various methods, including magnetic, and electrostatic separations, to optimize rutile recovery and assess the economic and environmental feasibility of processing eclogites for rutile production.

1.2 Research Objectives

This research aims to investigate and address the following questions through the analysis of eclogite samples from Vara Valley region:

1. Assessment of Rutile Content in Eclogite: Recognizing the percentage of rutile present in eclogite to evaluate whether the concentration is sufficient for potential titanium extraction from this region.
2. Technological Advancements in Rutile Quantification: Investigation of advancements in analytical techniques (XRD, XRF, Optical Microscopy) have improved the accuracy and reliability of rutile content determination in eclogite compared to previous studies.
3. Feasibility of Dry Separation for Rutile Beneficiation: Evaluating dry separation techniques, such as magnetic and electrostatic separation, are effective for rutile recovery from eclogite, and determining their efficiency compared to conventional methods.
4. Exploring Green Alternatives to Chemical-Based Separation: Investigating whether a sustainable, chemical-free approach to rutile separation can be developed as an alternative to flotation, focusing on its viability, efficiency, and environmental impact.
5. Optimization of Separation Methods for Energy Efficiency and Environmental Sustainability: Identifying the most efficient separation method that minimizes energy consumption, reduces environmental impact, and ensures sustainable rutile beneficiation.

1.3 Geographical Scope

The Vara Valley, located within the Voltri Group, is known for hosting significant eclogitic rock formations within serpentinite matrices. These eclogites, which appear as massive layers up to 100 meters thick, are of high petrogenetic interest due to their unique mineralogical and chemical characteristics. The region has been a subject of study due to its distinctive high-pressure metamorphic history and the presence of rutile-bearing mineral assemblages, which are critical for understanding the economic potential of titanium extraction(R. Bocchio & A. Mottana, 1974).

The eclogites of the Vara Valley are entirely contained within the Monte Beigua-Urbe tectonic unit, one of the three structural subdivisions of the Voltri Group. This unit is bounded by significant fault zones, notably the "Sassello-Monte Colma line" to the north and a fault descending from Ellera to Arenzano in the south. These tectonic movements have shaped the distribution and structural characteristics of the eclogite-bearing layers. The serpentinites in this region exhibit dome-like folding, and the eclogite layers are found concordant with the surrounding schistosity(R. Bocchio & A. Mottana, 1974).

Eclogites of Vara Valley can be categorized into three main types based on their mineralogical composition and textural characteristics:

1. Flaser Eclogites: Characterized by large chloromelanite crystals, sometimes preserving relics of magmatic pyroxenes. These are surrounded by garnet-coronas, often forming a honeycomb texture. Including omphacite, barroisite, and actinolite, indicative of high-pressure conditions.
2. Brecciated Eclogites: Consist of fragments of garnet + glaucophane, embedded in a chloromelanite matrix enriched with rutile and ilmenite. The brecciated elements are held together by diabasic actinolite, suggesting tectonic disruption and subsequent metamorphic overprinting.
3. Retrograde Eclogites: A heterogeneous group, predominantly composed of amphibolic rocks with epidote, albite, and quartz. These eclogites show clear evidence of retrogression from high-pressure eclogite facies to greenschist facies, preserving their eclogitic origins(R. Bocchio & A. Mottana, 1974).

The mineral composition of the Vara Valley eclogites aligns with ultramafic magmatic origins, but their chemistry is different from typical eclogites. These rocks are poorer in SiO_2 but significantly richer in FeO and TiO_2 compared to other known eclogites. Their geochemical profile closely resembles that ilmenite-rich gabbros found in association with the Tuscan ophiolites and ferrogabbros dredged from the Mid-Atlantic Ridge. These similarities suggest that the eclogites of Vara may have originated as iron-rich gabbros intruded into the peridotitic mass before undergoing multiple metamorphic transformations(R. Bocchio & A. Mottana, 1974).

The metamorphic history of these eclogites is highly complex, reflecting multiple stages of pressure-temperature conditions. Their transformation is believed to have occurred under high-pressure, low-temperature blueschist facies, leading to the initial formation of chloromelanite and glaucophane. A subsequent temperature increase under continued high pressure facilitated the crystallization of garnet, omphacite, and rutile, marking the eclogite facies stage. Further metamorphic evolution into the greenschist facies led to the development of barroisitic amphibole, actinolite, and epidote, indicating a progressive retrogression in mineral stability(R. Bocchio & A. Mottana, 1974).

The presence of rutile as an accessory mineral within these eclogites is particularly relevant for titanium resource potential. Rutile occurs both as fine inclusions within garnet and pyroxene and as larger, disseminated grains within brecciated textures. The high TiO_2 content of these rocks makes them promising candidates for titanium beneficiation, provided that efficient mineral liberation and separation techniques are employed. Understanding the metamorphic and textural controls on rutile distribution is crucial for developing a sustainable mineral processing strategy for titanium extraction(R. Bocchio & A. Mottana, 1974).

1.4 Overview

The eclogites of Vara Valley represent a geologically and economically viable alternative for titanium extraction, provided that efficient rutile separation techniques can be developed. This research will contribute to the advancement of sustainable mineral processing methods, improving the economic feasibility of rutile beneficiation while mitigating environmental impacts. Through a comprehensive mineralogical and geochemical assessment, this study will establish the potential of eclogites as a long-term alternative source of titanium, aligning with global efforts to secure critical raw materials for industrial applications.

2. Literature Review

2.1 Introduction

Critical raw materials (CRMs) are essential for modern industries, particularly in renewable energy, electronics, automotive, and defense sectors. Their classification is based on economic importance and supply risk, with rare earth elements (REEs) playing a crucial role due to their unique properties(Filho et al., 2023). The increasing global demand for CRMs has led to geopolitical tensions, environmental concerns, and economic challenges in ensuring a stable supply(Massari & Ruberti, 2013). China dominates the supply of REEs, accounting for over 60% of global production, creating vulnerabilities for other economies dependent on these materials(Salim et al., 2022). Supply chain risks have been highlighted by past trade restrictions, driving efforts to reduce reliance on single suppliers(Filho et al., 2023).

Environmental concerns about CRM extraction include high energy consumption, water pollution, and hazardous waste generation (Hofmann et al., 2018). Mining activities cause deforestation and habitat destruction, necessitating sustainable alternatives. Circular economy approaches, including CRM recycling from electronic waste and industrial byproducts, are being explored, but technological and economic constraints hinder widespread adoption(Filho et al., 2023). Current recycling methods struggle with the complexity of CRM-containing products, and primary extraction remains more cost-effective in many cases(Massari & Ruberti, 2013). Efforts to improve CRM sustainability focus on product design for easier disassembly, stricter regulations, and investments in recycling technologies (Hofmann et al., 2018).

Policymakers are taking action to secure CRM supply chains. The European Critical Raw Materials Act (CRMA) aims to increase domestic extraction, processing, and recycling while fostering strategic international partnerships(A. Hool et al., 2023). The U.S. Inflation Reduction Act (IRA) and the Minerals Security Partnership (MSP) also seek to diversify CRM supply sources and encourage responsible mining practices(Filho et al., 2023). Future strategies include exploring alternative sources such as deep-sea mining and urban mining, developing material substitutes, and strengthening global cooperation through ethical and environmentally responsible extraction frameworks (Salim et al., 2022).

The secure and sustainable management of CRMs is crucial for technological advancement and economic stability. Addressing supply chain vulnerabilities requires a combination of sustainable mining practices, enhanced recycling initiatives, strategic policies, and international partnerships (A. Hool et al., 2023). The implementation of regulatory measures like the CRMA and collaborative efforts will be vital in ensuring long-term CRM availability while minimizing environmental impact (Filho et al., 2023).

2.2 Eclogite

Eclogites are high-pressure metamorphic rocks composed primarily of garnet and omphacite. They are key indicators of subduction zone processes and reveal important insights into the deep Earth recycling of crustal materials. Eclogites form under conditions of high pressure (>1.2 GPa) and moderate to high temperatures (400–800°C), typically within subducted oceanic crust or continental crust that has been buried to mantle depths. They have a crucial role in understanding plate tectonics, the thermal structure of subduction zones, and element cycling in the mantle.

This summary explores eclogites' petrology, geochemistry, formation, exhumation, and tectonic implications based on various studies. Eclogites are primarily composed of garnet (Alm-Pyr-Grs) and omphacite (a sodic clinopyroxene with varying jadeite content). Accessory minerals include lawsonite, rutile, glaucophane, phengite, zoisite, epidote, and kyanite. Lawsonite eclogites, which form under very low-temperature conditions in subduction zones, are particularly significant due to their role in volatile element storage and transport (Tatsuki Tsujimori et al., 2006).

Garnet in eclogites often exhibits zoned compositions, with growth stages recorded in core-rim zoning. The garnet zoning reflects changes in pressure-temperature (P–T) conditions during subduction and exhumation. Prograde zoning is characterized by an increase in Mg/Fe ratio and the development of inclusions such as lawsonite, omphacite, and quartz, which mark specific stages of metamorphism(H.J. Massonne & B. Li, 2020).

Clinopyroxene in eclogites varies in composition, with jadeite content increasing under higher pressure conditions. Omphacite commonly contains 30–60 mol% jadeite, and its compositional variation is crucial for estimating the P–T conditions of eclogite formation.

Eclogites have a wide geochemical range depending on their protoliths (basaltic, sedimentary, or ultramafic rocks). They typically retain mid-ocean ridge basalt (MORB)-like signatures with enrichment in high-field-strength elements (HFSE) such as Nb, Ta, and Zr. However, some eclogites, particularly those associated with continental crust, show geochemical signatures resembling island-arc basalts or continental flood basalts (Tatsuki Tsujimori et al., 2006).

Trace element studies of eclogites reveal the importance of lawsonite and rutile as reservoirs for rare earth elements (REE), strontium (Sr), lead (Pb), and uranium (U). Lawsonite-rich eclogites are significant because they serve as high-pressure carriers of these elements into the mantle(Tatsuki Tsujimori et al., 2006).

Eclogites form primarily in subduction zones where oceanic crust descends into the mantle. The transition from blueschist to eclogite occurs at depths of 50–90 km, depending on geothermal gradients. The low-temperature lawsonite eclogites found in Guatemala and the Dominican Republic suggest that very cold subduction geotherms (~6°C/km) are necessary for their stability (Tatsuki Tsujimori et al., 2006).

The presence of lawsonite eclogites in serpentinite-matrix mélanges suggests that fluid-assisted metamorphism plays a critical role in their formation. Serpentinite provides a buoyant medium that aids the exhumation of these rocks from mantle depths (Tatsuki Tsujimori et al., 2006).

Egression of eclogites from mantle depths back to the surface is a complex process. The mechanisms proposed for exhumation include:

1. Buoyancy-Driven Return Flow: Low-density materials, such as serpentinites, help buoyantly lift eclogites back toward the crust.
2. Oblique Convergence and Strike-Slip Faulting: Many eclogites are associated with major strike-slip faults, where oblique subduction and lateral displacement enable their return to the surface (P. Agard et al., 2008)

3. Slab Detachment and Ridge Subduction: These processes create vertical uplift forces that exhumed eclogitic blocks to shallow levels (Sarah C. Penniston-Dorland et al., 2015)

The presence of retrograde mineral assemblages (e.g., glaucophane, albite, chlorite) in eclogites indicates a progressive transition from eclogite-facies to blueschist- or greenschist-facies conditions during exhumation (Feng Zhao et al., 2021).

Eclogites play a crucial role in reconstructing past tectonic environments. They provide direct evidence of subduction and continental collision, helping geologists determine subduction zone thermal structures and tectonic plate interactions (Tatsuki Tsujimori et al., 2006)

1. Subduction Zone Thermal Structure:

- The thermal evolution of subducting slabs is crucial for understanding dehydration reactions that generate arc magmas.
- Eclogite-bearing terranes preserve P–T conditions that help refine models of subduction dynamics (Sarah C. Penniston-Dorland et al., 2015)

2. Continental Collision and Exhumation:

- High-pressure eclogites in the Western Alps, Norway, and the Himalayas indicate deep subduction and subsequent uplift of continental crust.
- Some ultrahigh pressure (UHP) eclogites contain coesite, indicating burial to depths over 90 km before exhumation (Brown, 2023)

3. Element Cycling and Mantle Metasomatism:

- Eclogites contribute to the geochemical evolution of the mantle by recycling elements such as REE, Pb, U, and Sr through subduction.
- The breakdown of lawsonite and phengite in eclogites releases water and trace elements that influence the composition of arc magmas (Tatsuki Tsujimori et al., 2006)

2.3 Mineral Processing

Mineral processing involves the extraction and refinement of valuable minerals from ores, playing a critical role in ensuring a sustainable supply of raw materials for industrial applications. One of the main titanium-bearing minerals is rutile, which is widely used in pigment production, welding rods, and as a precursor for titanium metal production. The processing of rutile involves several techniques, including physical separation, chemical beneficiation, and innovative approaches for maximizing recovery efficiency. Given the growing demand for titanium-based materials, sustainable and efficient processing of rutile is critical for minimizing environmental impact and maximizing resource utilization.

Rutile is a naturally occurring titanium dioxide mineral found in various geological environments, including igneous, metamorphic, and sedimentary rocks. Eclogites, high-pressure metamorphic rocks, are known to host significant concentrations of rutile, particularly in regions like the Vendée eclogites of France, where rutile forms in association with quartz and omphacite(Alexandra Mauler et al., 2000). The mineral exhibits excellent resistance to chemical weathering, leading to its accumulation in heavy mineral sands, which are a major commercial source of titanium.

The processing of rutile primarily involves physical and chemical beneficiation techniques. The choice of method depends on the nature of the ore, mineral associations, and the desired product quality.

1. Physical separation techniques, such as gravity separation, magnetic separation, and flotation, are widely employed for rutile beneficiation.

- Gravity Separation: Due to its high density (4.2–5.6 g/cm^3), rutile is often concentrated using gravity separation techniques, including spirals and shaking tables.
- Magnetic Separation: Although rutile itself is non-magnetic, high-gradient magnetic separation (HGMS) has been employed to remove paramagnetic impurities such as ilmenite and iron oxides (Zheng et al., 2023)

- Flotation: Flotation is one of the most effective methods for rutile recovery, particularly from complex ores. The process relies on surfactants to selectively separate rutile from gangue minerals such as quartz and feldspar.

2. Chemical processing methods are important for recovering rutile and removing impurities that cannot be eliminated through physical separation.

- Leaching Techniques: Various acids, such as sulfuric and hydrochloric acid, are used to dissolve impurities and improve rutile purity.
- Chlorination Process: The chlorination process is a well-established method for producing titanium tetrachloride ($TiCl_4$), a key intermediate for titanium metal and titanium dioxide production (Dai et al., 2024)

3. Novel and Sustainable Approaches: Recent advancements have focused on sustainable and innovative methods for rutile recovery, reducing environmental impact and enhancing efficiency.

- Bioleaching: Microorganisms are being explored for their potential to leach impurities selectively from rutile-bearing ores.
- Magnetic Fluid Separation: High-gradient magnetic separation coupled with magnetic fluids has demonstrated promising results in enhancing rutile recovery (Zheng et al., 2023)
- Recovery from Secondary Resources: With the depletion of primary rutile sources, alternative sources such as industrial waste and recycled materials are gaining importance (Feng et al., 2023)

2.4 Characterization

Mineral characterization is a fundamental aspect of geosciences, materials science, and mineral processing, as it enables the identification, composition analysis, and structural determination of minerals. Among the most widely employed techniques in mineral characterization are X-ray Fluorescence (XRF), X-ray Diffraction (XRD), and Optical Microscopy (OM). These methods provide critical insights into the chemical composition, crystalline structure, and morphological characteristics of minerals. Each technique has its advantages, limitations, and specific applications in mineralogical and industrial research. This paper discusses the principles, applications, and significance of these techniques based on recent studies.

2.4.1 XRF

XRF is a widely used non-destructive analytical technique that determines the elemental composition of minerals. The method is based on the emission of characteristic secondary (or fluorescent) X-rays from a material when it is excited by a high-energy X-ray source. The emitted X-rays have wavelengths unique to each element, allowing precise quantification of elemental concentrations. XRF has been extensively applied in geochemistry and mineral processing to analyze major, minor, and trace elements in various matrices. A study on the synchrotron-based micro-XRF (μ-XRF) highlights its application in analyzing element distribution in environmental matrices (e.g., soil and biological samples), demonstrating high spatial resolution and sensitivity, making it useful for investigating heterogeneous samples (Majumdar et al., 2012)

Moreover, XRF plays a key role in quality control in mineral processing industries, particularly in determining the composition of ores and raw materials. For instance, in titanium dioxide (TiO_2) production, XRF is used to analyze ilmenite ore before and after beneficiation to optimize extraction processes (Klepka et al., 2005)

Despite its advantages, XRF has limitations, including:

- Inability to detect light elements (e.g., H, Li, Be, B) effectively due to low-energy X-ray emission.
- Dependence on sample homogeneity, varying mineral phases may affect accuracy.
- Requirement of standards for accurate quantification, particularly in complex mineral matrices.(Majumdar et al., 2012)

2.4.2 XRD

XRD is an essential technique used to determine the crystalline structure of minerals by measuring the diffraction pattern of X-rays interacting with a mineral's atomic lattice. This technique is fundamental in phase identification, mineral polymorphism studies, and high-temperature phase transformations.

One of the important applications of XRD is in the detection of polymorphs and phase transitions in minerals. For instance, the anatase-to-rutile phase transformation in TiO_2 has been extensively studied using in situ high-temperature XRD, providing insights into the structural evolution and kinetics of this phase change, which is crucial in pigment and photocatalyst applications (Karunadasa & Manoratne, 2022).

In another study, high-pressure and high-temperature deformation experiments on eclogite, dunite, and clinopyroxenite used in situ XRD to investigate their structural modifications. These studies are particularly relevant for understanding the deformation mechanisms of minerals under extreme conditions, such as those found in the Earth's mantle (Farla et al., 2017).

Furthermore, XRD is commonly used in ore characterization for mining and metallurgical applications. It enables the identification of mineral assemblages and helps optimize mineral processing strategies. A review of synchrotron-based XRD in ore geology research demonstrates its application in the characterization of fine-grained mineral phases, where conventional techniques fail to provide adequate resolution (Huang et al., 2021).

However, XRD also has limitations, such as:

Poor sensitivity to amorphous materials, which lack long-range atomic order.

Overlapping diffraction peaks, which can complicate the identification of mixed-phase samples.

Sample preparation requirements, such as fine grinding, to avoid preferred orientation effects.(Karunadasa & Manoratne, 2022)

2.4.3 Optical Microscopy

Optical microscopy remains one of the oldest and most fundamental techniques for mineral characterization. It allows for the visual examination of mineral textures, grain boundaries, inclusions, and crystal defects under polarized or reflected light.

Thanks to the recent advancements, automated mineralogical characterization using optical microscopy, where image-processing algorithms enhance the efficiency of mineral identification and quantification (De Castro et al., 2022) is possible. This technique is particularly useful in mineralogical studies and petrography, where the morphology and relationships between minerals provide crucial geological insights.

Additionally, in situ optical microscopy has been employed to observe crystal growth and surface modifications in real-time, allowing researchers to study crystallization dynamics and mineral weathering processes (Sazaki et al., 2016). This application is particularly useful in mineral surface chemistry studies, which are crucial for improving flotation and leaching processes in mineral processing.

Despite its significance, optical microscopy has certain limitations:

- Limited magnification and resolution compared to electron microscopy techniques.
- Subjectivity in mineral identification, which relies on operator expertise.
- Challenges in characterizing fine-grained minerals, where phase boundaries are unclear (Sazaki et al., 2016).

2.5 Separation

2.5.1 Magnetic Separation

Magnetic separation is a crucial technique in mineral processing, particularly for the concentration of paramagnetic and ferromagnetic minerals. This method exploits differences in magnetic susceptibility between minerals, facilitating the removal of impurities or the concentration of valuable ores. The application of magnetic separation spans a broad range of minerals, including iron ores, titanium-bearing minerals, and other critical raw materials. This paper provides an academic discussion on both dry and wet magnetic separation techniques, analyzing their principles, advantages, and challenges based on recent studies.(H. Xu et al., 2009)

Magnetic separation utilizes magnetic forces to separate minerals based on their magnetic susceptibility. This property defines a mineral's ability to be attracted by a magnetic field. Minerals are generally classified as ferromagnetic, paramagnetic, or diamagnetic:

- Ferromagnetic minerals, such as magnetite (Fe_3O_4), exhibit strong magnetization and can be easily separated.
- Paramagnetic minerals, such as ilmenite ($FeTiO_3$) and chromite ($FeCr_2O_4$), have weak magnetic susceptibility, but can still be separated by using high-intensity magnetic separators.
- Diamagnetic minerals, including quartz and feldspar, show no attraction to a magnetic field and remain unaffected.(H. Xu et al., 2009)

Magnetic separation processes are broadly categorized into dry and wet magnetic separation, depending on the state of the feed material.

Dry magnetic separation is preferred when dealing with coarse particle sizes and when availability of water is limited. It is widely applied in processing iron ores, heavy mineral sands, and industrial minerals.

Advantages of Dry Magnetic Separation:

- Lower operational costs as no water is required

- Environmentally friendly due to minimal waste generation
- Suitable for coarse-grained minerals, ensuring higher throughput and efficiency (Nzeh & Popoola, 2024).

Challenges of Dry Magnetic Separation:

- Limited efficiency for fine particles, as inter-particle forces hinder proper separation.
- Dust generation, requiring additional measures for dust control.
- Lower recovery rates in some mineral systems compared to wet separation (Nzeh & Popoola, 2024).

Applications in ilmenite and titanium recovery: Dry magnetic separation has been studied for ilmenite recovery, a key source of titanium. Studies have highlighted the role of magnetic property modification in improving separation efficiency. For instance, researchers have successfully enhanced the magnetic properties of ilmenite concentrates by inducing phase transformations before subjecting them to magnetic separation (Lv et al., 2017).

Wet magnetic separation is generally more effective for fine particles and slurries. This method is used extensively in iron ore beneficiation, recovery of ultrafine mineral fractions, and treatment of oxidized ores.

Advantages of wet magnetic separation:

- Higher recovery rates, especially for fine-grained materials.
- Reduced dust emissions, leading to better environmental sustainability.
- Improved selectivity, as the presence of water minimizes unwanted interactions between particles(L. Chen et al., 2015).

Challenges of wet magnetic separation:

- Higher water consumption, which may limit its use in arid regions.
- Slurry handling issues, such as pump wear and sedimentation.
- More expensive infrastructure and maintenance requirements (L. Chen et al., 2015).

Innovative applications in high gradient and centrifugal magnetic separation: Recent advancements in high gradient magnetic separation (HGMS) and centrifugal field magnetic separation have significantly improved the recovery of weakly magnetic minerals. HGMS applies a strong, non-uniform magnetic field to capture fine particles in a matrix of ferromagnetic material, making it effective for recovering ultrafine minerals. A centrifugal field further enhances separation by counteracting the effect of water drag on fine particles (L. Chen et al., 2015).

Magnetic Susceptibility and Its Role in Separation Efficiency: The efficiency of magnetic separation depends largely on mineral magnetic susceptibility, which varies under different geological and metamorphic conditions. Studies on ultrahigh-pressure eclogites have demonstrated how changes in mineral composition during retrogression impact their magnetic properties (H. Xu et al., 2009). This understanding is crucial for optimizing magnetic separation in complex ore deposits.

2.5.2 Electrostatic Separation

Electrostatic separation is a key technique in mineral processing, particularly in the beneficiation of titanium-bearing minerals such as rutile (TiO_2). This method exploits differences in electrical conductivity between minerals to achieve selective separation. The use of electrostatic separation in rutile processing is favored due to its dry operation, energy efficiency, and reduced reliance on chemical reagents. Recent advancements in electrostatic separation technology have further improved its efficiency, making it an essential process for high-purity rutile recovery.

Electrostatic separation relies on the selection of charging of mineral particles when exposed to an electric field. Conductive minerals like rutile rapidly acquire and dissipate charge, whereas non-conductive minerals retain charge for longer periods. This difference in electrical behavior enables the effective separation of rutile from gangue minerals, such as zircon and quartz.

Different electrostatic separation techniques have been developed to optimize mineral separation efficiency. Among them, electrostatic deflection and triboelectric separation are widely applied. Electrostatic deflection utilizes high-voltage electric fields to cause differential movement of mineral particles based on their charge properties (Nzeh & Popoola, 2024). Triboelectric separation, on the other hand, involves frictional charging between minerals and a charging medium, facilitating selective adherence of charged particles (Z. Zhao et al., 2022).

Electrostatic separation is extensively used in rutile processing due to its ability to achieve high-purity concentrates with minimal environmental impact. In a typical mineral processing plant, rutile is first pre-concentrated using gravity and magnetic separation techniques before being subjected to electrostatic separation. This step is crucial in removing impurities such as ilmenite and zircon, which have different electrostatic properties.

Key factors influencing electrostatic separation efficiency include:

- Moisture Content: High moisture levels can reduce charge accumulation on mineral surfaces, leading to poor separation efficiency. Drying the feed material before electrostatic separation is necessary for optimal results (Nzeh & Popoola, 2024).
- Particle Size Distribution: Fine particles tend to form agglomerates due to strong electrostatic interactions, which can negatively impact separation efficiency. Advanced electrostatic separation technologies are being developed to address this limitation (Y. Yu et al., 2022).
- Surface Charge Characteristics: The electrostatic properties of rutile surfaces significantly influence its separation behavior. Modifying surface charge through controlled charging mechanisms enhances the selectivity of the separation process (Romero-Morán et al., 2022).

Recent research has led to significant advancements in electrostatic separation technology, improving the efficiency and selectivity of rutile processing. One of the key innovations is the application of electrostatic traveling wave methods, which utilize dynamic electric fields to manipulate particle trajectories more effectively. This technique has proven particularly beneficial for the separation of fine rutile particles, which traditionally posed challenges for conventional electrostatic separators (Y. Yu et al., 2022).

Another major breakthrough is the development of direct-current (DC) triboelectric nanogenerators, which enhance charge transfer efficiency between mineral particles and the charging medium. This technology has shown promising results in improving the differential charging of rutile and gangue minerals, leading to higher separation efficiency (Z. Zhao et al., 2022).

The surface charge behavior of rutile plays a crucial role in determining its electrostatic separation efficiency. Studies have shown that the surface charge of rutile can be modified through various means, including triboelectric charging and corona discharge. These modifications influence charge retention and separation efficiency (Romero-Morán et al., 2022).

Additionally, rutile's surface electrostatic properties are of interest beyond mineral processing. Research has explored how electrostatically charged rutile surfaces exhibit enhanced photocatalytic activity, which has implications for environmental applications such as bacterial inactivation. This suggests that understanding the electrostatic behavior of rutile can provide insights for both mineral processing and other technological applications (Romero-Morán et al., 2022).

Despite its advantages, electrostatic separation in rutile processing faces several challenges:

1. Moisture Sensitivity: Electrostatic separation is highly sensitive to moisture, which can alter charge accumulation and affect separation performance. Effective drying and environmental control are necessary to maintain efficiency (Nzeh & Popoola, 2024).
2. Fine Particle Processing: The separation of fine rutile particles is problematic due to their strong electrostatic interactions, leading to reduced efficiency. Advanced separation methods, such as traveling wave separation, are being developed to overcome this issue (Y. Yu et al., 2022).
3. Surface Contamination: Surface coatings and impurities can influence charge transfer mechanisms, leading to suboptimal separation results. Surface pre-treatment methods, such as controlled conditioning, can improve process efficiency (Siddique et al., 2011).

2.6 Italian work

This section provides a comprehensive review of Italian literatures (Geomineraria Italiana 1976) and the findings derived from studies and experiments conducted on the same eclogite samples from the Vara Valley.

The extraction and processing of rutile from rock-hosted deposits present significant technical and economic challenges compared to traditional sources such as placer sands. Rutile (TiO_2), a high-value titanium mineral, is typically recovered from heavy mineral sands; however, interest in hard-rock sources has grown due to increasing demand for high-purity titanium dioxide for the pigment and aerospace industries (R. Bocchio & A. Mottana, 1974)

This study examines the processing of titanium-bearing eclogites from the Vara Valley in Italy, a region within the Ligurian ophiolite complex. The deposit contains rutile, ilmenite, and titanite as the primary titanium minerals. Research conducted by the Institute of Mining at the University of Rome, in collaboration with Solmine Company, focused on developing an optimized extraction route. The investigation involved a sequence of physical and chemical separation techniques, including comminution, gravity concentration, magnetic separation, and flotation, followed by metallurgical treatment.

The Pianpaludo titanium deposit, located within the ophiolitic Voltri Group, is among the most promising hard-rock titanium deposits in Italy. The mineralization is hosted in eclogitic amphibolites, intercalated within serpentinite and prasinite formations (R. Bocchio & A. Mottana, 1974). The presence of major regional faults influences the distribution and concentration of titanium-bearing phases.

The primary titanium-bearing minerals in the deposit include:

- Rutile (TiO_2): The dominant titanium phase, occurring in variable proportions with ilmenite.
- Ilmenite ($FeTiO_3$): Often intergrown with rutile, contributing to overall titanium content.
- Titanite ($CaTiSiO_5$): A secondary titanium-bearing mineral present in subordinate amounts.

The gangue minerals primarily consist of garnet (almandine-spessartine), clinopyroxenes (diopside-omphacite), amphiboles (actinolite, barroisite, glaucophane), quartz, epidote, and plagioclase. The overall TiO_2 content ranges from 4.5% to 7.5%, necessitating beneficiation to produce a marketable concentrate (Di Prisco & Sun, 1976).

Due to the fine intergrowth of rutile with silicate minerals, multiple separation techniques were employed to achieve optimal recovery. The processing stages included crushing and grinding, gravity concentration, magnetic separation, and flotation.

To liberate rutile from the host rock, the material was subjected to crushing and grinding. However, the mechanical properties of the eclogitic rock posed significant challenges, with recorded values including:

- Compressive strength: 5,500 kg/cm^2
- Tensile strength: 1,000 kg/cm^2
- Shear strength: 2,700 kg/cm^2
- Bond Work Index: 14

Liberation Challenges:

- Rutile occurs as fine inclusions, necessitating fine grinding (<50 μm) for complete liberation.
- Excessive grinding led to high ultrafine content, complicating downstream separation.
- A compromise was established by grinding to 65 mesh (U.S. Tyler standard) to balance liberation and operational efficiency (R. Bocchio & A. Mottana, 1974)

Gravity separation was tested due to the high specific gravity of rutile (4.2–5.6 g/cm^3).

- Shaking tables were employed to separate heavy minerals from lighter silicates.
- The resulting garnet-rutile concentrate had a concentration ratio of approximately 2:1, necessitating further refining.
- Gravity separation was limited by fine particle size, leaving a significant portion of rutile interlocked with other minerals.

Magnetic separation was employed to remove iron-bearing minerals (ilmenite, magnetite, and iron-rich garnet) from the rutile fraction.

- Drum magnetic separators were used with field strengths up to 8000 Gauss.

- The process was applied to two size fractions: 65–325 mesh (raw ore) and 150–325 mesh (preconcentrate).
- Applying magnetic separation after gravity concentration yielded better results, removing iron-bearing phases while retaining rutile (Purcell & Sun, 1974).

Flotation was used to further refine the rutile concentrate, leveraging the surface chemistry of titanium minerals.

Surface Chemistry and pH Optimization:

- The isoelectric point of rutile was determined to be pH 6.0.
- Anionic flotation was conducted in an acidic pH range (4–6) using oleic acid as a collector.
- Cationic flotation at higher pH values was ineffective due to competing adsorption effects (Di Prisco & Sun, 1976)

Collector Selection:

- Oleic acid (800 g/t) was identified as the most effective collector.
- Sodium fluoride (NaF) acted as a silicate depressant, enhancing flotation selectivity.
- A two-stage flotation sequence was adopted: Step 1: Removal of pyrite impurities with potassium amyl xanthate at pH 5.5. Step 2: Rutile flotation under moderately acidic conditions, achieving a purer TiO_2 concentrate.

Results of Flotation:

- Selectivity peaked within pH 4–6, beyond which gangue minerals entered the concentrate.
- A 30% overall rutile recovery was achieved, a favorable outcome given the fine-grained nature of the deposit (Purcell & Sun, 1974)

The optimized beneficiation process follows this sequence:

1. Crushing & Grinding → Reduction to 65 mesh
2. Gravity Preconcentration → Shaking table separation for garnet-rutile concentrate

3. Magnetic Separation → Removal of iron-bearing minerals
4. Flotation → pH 4–6 anionic flotation with oleic acid
5. Final Processing → Refinement and drying of rutile concentrate

Challenges and Future Considerations

1. High Energy Costs: The rock's hardness increases comminution costs, necessitating alternative strategies.
2. Selective Mining Strategies: Variable rutile-ilmenite ratio suggests that targeted extraction could improve processing efficiency.
3. Waste Disposal: A hydraulic transport system for tailings is proposed due to large gangue volumes.
4. Flotation Optimization: Advanced preconcentration techniques, such as centrifugal separators, could enhance rutile recovery (R. Bocchio & A. Mottana, 1974)

The extraction of rutile from Vara Valley eclogites was demonstrated to be feasible using an integrated beneficiation approach combining gravity separation, magnetic separation, and flotation. The optimized process flow achieved a 30% rutile recovery, highlighting the technical potential of rock-hosted titanium deposits.

Given the growing demand for high-purity rutile, further research should focus on refining flotation techniques and reducing comminution costs to enhance economic viability. The study provides a framework for future large-scale extraction from metamorphic titanium sources.

2.7 Overview

The research and discussion presented in this chapter highlight the critical importance of sustainable approaches to mineral processing, particularly in the context of critical raw materials

(CRMs) such as rutile. As industrial demand for CRMs continues to rise, understanding their geological occurrence, extraction methods, and processing technologies becomes increasingly essential for ensuring long-term resource availability while minimizing environmental impact (Filho et al., 2023)

The characterization and processing of eclogite-hosted rutile deposits offer valuable insights into the challenges and advancements in mineral beneficiation. The geological significance of eclogites, particularly their role in subduction zone dynamics and element cycling, underscores their relevance not only in geoscientific research but also in resource exploration (Tatsuki Tsujimori et al., 2006). The petrological and geochemical studies on eclogites provide crucial data for optimizing mineral recovery, particularly in distinguishing valuable minerals such as rutile from complex gangue minerals (H.J. Massonne & B. Li, 2020)

The mineral processing techniques explored in this chapter, gravity separation, magnetic separation, electrostatic separation, and flotation demonstrate the need for an integrated approach in rutile beneficiation. While gravity and magnetic separation methods serve as effective pre-concentration strategies, flotation remains the primary method for achieving high-purity rutile concentrates, especially from complex ore bodies (Zheng et al., 2023). Innovations in electrostatic separation and high-gradient magnetic separation further enhance the efficiency of mineral recovery, contributing to the advancement of sustainable processing technologies (Dai et al., 2024)

The role of advanced characterization techniques such as X-ray Fluorescence (XRF), X-ray Diffraction (XRD), and Optical Microscopy (OM) is indispensable in mineral processing. These methods provide fundamental insights into mineral composition, crystallography, and surface morphology, thereby guiding process optimization and enhancing recovery efficiency (Majumdar et al., 2012). The integration of mineralogical and process data through these techniques ensures that beneficiation strategies are tailored to the specific characteristics of each ore deposit.

The case study on Italian eclogites, particularly from the Vara Valley, underrates the potential of alternative rutile sources beyond traditional placer deposits. The processing challenges associated with hard-rock rutile deposits necessitate advanced beneficiation techniques, with a focus on flotation optimization, selective mining strategies, and cost-effective comminution methods (R. Bocchio & A. Mottana, 1974). The research findings from the Italian studies provide a framework for future exploration and processing of similar deposits globally, reinforcing the need for sustainable and economically viable extraction methods.

Ultimately, the sustainable management of CRMs requires a multi-faceted methods that integrates geological understanding, advanced processing techniques, and policy-driven frameworks such as the European Critical Raw Materials Act (CRMA) and the U.S. Inflation Reduction Act (IRA) (A. Hool et al., 2023). Addressing the challenges associated with CRM extraction and supply chain vulnerabilities necessitates continued innovation in mineral processing, enhanced recycling initiatives, and international collaboration to develop environmentally responsible extraction frameworks (Salim et al., 2022)

Future research should focus on improving the efficiency of rutile recovery from eclogite sources, particularly through novel flotation reagents, alternative separation techniques, and energy-efficient processing methods. Additionally, advances in mineral characterization, such as synchrotron-based techniques and real-time monitoring technologies, could further refine beneficiation strategies and contribute to the development of more sustainable mineral processing solutions.

In conclusion, this chapter has demonstrated the complexities and opportunities associated with CRM extraction and processing. By adopting a holistic approach that combines geological research, innovative processing methods, and strategic policy implementation, the mineral industry can move towards a more sustainable future while ensuring a stable and secure supply of critical raw materials for technological and economic development.

3. Materials and Methodology

3.1 Introduction

The study of sustainable mineral processing for critical raw materials requires a comprehensive and systematic approach to sample preparation, characterization, and separation techniques. This research focuses on the processing of rutile from eclogitic ores, as a valuable mineral. A sequence of experimental procedures has been carefully followed to ensure the reliability and reproducibility of results, incorporating standard mineral processing techniques such as sieving, crushing and milling, X-ray fluorescence (XRF), X-ray powder diffraction (XRD), optical microscopy, and various separation methods including wet magnetic separation, dry magnetic separation, and electrostatic separation.

The primary phase of this study included a detailed assessment of the sample material. An old bulk sample of 9.85 kg was acquired from the Vara Valley and subjected to a sampling process using a riffle splitter to ensure a representative sub-sample was obtained. A predefined series of sieves ranging from 6.7 mm to 0.053 mm was employed to classify the sample into different size fractions. Granulometric analysis was conducted to determine key parameters such as D80, D50, and D20 values, which provided essential insights into particle size distribution.

Subsequent steps included packing, manual milling of selected samples to achieve a fine fraction suitable for XRF and XRD analyses. Due to the high hardness of eclogitic minerals, manual milling proved challenging and required extended processing times. The resulting powdered samples were subjected to XRF analysis to determine elemental composition, utilizing a portable X-ray fluorescence device under multiple tests for enhanced accuracy. XRD analysis followed, using a calibrated diffractometer, SmartLab Studio II software and its library for mineralogical identification.

To the study within mineral separation, optical microscopy was employed to evaluate the liberation characteristics of rutile within different particle size fractions. Samples were then processed using a series of separation techniques. Wet and dry magnetic separation were conducted at multiple magnetic field intensities to assess the feasibility of magnetically isolating valuable minerals among gangue minerals. Electrostatic separation were conducted under various operational conditions, including voltage, and temperature. Optical microscopic and XRF analyses of different test conditions were performed to validate the separation outcomes. Separation includes two optimization tests, Test , and Test2.

3.2 Sampling

In the context of my study, two distinct sampling methods were tested to identify the most efficient approach for obtaining a representative and reliable sample: Coning and Quartering and Riffle Splitting.

- Coning and quartering:

The first method applied was coning and quartering, a well-established technique for sample reduction. This process involved piling the bulk material into a conical shape, resembling an inverted funnel. The material was then leveled to ensure uniformity and divided into four equal sections. Two diagonally opposite quadrants were selected for further processing, while the remaining two were preserved as reserve material. This method was aimed at reducing sampling bias and ensuring homogeneity. However, its reliance on manual handling introduced potential inconsistencies, making its reproducibility dependent on the operator's precision.

- Riffle splitter:

The second method employed was riffle splitter (figure1), a mechanical approach designed for accurate and systematic sample division. To achieve the final sample needed for analysis, the process was executed in three sequential steps using two different splitter sizes:

a) First Stage: A large-sized riffle splitter was utilized to halve the bulk sample.

b) Second Stage: Given the reduced volume sample, a medium-sized riffle splitter was then employed to further divide the material, ultimately yielding a quarter of the first step bulk sample for subsequent testing.

Figure 1: Riffle splitter and dust control system

3.3 Screening and sieving

To categorize the material into distinct size fractions, a sieving method was employed using a set of sieves with mesh sizes of 6.7 mm, 4 mm, 2 mm, 1 mm, 0.5 mm, 0.212 mm, 0.106 mm, 0.053 mm (figure2), and a pan to collect particles smaller than 0.053 mm.

Sieving Procedure:

- Sample Introduction: The material was introduced into the top sieve (6.7 mm) to allow sequential separation of particles by size.
- Shaking Process: The sieves were securely sealed, and sieving was conducted using an electromechanical shaker to ensure a uniform and controlled vibration by modifying power, cycle and time. Setup include: (power: 8, cycle: 1, time: 5). This facilitated effective size separation by promoting particle movement through the sieve apertures.
- Dust control and safety measures: The sieving process was performed under a fume hood to control dust dispersion and maintain a safe working environment while wearing mask, jacket, and gloves.
- Collection, Weighing, and Packaging: After sieving, the material retained on each sieve was carefully collected, weighed, and stored in labelled containers, Eclo-1 to Eclo-9 are

particle sizes from coarse to fine; Eclo-1 contains retaining on 6.7 mm, Eclo-9 is retaining in pan.

Figure 2: Sieves used and electromechanical shaker

3.4 Characterization

3.4.1 XRF

To prepare the samples for X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) analysis, it was essential to reduce the particle size of the material. Given the limited volume of the samples, manual milling was chosen over mechanical milling methods, which are typically more efficient but require larger sample sizes. The milling process aimed to achieve a particle size below 30 microns to ensure homogeneity and analytical accuracy. The key considerations during manual milling were:

- Sample selection and size reduction: A representative portion of each size fraction was taken to undergo milling. The objective was to reduce the particle size to below 30 microns, ensuring it was fine enough not to be felt between two fingers.
- Challenges in milling process: The material revealed high hardness, making size reduction a time-consuming process. Each sample required approximately 45 minutes of manual milling, emphasizing the difficulty of the process.
- Multi-purpose milling: The finely milled powder was intended for both XRF and XRD analyses, requiring a balance in particle size to ensure consistency across both methods.

After achieving the target particle size, the samples were prepared for XRF analysis as follows:

- Sample packaging: Each size fraction was transferred into a plastic cup designed for XRF analysis. A special nylon cover was placed over the sample, ensuring that it did not interfere with X-ray reflection. Two plastic rings were used to secure the cover (one fitting around the cup and the other fixing the top).
- Weighting adjustment: Each sample was carefully weighed to ensure it contained at least 7 grams of material, sufficient for accurate XRF measurement as a dense material. The powder was evenly spread within the cup to form a uniform layer, a critical factor in obtaining precise XRF readings.

- Final check: Before analysis, the sample integrity was confirmed, ensuring no air gaps or inconsistencies in the coverage that might affect the XRF results.

To conduct the X-ray Fluorescence (XRF) analysis, the SciAps X-555 portable XRF analyzer (figure 3) was utilized. To ensure safety and measurement accuracy, a protective shield (figure 4) was employed during testing. The device was connected to a laptop for remote operation, ensuring that all tests were conducted under stable conditions with minimal interference (figure 5).

For consistency and reproducibility:

- The XRF analyzer was fixed on a stand to prevent movement during testing.
- The system was controlled remotely, eliminating variations caused by manual handling.

Testing Procedure

XRF tests were conducted on 9 samples (Eclo-1 to Eclo-9), using two distinct modes to ensure comprehensive elemental detection:

1. Mineral Mode – optimized for detecting metallic and high-density mineral components.
2. Soil Mode – designed to detect lighter elements and trace contaminants.

Each sample underwent two separate tests in both modes, each one for three times:

- Mineral Mode used two X-ray beams to analyze the sample. Each test had an acquisition time of 60 seconds.
- Soil Mode utilized three X-ray beams for broader elemental detection. Each test lasted 90 seconds.

To ensure precision and reliability, each test was performed in triplicate, three repeat measurements per mode were averaged to mitigate measurement variability and the final data was exported to an Excel spreadsheet.

Figure 3: SciAps X-555 portable XRF analyzer

Figure 4: X-ray shield

Figure 5: Testing setup with shields

3.4.2 XRD

The XRD analysis begins with the careful preparation of eclogite samples. The powdered sample is placed into a specialized metal plate containing a glass layer, which is secured using a clamp mechanism. A small empty space exists between the plate and the glass, where the finely ground sample is carefully inserted. Once the sample is placed, the glass and clamp are removed, and the sample holder is ready to be inserted into the XRD instrument (figure 6, figure 7).

Before initiating the XRD test, the following steps are undertaken to ensure proper operation:

- System Activation: The chiller is turned on first to maintain the system's optimal operating temperature. The XRD machine is powered on, the computer connected to the instrument is booted up. The software SmartLab Studio II is launched to interface with the instrument.
- Calibration Process: Calibration is a crucial step to ensure accurate diffraction measurements. The lead sheet is replaced with a copper sheet for calibration. Once calibration is complete, the system is reset to its initial configuration by reinstating the lead

sheet. This calibration step ensures that any instrumental deviations are corrected before measurement.

After calibration, the measurement process is initiated using the following setup:

- Measurement Mode: General measurement
- Radiation Source: Cu Kβ radiation
- Wavelength (λ): 1.3923 Å
- Scanning Range: 5° to 95° 2θ
- Scan Speed: 1° per second
- Total Measurement Time: 94 minutes

For data acquisition and interpretation, the XRD test is performed for 94 minutes, during which the diffraction pattern of the sample is recorded. The acquired data will be used to determine the mineralogical composition of the sample. The results will be cross-verified with XRF data and later analysed in combination with SEM imaging to provide a comprehensive understanding of the sample's mineral phases. Acquired data is provided in one graph in Figure 8 below.

Figure 6: Rigaku SmartLab XRD

Figure 7: Inside XRD, illustrating sample, and X-ray tube

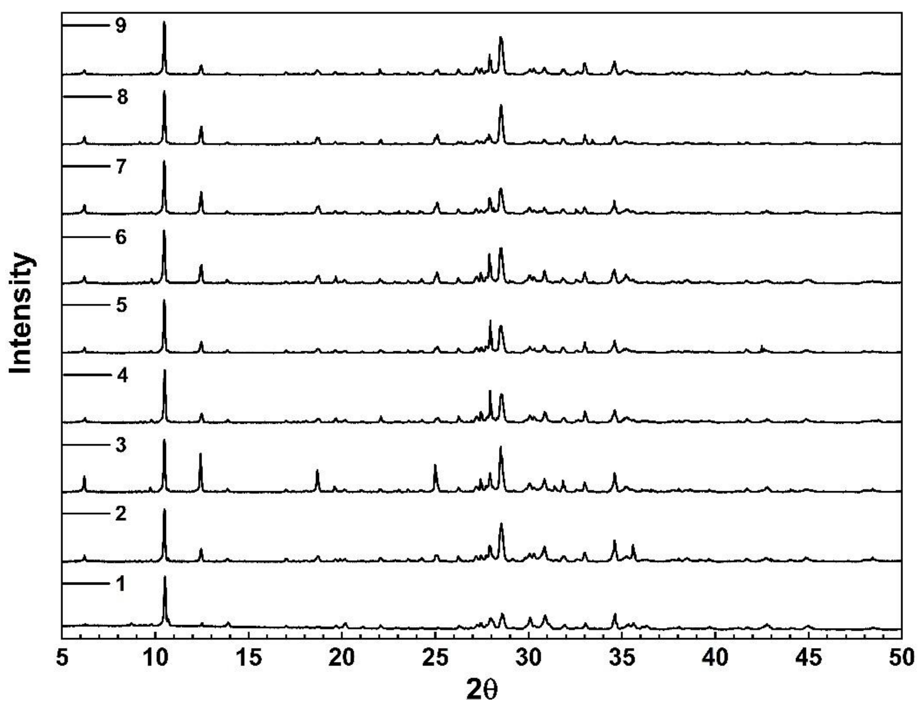

Figure 8: Acquired data by XRD from Eclo-1 to Eclo-9

3.4.3 Optical Microscopic

To ensure the accuracy and reliability of the XRD and XRF analyses, optical microscopy was extensively employed throughout the process. This method provided a direct visual verification of the mineralogical composition and grain characteristics, allowing for cross-validation of the obtained data.

Role of Optical Microscopy in the Analysis:

- Initial Sample Inspection before conducting XRD and XRF, optical microscopy was used to examine the morphology of the mineral grains, identifying potential impurities or inconsistencies in the sample preparation. This step helped ensure that the samples selected for XRD and XRF were representative of the bulk material.
- Cross-Validation of XRD Results, since XRD provides phase identification based on diffraction patterns, optical microscopy was used to visually confirm the presence of specific minerals such as rutile, ilmenite, garnet and quartz. By observing the grain boundaries, mineral textures, and cleavage patterns, the optical data served as a quality check against the XRD-generated phase composition.
- Result of XRF provides elemental composition data, but it does not distinguish between different mineral phases containing the same element. Optical microscopy allowed for the identification of mineral liberation, especially for Ti-bearing minerals such as rutile. The extent of rutile grain separation was assessed under the microscope, helping to interpret the XRF data more accurately.
- Monitoring During the Processing Steps including magnetic separation, and electrostatic separation, optical microscopy was repeatedly used to observe the changes in particle morphology and liberation characteristics. This ensured that separation processes were effective and that the obtained mineral concentrates were as pure as possible.
- Final Confirmation of Mineral Liberation of rutile was a critical parameter, as it directly influenced the effectiveness of downstream separation methods. Optical microscopy was used to evaluate how well rutile was liberated from the gangue minerals, which was essential for optimizing the separation efficiency.

3.5 Crushing and Milling

In order to obtain an adequate amount of material within the 106–53 µm size range for further separation experiments (Test 2, Test 3,and Test 4), a structured milling and sieving process was carried out. The initial particle size distribution revealed a deficiency of material in this range, necessitating the controlled reduction of additional coarse material. This was achieved through multiple steps of mechanical size reduction, followed by dry and wet sieving.

1. Sample Preparation and Sieving:

The available raw material was first subjected to the dry sieving with the following criteria:

- Particles larger than 0.5 mm were directed to the disc miller (figure 9)to reduce their size below 0.5 mm.
- The newly milled material was sieved again to separate <106 µm fraction.
- A wet sieving method was employed to isolate the <53 µm fraction, ensuring that only appropriately sized material was retained for further processing.

The necessity of a wet sieving step was dictated by the tendency of fine particles to agglomerate and adhere to sieve meshes during dry sieving, leading to inefficiencies in classification.

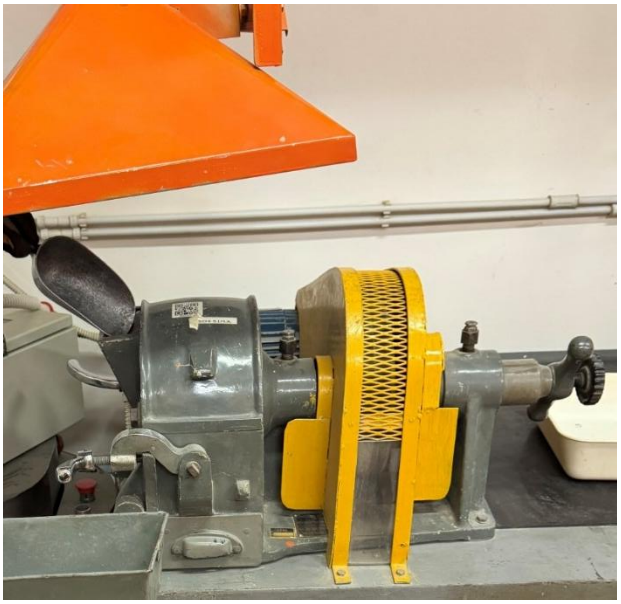

Figure 9: Disc miller

2. Bar Milling:

To optimize the quantity of material in the 53-106 µm range, a bar milling (figure 10) technique was employed. This involved precise time-controlled grinding with periodic sieving and weighing to track the size reduction kinetics.

Milling procedure:

- Initial Milling Setup: The milling cylinder was loaded with 10 milling bars, ensuring efficient breakage while preventing excessive wear on the equipment.
- Two-Minute Milling Cycles: The milling process was conducted in two-minute intervals. After each cycle, the material was completely removed, sieved, and weighed before being returned to the mill. This approach was essential for understanding the fragmentation behaviour of eclogite over time.
- Progressive Size Reduction Monitoring: After 30 minutes of cumulative milling, the fraction smaller than 106 µm was extracted to prevent excessive particle breakage. A subsequent wet sieving step was conducted to isolate the <53 µm fraction.
- Extended Milling Cycles: The remaining material underwent additional 4-minute milling cycles, followed by sieving and weighing. This ensured that the liberation of minerals continued in a controlled manner.
- Final Milling Phase: After 38 minutes of total milling time, another <53 µm fraction was extracted, and a final 4-minute milling session was conducted to complete the process.

Figure 10: Bar/Ball miller

3. Drying and storage:

Once the desired fractions were obtained, they were subjected to a 24-hour drying process in an oven at 70°C to eliminate any residual moisture.

3.6 Separation

3.6.1 Optimization of Wet magnetic separation parameters

Magnetic separation was conducted in both wet and dry modes across multiple intensity levels to optimize the separation efficiency of critical raw materials, particularly focusing on rutile-bearing eclogite samples.

1. Wet Magnetic Separation (WMS)

The wet magnetic separation (figure 11) process was designed as a three-stage sequential separation to progressively refine the non-magnetic fraction for subsequent electrostatic separation.

The process started with 123.88 grams of milled material suspended in water. Three different magnetic field intensities were applied sequentially with a double check using magnetometer (figure 12):

- 420 mT (milliTesla)
- 700 mT
- 800 mT

In each stage the magnetic fraction was captured on iron plates inside the separator. The non-magnetic fraction was passed on to the next stage with increased magnetic intensity. The collected magnetic portions from each stage were separately filtered and dried.

All separated fractions were filtered to remove water and then placed in an oven at 200°C for 24 hours to ensure complete drying.

The samples were categorized into:

- M1 (420 mT magnetic fraction)
- M2 (700 mT magnetic fraction)
- M3 (800 mT magnetic fraction)
- NM (final non-magnetic fraction)

Each fraction underwent XRF analysis to determine elemental composition and microscopic analysis to assess mineral liberation.

Figure 11: Wet magnetic separator

Figure 12: Magnetometer used for magnetic intensity modification

2. Dry Magnetic Separation (DMS)

Dry magnetic separation (figure 13) was tested with an alternative approach using a two-stage stepwise intensity increase. The process was conducted using a dry magnetic separator in two steps, 250 mT initial separation. The first stage aimed to remove any highly magnetic minerals. Re-separation with 1.1 T (Tesla), the non-magnetic fraction from the first stage was subjected to a stronger field to remove weakly magnetic phases. The final non-magnetic fraction was collected for further processing.

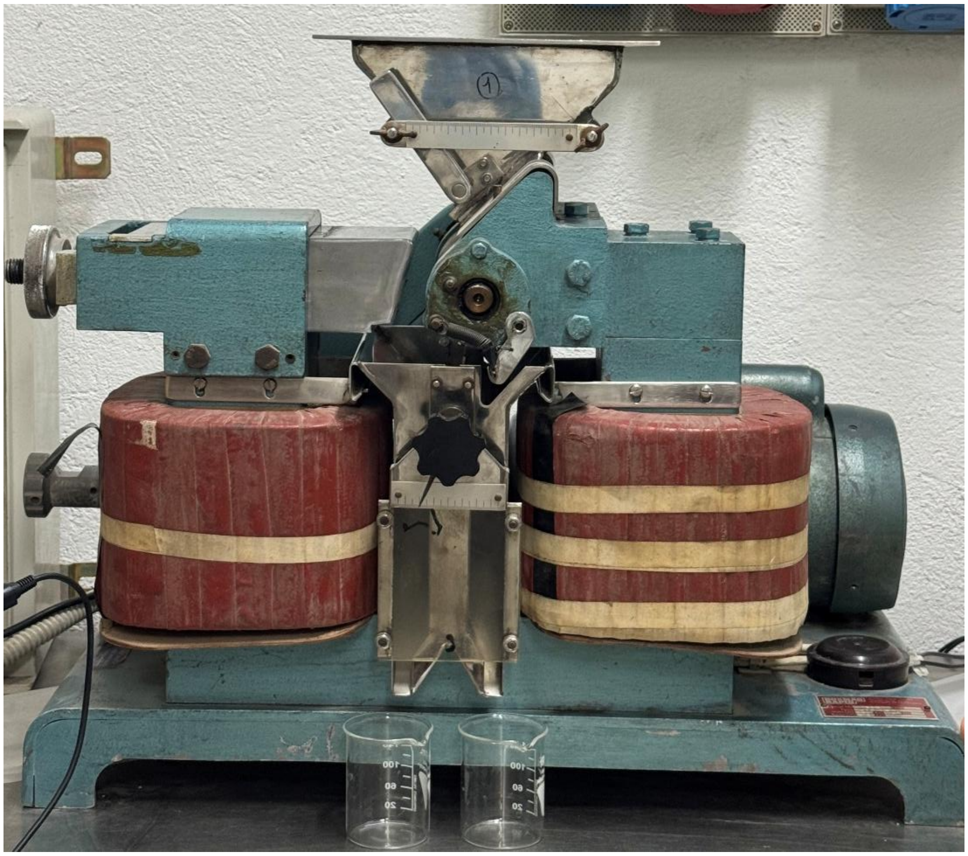

Figure 13: Dry magnetic separator

3.6.2 Optimization of Electrostatic separation parameters

The initial material mass of 333 g was first divided using a riffle splitter into three samples: one half and two quarters. The main sample was returned to the oven to be heated above 100°C, while the two smaller samples were processed separately under different temperature conditions. One of the samples remained at room temperature (21°C), while the other was returned to the oven.

Each test was performed using a setup that included an electrostatic separator, where various parameters such as drum speed, feeder vibration, temperature, and voltage were varied. The results of the separation were analysed using optical microscopy, and three of the better candidates based on microscopy analysis were analysed using XRF and will be explained in chapter 4. The procedure was carried out as follows:

1. **Test 1:** Room temperature (21°C), drum speed of 30 Hz, feeder vibration of 15 Hz, voltage of 28 kV.
2. **Test 2:** Room temperature (21°C), drum speed of 30 Hz, feeder vibration of 15 Hz, voltage reduced to 25 kV.
3. **Test 3:** Room temperature (21°C), drum speed of 30 Hz, feeder vibration of 15 Hz, voltage maintained at 25 kV, but polarity of the drum was reversed from negative to positive.
4. **Test 4:** Higher temperature (100°C), drum speed of 30 Hz, feeder vibration of 15 Hz, voltage decreased to 20 kV, drum polarity set to negative.
5. **Test 5:** Higher temperature (100°C), drum speed of 30 Hz, feeder vibration of 15 Hz, voltage decreased to 20 kV.
6. **Test 6:** Higher temperature (100°C), drum speed of 30 Hz, feeder vibration of 15 Hz, voltage maintained at 20 kV, with manual triboelectric charging applied for 60 seconds before starting the separation inside PTFE.
7. **Test 7:** Higher temperature (100°C), drum speed of 30 Hz, feeder vibration of 15 Hz, voltage increased to 28 kV, with manual triboelectric charging applied for 60 seconds before starting the separation inside PTFE.

The results of each test were carefully analysed using optical microscope, and the most promising candidates underwent further testing with XRF analysis (test 2, test 6, test 7).

3.6.3 Test 1

All materials from Test 1 were recombined and then transferred to the oven, where they were heated to a temperature of 125°C. A small portion of the sample was retained for XRF analysis, while the remaining material was divided into three 85 g portions for controlling the temperature for electrostatic separation.

Prior to electrostatic separation, each portion had a manual triboelectric charging process for 60 seconds in a PTFE container. The charged material was then fed into the electrostatic separator, operating at a drum speed of 30 Hz, vibration frequency of 15 Hz, and the voltage of 28 kV. After three sequential feed cycles, the non-conductive fraction was collected, weighed, and analysed using optical microscope and XRF. Simultaneously, the conductive and mixed fractions were also weighed.

The mixed fraction was returned into the oven and reached to 125°C before undergoing a second round of electrostatic separation under identical conditions, including the 60-second triboelectric charging step. Following this process, the newly obtained non-conductive and mixed fractions were weighed and subjected to optical microscope and XRF analysis.

The conductive fractions from the first and second separations were then combined and processed through a dry magnetic separator an intensity of 500 mT. The resulting magnetic and non-magnetic fractions were collected, weighed, and analysed using both microscopic examination and XRF.

3.6.4 Test 2

The processed materials from Test 2 were mixed, and a total of 261.5 g of the sample was subjected to dry magnetic separation at an intensity of 250 mT. The magnetic fraction was collected, weighed, and analysed using XRF. The non-magnetic fraction was introduced as a slurry into a wet magnetic separator, operating at 420 mT.

Following this stage, the magnetic fraction underwent filtration and was then transferred to a dryer oven at 70°C. After 24 hours, it was weighed and analysed via XRF. Meanwhile, the non-magnetic fraction proceeded directly to the next separation stage, where it was introduce as a slurry and subjected to wet magnetic separation at an increased field strength of 700 mT. The newly obtained magnetic fraction was filtered, heated in the dryer oven at 70°C for 24 hours, and subsequently weighed and analysed using XRF.

The non-magnetic fraction from this stage was sieved with a wet sieving process using a 53 μm mesh screen to remove finer particles that could interfere with subsequent electrostatic separation. The retained fraction was then filtered and put in an oven at 125°C for 24 hours.

Finally, the dried material was introduced into the electrostatic separation system under the same operating conditions as Test 3. The non-conductive, mixed, and conductive fractions were collected, weighed, and analyzed using XRF.

3.7 Overview

In the procedure of optimizing sustainable mineral processing techniques, a series of systematic sampling, screening, and characterization methods were employed to ensure representative sample selection, exact particle classification, and accurate compositional analysis. Two distinct sampling methods (Coning and Quartering and Riffle Splitting) were evaluated.

For classification of the processed material, a sieving method was applied using a range of mesh sizes for wet and dry, ensuring precise separation of particles for upcoming tests. The dry sieving process was conducted under controlled conditions using an electromechanical shaker while implementing dust control measures to maintain a safe working environment.

For material characterization, X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) analysis were conducted to determine the elemental and mineralogical composition of the processed samples. Due to limitation of samples volume, manual milling was performed to achieve a particle size below 30 µm, optimizing homogeneity and analytical accuracy. To ensure precision and reproducibility, XRF analysis was carried out using a portable XRF analyser with strict calibration and testing protocols. In addition, XRD analysis was executed following a sample preparation and calibration process to obtain high-resolution diffraction patterns.

Optical microscopy played a critical role in cross validating the results obtained from XRD and XRF by providing direct visualization. This method was essential for monitoring separation efficiency, detecting potential impurities, and ensuring the effectiveness of the processing at each step.

Through the integration of these sampling, screening, and characterization techniques, the study established a solid framework for evaluating mineral separation efficiency. These methods not only ensured the reliability of experimental results, but also contributed to the development of more sustainable mineral processing strategies, particularly in the recovery of critical raw materials such as rutile from eclogite.

4. Results and Discussion

4.1 Introduction

This chapter presents the results gained from the experimental procedures described in Chapter 3, focusing on the characterization and processing of eclogitic rocks for sustainable rutile extraction. The analysis provides insights into the efficiency of different separation techniques and their potential for increasing the recovery of titanium dioxide while minimizing environmental impact. The discussion integrates the findings from granulometric analysis, mineralogical and chemical characterization, crushing and milling performance, and the outcomes of various separation methods.

The results presented in this chapter aim to address key research questions concerning the feasibility of dry separation methods as an alternative to conventional chemical-based beneficiation. The findings contribute to a broader vision of how physical separation techniques can optimize rutile recovery while reducing reliance on energy-intensive and environmentally hazardous methods.

4.2 Screening

The granulometric analysis provides a detailed assessment of particle size distribution within the processed eclogite samples. The results, summarized in Table 1, indicate the range of particle sizes. The cumulative passing percentages, shown in Figure 14, illustrates D80, D50, and D20.

Also the mass balance of primary sieving, is illustrated in figure 15. Notably, the Eclo-3 and Eclo-4 fractions represent the highest retained mass, suggesting a need for additional grinding to enhance liberation of rutile. The Eclo-9 fraction (<0.053 mm), is 6.33% of total mass, introduces challenges in separation due to its fine nature and potential for entrainment losses, in fact, we cannot process it.

The size distribution results indicate that coarser fractions (Eclo-1 to Eclo-3) retain a significant portion of accessory minerals, potentially reducing the effectiveness of dry separation techniques. On the other side, finer fractions (Eclo-6 to Eclo-8) may require additional handling precautions to avoid overgrinding, which could impact electrostatic separation efficiency.

From a practical standpoint, achieving an optimal balance in particle size is crucial to maximize rutile recovery. The high proportion of Eclo-3 (49.5% retained) and Eclo-4 (30.95% retained) suggests that controlled milling strategies could be implemented to further liberate rutile while preventing over generation of ultra-fine particles.

Table 1:Result of the primary dry sieving

Particle name	**Particle size(Retaining) (mm)**	**weight (g)**	**Comulative Passing**	**Comulative retained**
Eclo-1	6.7	4.1	99.66%	0.34%
Eclo-2	4	79.69	93.15%	6.85%
Eclo-3	2	533.7	49.50%	50.50%
Eclo-4	1	226.85	30.95%	69.05%
Eclo-5	0.5	114.18	21.61%	78.39%
Eclo-6	0.212	80.36	15.04%	84.96%
Eclo-7	0.106	58.35	10.27%	89.73%
Eclo-8	0.053	48.25	6.33%	93.67%
Eclo-9	0.001	77.35	0.00%	100.00%
Total		1222.83		

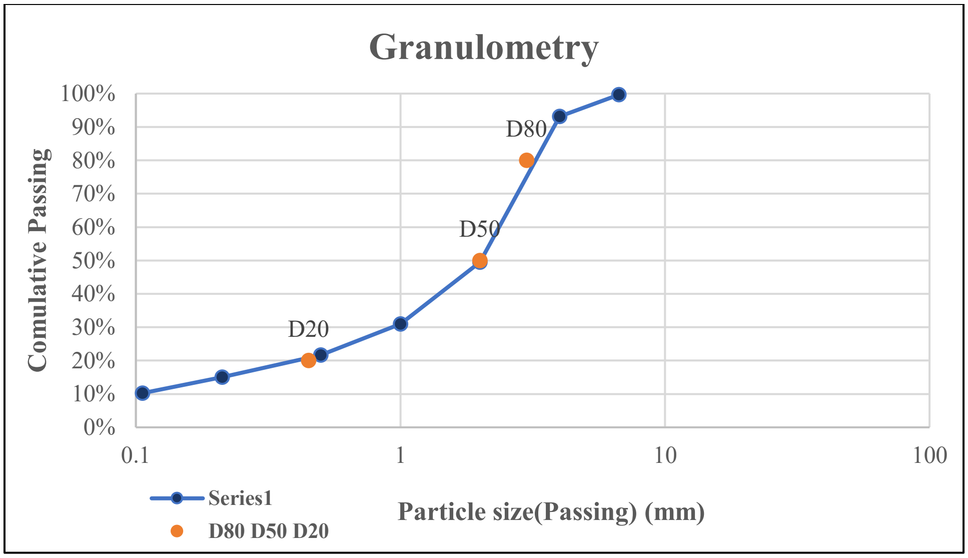

Figure 14:Granulometry of primary sieving

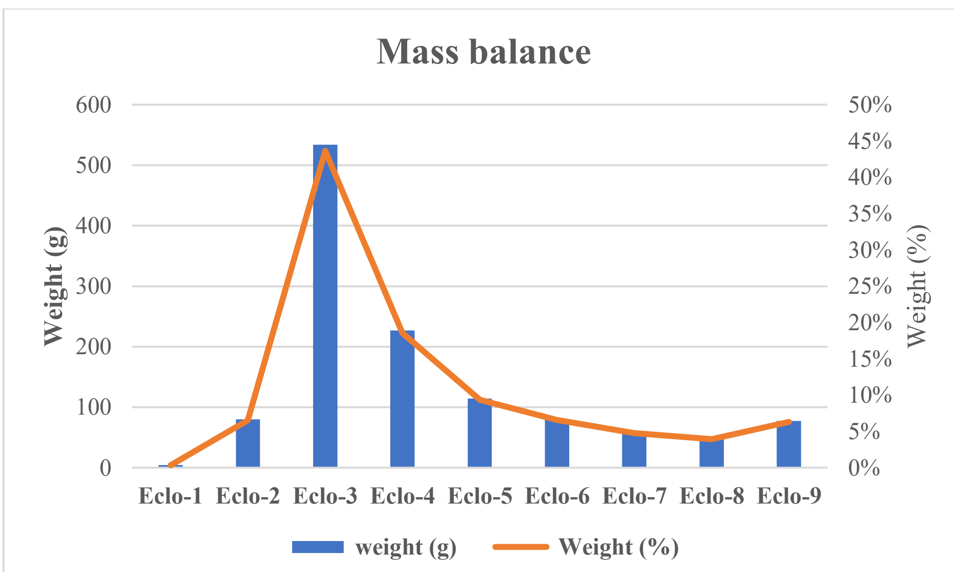

Figure 15: Mass balance of the primary sieving

4.3 Characterization

The X-ray Fluorescence (XRF) analysis provided a comprehensive overview of the elemental composition of the Eclo-1 to Eclo-9. The results, presented in Table 3 and Figure 16, highlight that titanium (Ti), iron (Fe), magnesium (Mg), and aluminium (Al) are the predominant elements. The

Ti grade varies across different granulometric fractions, with Eclo-8 showing the highest Ti content (3.70%), indicating a stronger concentration of rutile in finer fractions.

Comparison of XRF and XRD results (Table 2) confirms that rutile and ilmenite are Ti-bearing minerals. Rutile being more abundant in finer fractions (Eclo-8 and Eclo-9), while coarser fractions (Eclo-3 to Eclo-5) shows a more mixed distribution with ilmenite and other accessory minerals.

X-ray Diffraction (XRD) analysis identified main mineral phases within the eclogite samples. The primary phases detected include rutile, ilmenite, clinochlore, albite, glaucophane, and quartz. The Eclo-8 fraction exhibits the highest rutile content (5.0%), supporting the XRF findings and indicating that finer fractions are better for rutile recovery.

In contrast, coarser fractions (Eclo-1 to Eclo-4) contain higher concentrations of silicate minerals such as clinochlore and glaucophane. The presence of almandine, and garnet in Eclo-3 and Eclo-4 suggests a need for additional liberation through further grinding.

The optical microscopy analysis was utilized to cross-validate the XRD and XRF findings, providing insight into mineral grain distribution and liberation characteristics. Rutile grains are well-liberated (figure 17, figure 18) in finer fractions (Eclo-8 and Eclo-9), enhancing the potential for effective electrostatic separation.

The characterization results provide key insights into the expected performance of separation methods:

- Magnetic Separation: More effective in removing iron-bearing minerals such as ilmenite and garnet from coarser fractions (Eclo-3 to Eclo-5).
- Electrostatic Separation: Expected to be highly efficient for rutile-rich finer fractions (Eclo-8 and Eclo-9), as these show better liberation and lower iron content.

In conclusion, the XRF, XRD, and optical microscopy analysis confirm that rutile concentration is highest in finer fractions, due to this and researches, target size for further process was chosen the same as Elco-8, while coarser fractions require additional grinding for better liberation.

The important point should be mentioned is that XRD recognition threshold is limited to 5% of the volume, therefor, when focusing on the data acquired and interpreted by XRD, must be carefull.

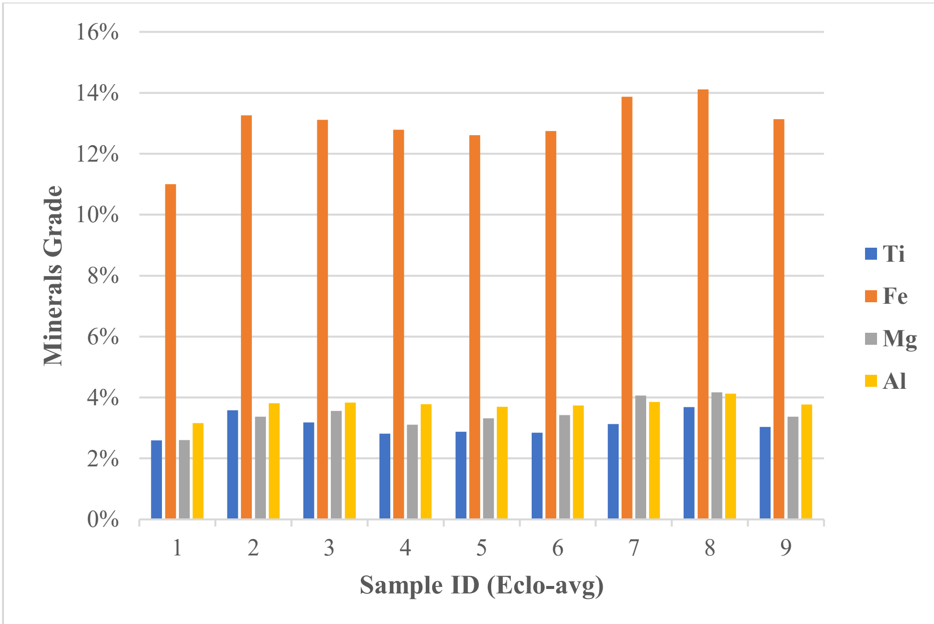

Figure 16: Important elements present in Eclo samples detected by XRF

Table 2: Comparison of XRF and XRD results based on Rutile and Ilmenite contents

Sample Name	Total Ti Grade (%) (XRF)	Total Ti Grade (%) (XRD)	Rutile Grade (%) (XRD)	Ilmenite grade (%) (XRD)
Eclo-1	2.60	3.30	4.1	2.8
Eclo-2	3.60	2.91	1	1.7
Eclo-3	3.20	2.88	3.8	2
Eclo-4	2.80	1.03	1.29	0.85
Eclo-5	2.90	1.95	2	2.5
Eclo-6	2.90	0.72	1.2	0
Eclo-7	3.10	1.59	1.2	2.9
Eclo-8	3.70	3.00	5	0
Eclo-9	3.00	0.96	1.6	0

Table 3: Elements found in each sample by XRD

Mineral name	Eclo-1 (%)	Eclo-2 (%)	Eclo-3 (%)	Eclo-4 (%)	Eclo-5 (%)	Eclo-6 (%)	Eclo-7 (%)	Eclo-8 (%)	Eclo-9 (%)
Rutile	4.1	4	3.8	1.29	2	1.2	1.2	5	1.6
Ilmenite	2.8	1.7	2	0.85	2.5	-	2.9	-	-
Paragonite	7	-	-	-	-	6	-	7	-
Clinochlorite	5.4	33	9	32.6	9	20	4.6	16	8
Almandine	13.9	8	-	-	-	-	-	-	-
Kosmochlore	12.6	10	7.8	9.1	10	19	11	-	-
Albite	14.6	12	18	8	22	9	16	3	7
Glaucophane	8.6	4	-	-	9	-	-	18	16
Quartz	3	0.6	-	-	-	6	-	-	9
Hornblend	28	22	35	32.8	22	33	44	37	26
Muscovite	-	2	17	1.8	4	-	-	-	-
Constedtite	-	2	-	-	-	-	-	-	-
Spessartine	-	-	7.2	-	7	5.4	10.3	-	8
Titanite	-	-	-	3.5	-	-	-	-	-
Clinozoisite	-	-	-	9.9	12	8	11	13	15

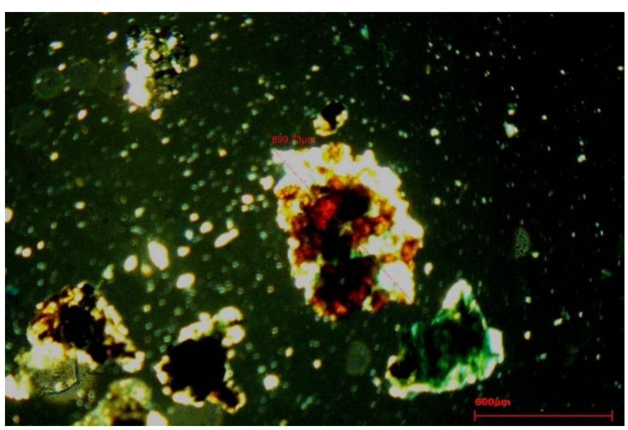

Figure 17: Microscopic view of Eclo-3 Rutile liberation

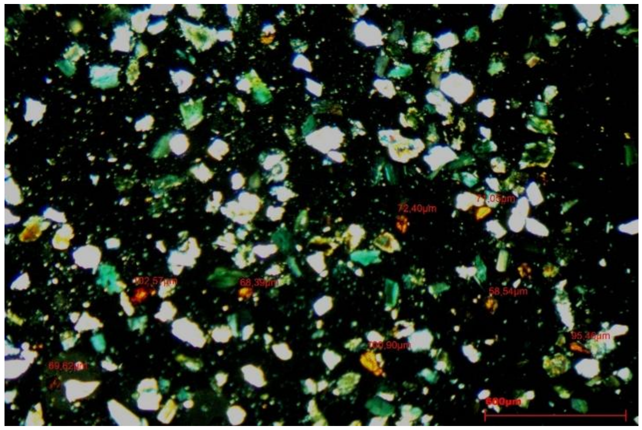

Figure 18: Microscopic view of Eclo-8 Rutile liberation

4.4 Crushing and milling

As shown in the results (Table 4, and figure 19), even after 30 minutes of bar milling, particles larger than 106 µm still constituted a significant portion of the sample. Following an analysis of this behaviour, the portion of the material smaller than 106 µm was removed to enhance milling efficiency, which proved to be highly effective. This adjustment demonstrated that optimizing milling conditions by controlling material removal significantly improves processing outcomes.

Based on the acquired data, it was determined that the optimal continuous milling duration without interruption and material discharge could be inferred from this stage. This data was also applied in subsequent milling operations, where after 10 minutes, particles smaller than 106 µm were extracted. Then, within an additional 5 minutes, the remaining material reached the desired size. This approach significantly reduced the percentage of material loss due to over milling, achieving a more controlled and efficient milling process with energy saving.

Table 4: Result of bar milling

Time (min)	Mass (g) (>106 µm class)	Mass (g) (>53 µm class)	Mass (g) (<53 µm class)	Total mass (g)
0	531.1	0	0	531.1
2	512.54	15.13	2.59	530.26
4	487.54	30.78	11.72	530.04
6	459.73	47.5	22.31	529.54
8	430.36	52.74	45.68	528.78
10	410.16	76.68	41.23	528.07
14	359.55	92.56	74.43	526.54
18	332.17	108.91	84.81	525.17
22	314.32	138.19	72.2	524.17
26	271.46	151.3	100.98	523.74
30	231.71	177.94	113.63	523.28

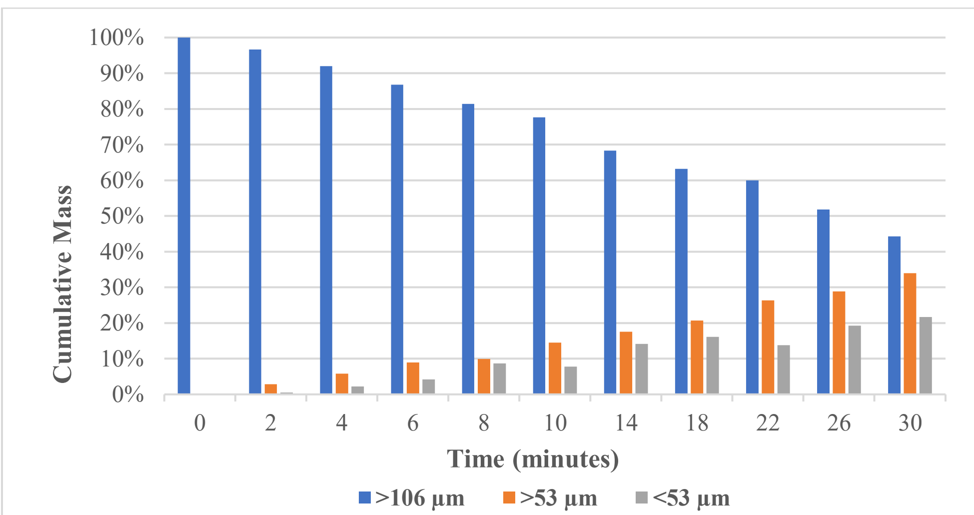

Figure 19: Fragmentation behaviour of eclogite over time by bar milling

After the milling process reached the desired particle size, a noticeable difference in color was observed between the milled material and the size matched fraction obtained from the initial sieving. This variation in coloration caused further investigation. XRF analysis of both samples revealed (figure 20) that the darker color of the sieved, non-milled material could be attributed to a higher presence of silicon (indicating a greater quartz content) and a lower concentration of iron.

This suggests that the color difference may be a direct consequence of the compositional variation, particularly the higher presence of quartz and lower iron content in the non-milled fraction.

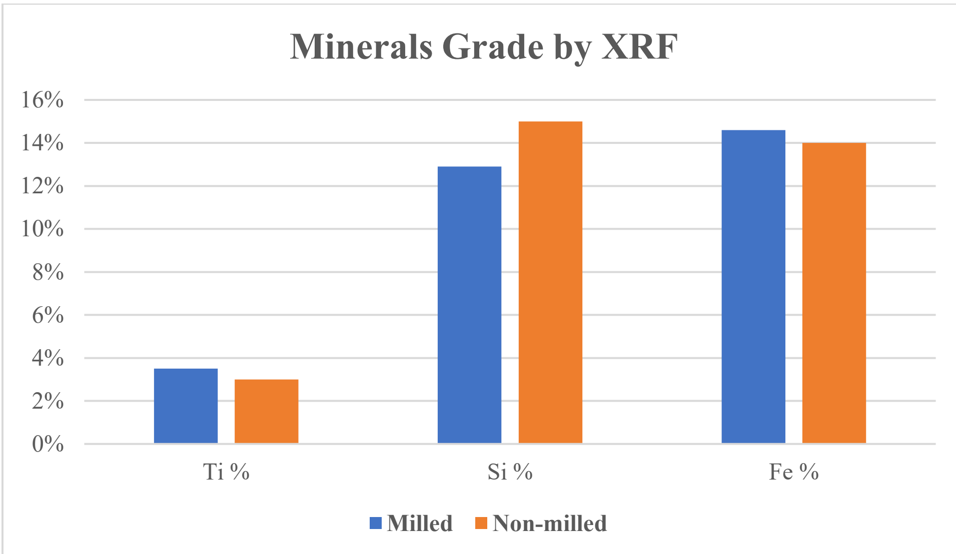

Figure 20: Result of XRF showing the difference of milled and non-milled Eclo in the same size

4.5 Separation

4.5.1 Optimization of Wet magnetic separation parameters

The results (Table 5, figure 21) indicate that wet magnetic separation cannot be considered as an independent separation technique. The findings suggest that magnetic separation requires a complementary approach to achieve effective mineral separation. This was conducted to compare its efficiency with Electrostatic separation on Eclo samples. In the wet magnetic separation test, magnetic field strengths of 420 mT and 700 mT demonstrated the highest efficiency in separating the target minerals. Based on these results, medium-intensity magnetic separation will be considered as the optimal setup for future tests, ensuring an effective balance between separation efficiency and material recovery.

Table 5: Result of Magnetic separation

Product	Mass (g)	Yield (%)	Ti Grade(%)	R (%)
Feed	124.44	-	3.14%	-
M1	39.23	31.5%	3.11%	31.17%
M2	40.21	32.3%	2.98%	30.62%
M3	19.46	15.6%	3.13%	15.57%
NM	25.54	20.5%	4.19%	27.34%
Total M	98.9	79.5%		

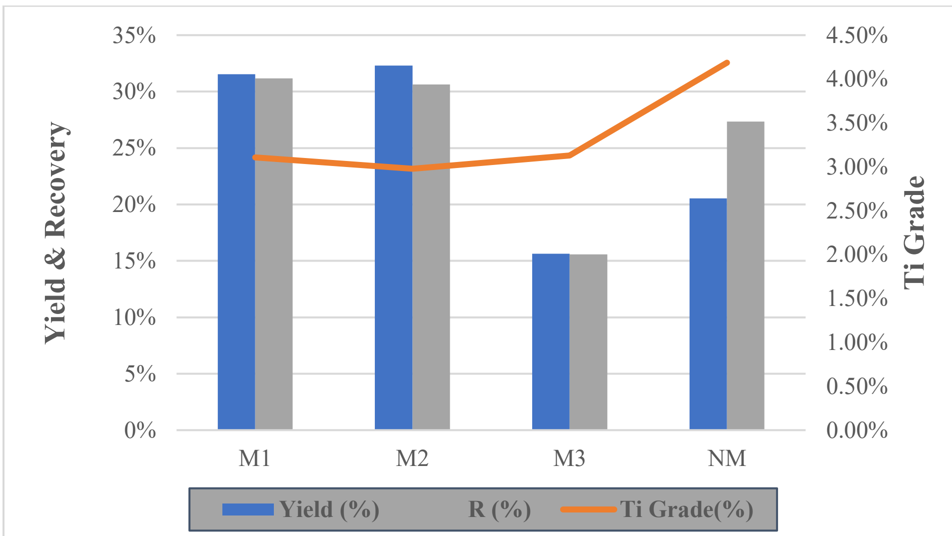

Figure 21: Result of wet magnetic separation

4.5.2 Optimization of Electrostatic separation parameters

Electrostatic separation result tested using optical microscopy, and the observations were systematically recorded to determine the optimal setup for processing the available material. Based on the microscopic analysis, the most effective separation results were obtained at a temperature of 100°C combined with manual triboelectric charging for 60 seconds in PTFE. Additionally, a voltage of 28 kV was selected for the main tests due to its superior yield, which allowed for multi stage processing, thereby enhancing the overall efficiency of the separation process. Result of XRF of candidates are also provided below in figure 22, figure 23, and figure 24.

Sample name	Test 1 mass (g)	Test 1 mass (%)	Test 2 mass (g)	Test 2 mass (%)	Test 3 mass (g)	Test 3 mass (%)	Test 5 mass (g)	Test 5 mass (%)	Test 6 mass (g)	Test 6 mass (%)	Test 7 mass (g)	Test 7 mass (%)
Conductive	0.12	1.70%	0.47	4.32%	1.15	14.82%	0.31	1.30%	3.34	5.55%	4.44	18.67%
Mixed	3.6	50.92%	4.54	41.69%	6.57	84.66%	7.64	32.07%	44.82	74.51%	15.35	64.55%
Non-conducitive	3.35	47.38%	5.88	53.99%	0.04	0.52%	15.87	66.62%	11.99	19.93%	3.99	16.78%

Table 6:Mass balance of Electrostatic optimization tests

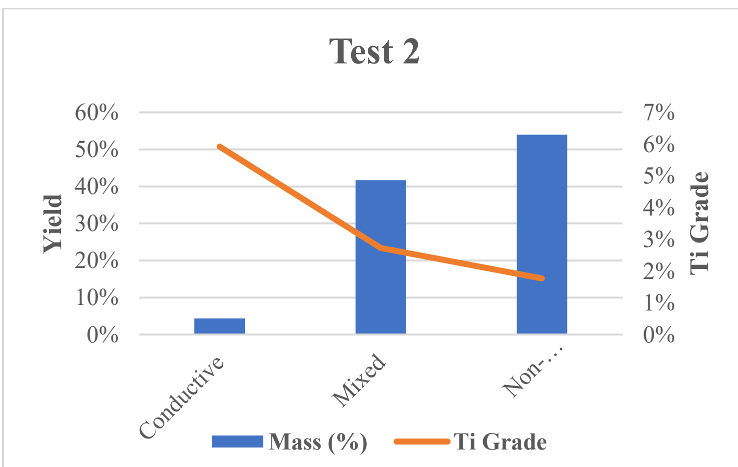

Figure 22: Yield vs Ti grade by XRF

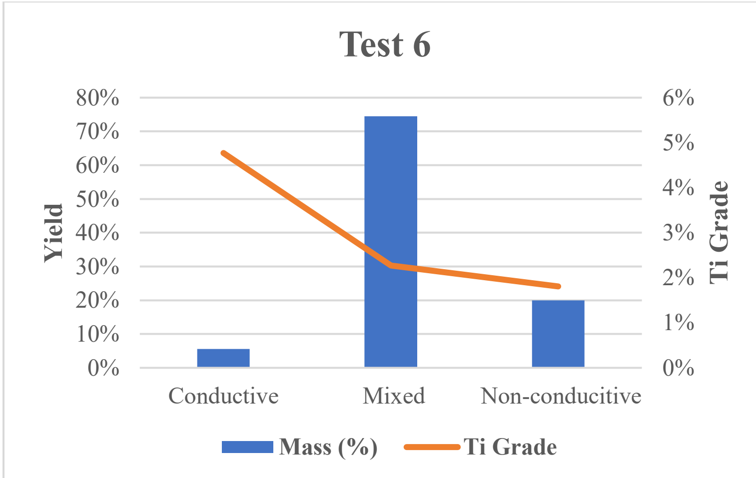

Figure 23:Yield vs Ti grade by XRF

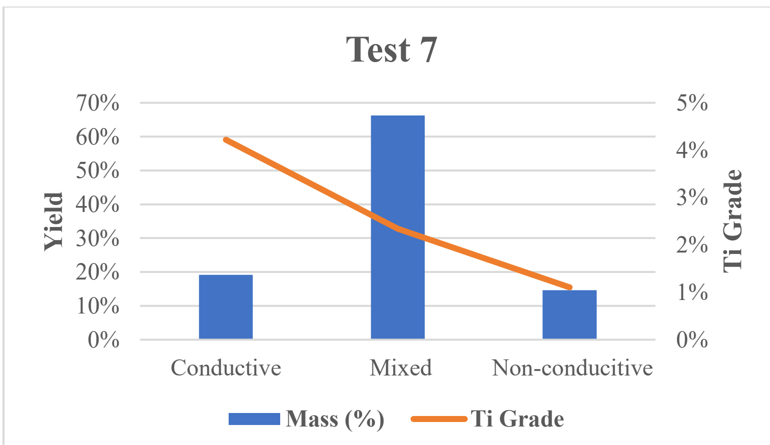

Figure 24:Yield vs Ti grade by XRF

4.5.3 Test 1

Test 1 was described in Chapter 3, and its results were determined based on mass balance and titanium grade obtained through XRF analysis for each fraction in Table 7 and Figure 28. In addition, some optical microscopic pictures are provided (figure 25, figure 26, and figure 27) in order to illustrate better the results. The whole process of Test 1 is provided by Figure 29 as a flowchart. Although, in Non-magnetic the Ti grade is the highest, due to the yield, recovery is not high enough. Based on the microscopic images, no significant differences in separation efficiency are observed across different sections. This indicates a relatively uniform distribution of minerals, suggesting that the applied separation techniques do not exhibit perfectly substantial spatial variability in performance.

Figure 25: Conductive 1 microscopic picture

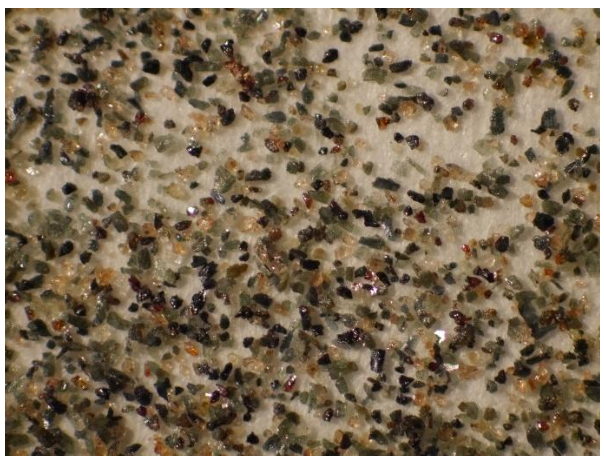

Figure 26: 500mT Magnetic microscopic picture

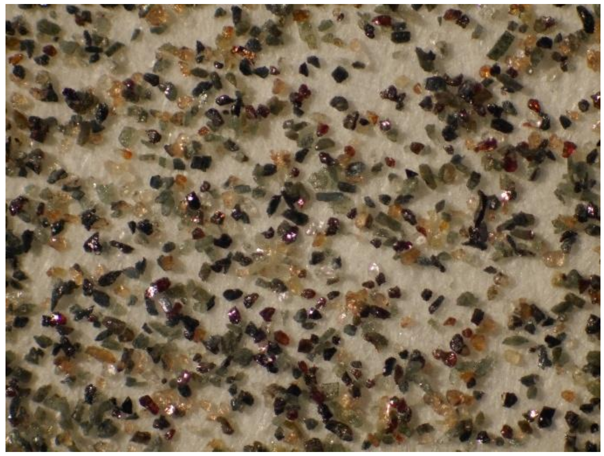

Figure 27: 500mT Non-magnetic microscopic picture

Table 7: Result of Test 1

Sample ID	**Mass (g)**	**Ti grade**	**Recovery**	**Mass**
Non-Mag	5.35	7.60%	4.43%	2.10%
Mag	44.5	5.36%	25.98%	17.45%
Mix2	92.84	3.33%	33.69%	36.41%
Non-Cond2	30.81	3.33%	11.19%	12.08%
Non-Cond1	71.3	2.36%	18.34%	27.96%
Feed	255	3.60%		

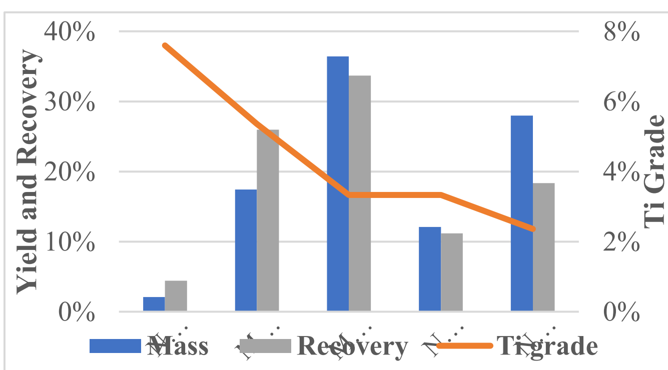

Figure 28: Result of Test 1

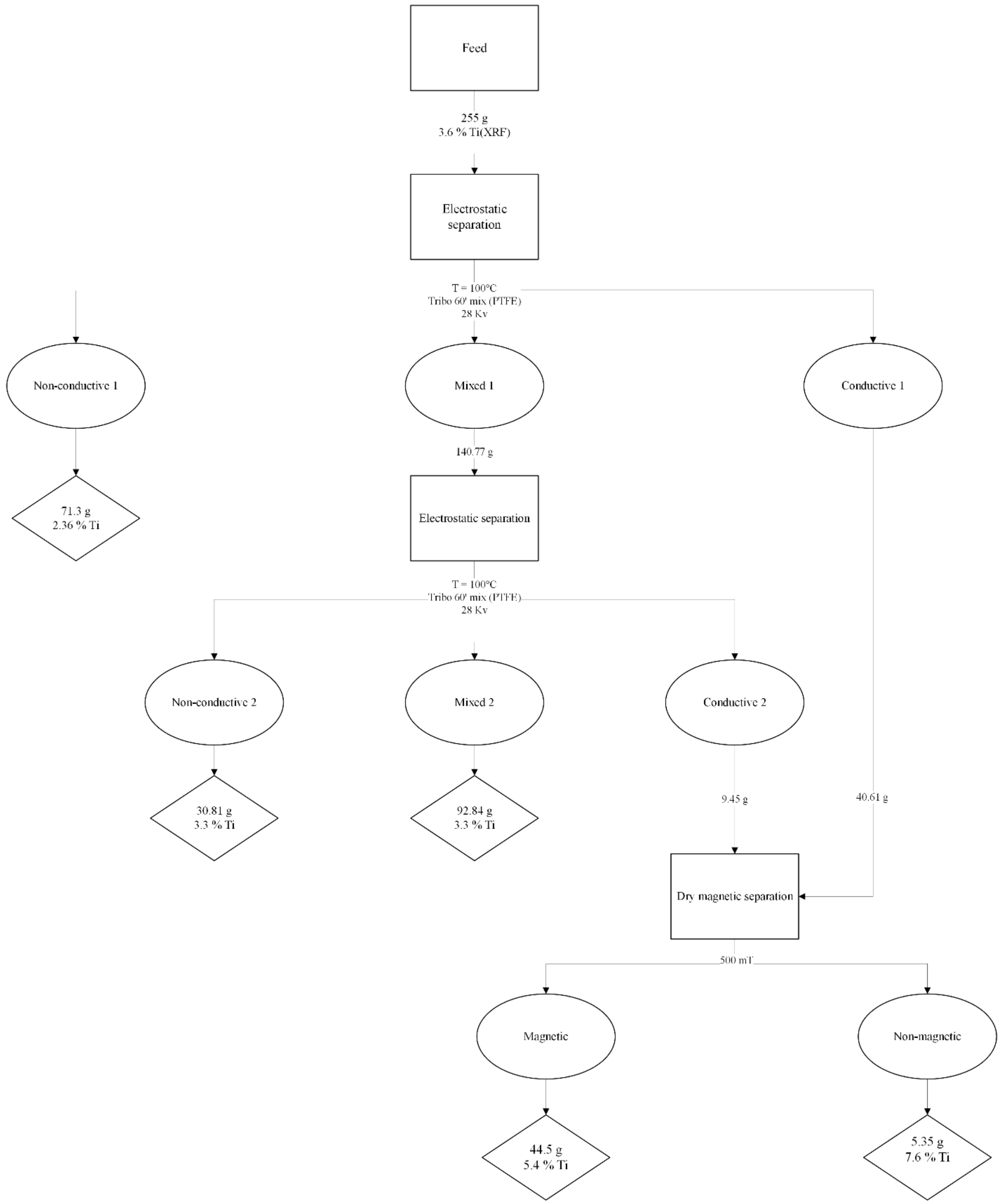

Figure 29: Flowchart of Test1 including mass balance and XRF result

4.5.4 Test 2

Based on the methodology outlined in Chapter 3, was conducted to assess the effectiveness of electrostatic separation for rutile recovery. The results obtained from this test (Table 8, Figure 30) are presented in the form of a flowchart (Figure 31), summarizing the outcomes of different separation conditions and their impact on mineral recovery efficiency. In addition, optical microscopic picture of all mentioned material is included by Figure 32 to Figure 37. Clearly, in Magnetic 1, there is a small portion of rutile, and in Magnetic 2 and Magnetic 3, rutile has diminished significantly, while they have ilmenite. Conductive and Mixed include the most rutile and in Non-conductive there is a few amount of rutile.

Table 8: Result of Test2

Sample ID	Mass (g)	Mass	Ti grade	Recovery
Mag 1	136.21	52.09%	3.45%	49.92%
Mag2	33.46	12.80%	3.94%	14.02%
Mag 3	33.52	12.82%	3.34%	11.89%
Cond	14.84	5.67%	6.09%	9.60%
Mix	11.89	4.55%	6.99%	8.82%
Non-Cond	23.2	8.87%	3.70%	9.11%
Feed	261.5		3.60%	

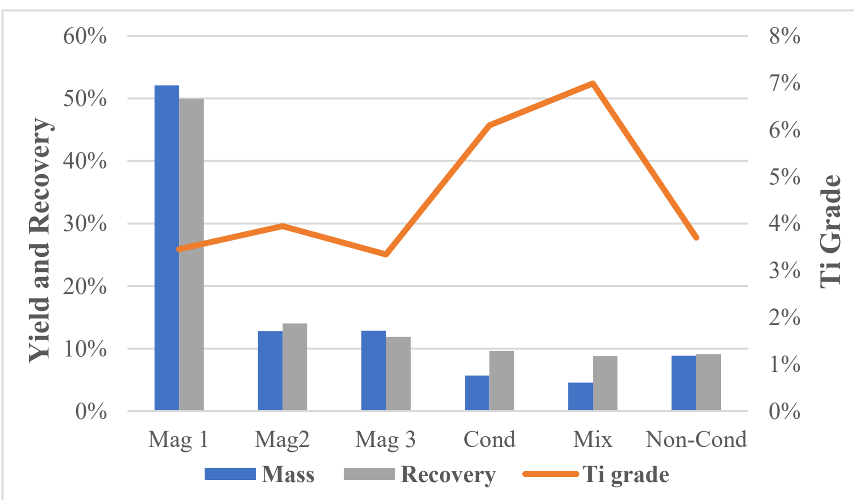

Figure 30: Result of Test 2

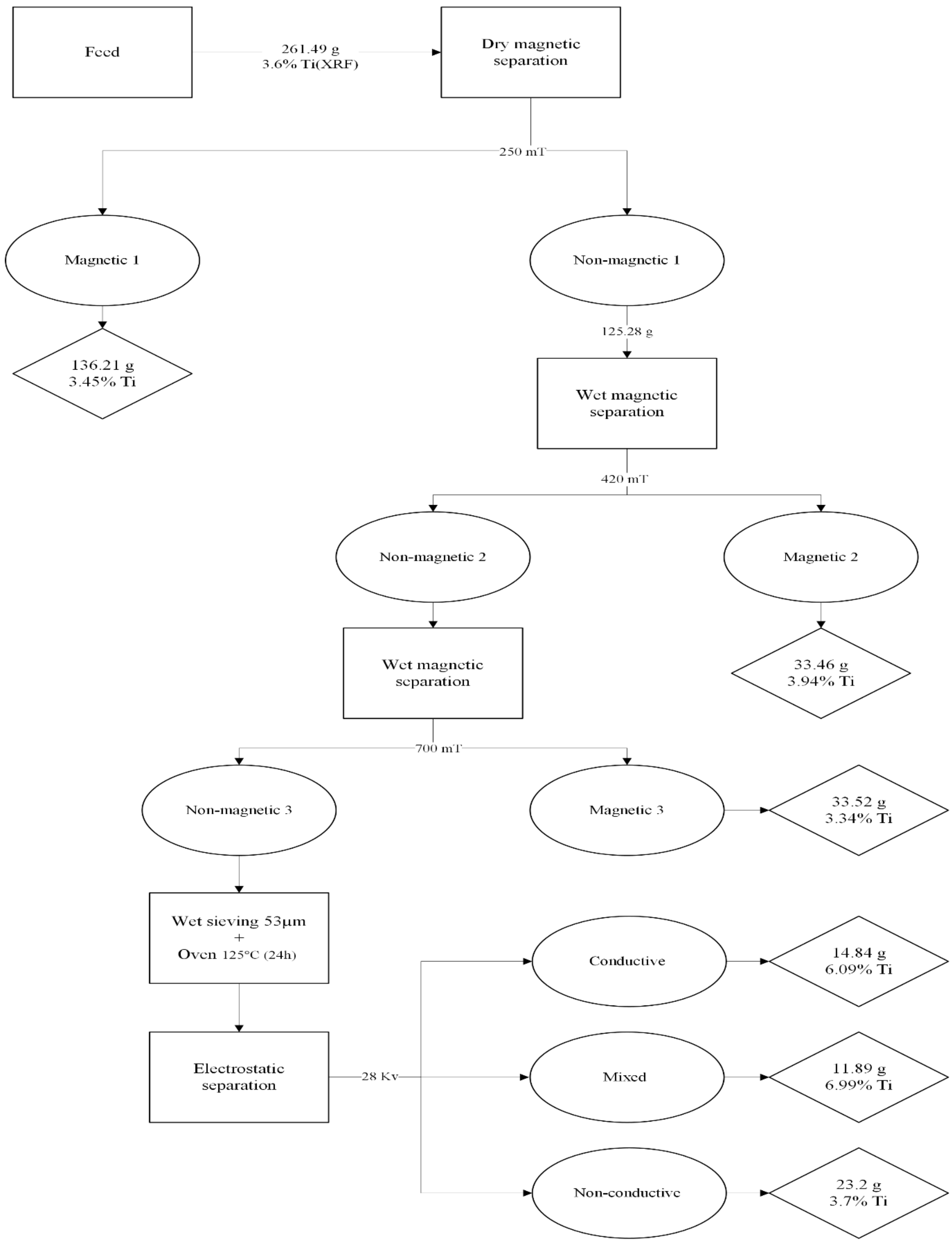

Figure 31: Flowchart of Test2 including mass balance and XRF result

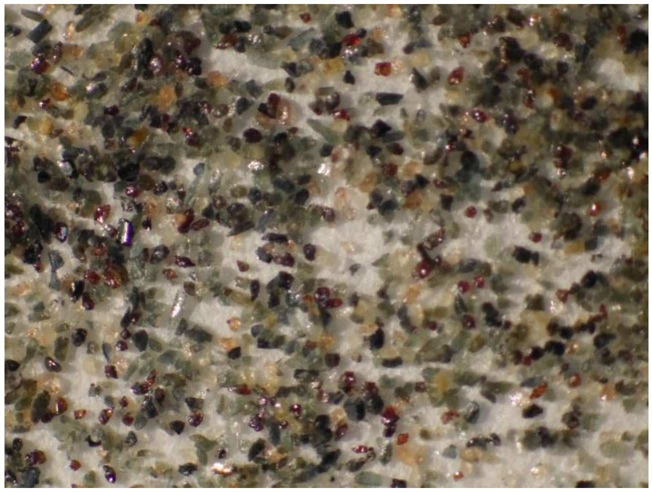

Figure 32: Optical microscopy picture of Conductive material from test 2

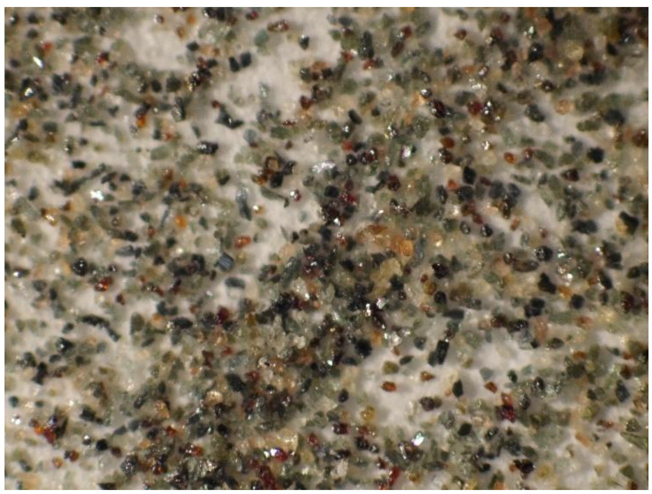

Figure 33: Optical microscopy picture of Mixed material from test 2

Figure 34: Optical microscopy picture of Non-conductive material from test 2

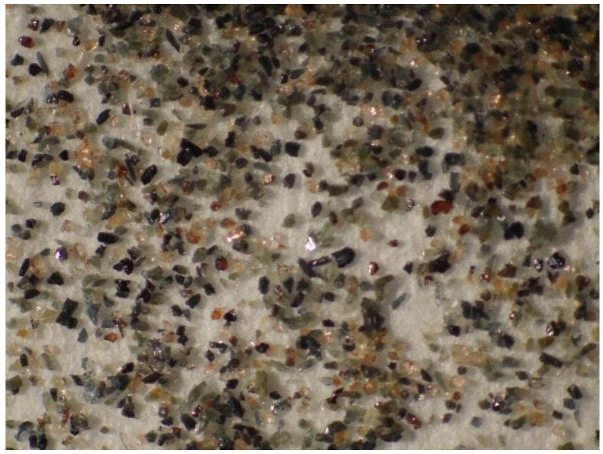

Figure 35: Optical microscopy picture of Magnetic 1 material from test 2

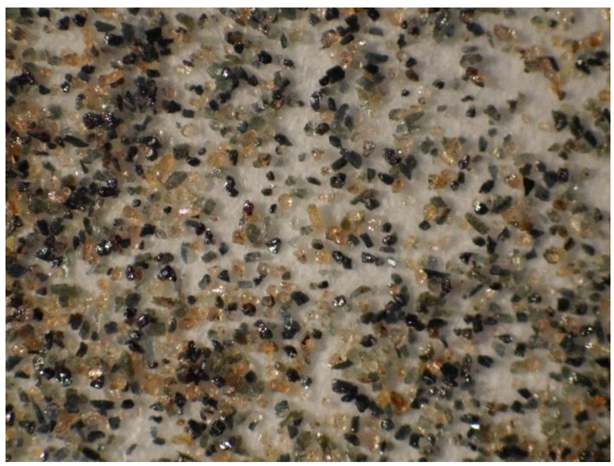

Figure 36: Optical microscopy picture of Magnetic 2 material from test 2

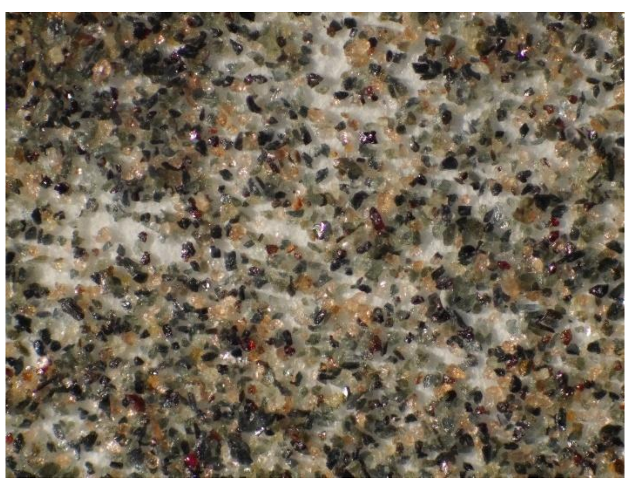

Figure 37: Optical microscopy picture of Magnetic 3 material from test 2

4.6 Conclusion

This study investigated sustainable mineral processing techniques for rutile extraction from eclogites, focusing on physical separation methods to minimize environmental impact. Various screening, characterization, and separation techniques were tested, with Test 2 (a combination of magnetic and electrostatic separation) proving to be the most effective method.

Test 2 demonstrated that a stepwise approach (first applying magnetic separation and then electrostatic separation) yields a better rutile recovery. The key advantages of this approach are efficient removal of iron-rich minerals, enhanced electrostatic separation efficiency by reducing the presence of iron and conductive minerals in the feed, electrostatic separation became more selective for rutile, leading to higher purity and better recovery rates, and optimal processing conditions by heating to 100°C and manual triboelectric charging.

These findings illuminate a promising path toward the elimination of chemical reagents by emphasizing dry and environmentally friendly processing methods. The results demonstrate the potential of these approaches to enhance efficiency while simultaneously reducing costs associated with energy consumption, processing time, and environmental pollution.

Key Findings and Contributions

1. Sustainable alternative to chemical processing: The results demonstrate that physical separation techniques can concentrate rutile without the need for chemical-intensive flotation methods, reducing environmental impact.

2. Importance of particle size control: Proper milling and screening ensured better liberation of rutile, improving separation outcomes.

3. Process optimization leads to higher recovery: The combination of medium-intensity magnetic separation and high voltage electrostatic separation (28 kV) with triboelectric charging was the most successful approach.

5. Recommendations and Future Research

Based on the findings of this research, it is recommended to conduct tests on coarser particle sizes (recommended particle size: 125µm to 500µm) to evaluate their impact on separation efficiency despite being over rutile liberation size to compare the results of different sizes. Additionally, gravity separation should be considered as a complementary technique, as it may play a key role in enhancing the overall beneficiation process. Furthermore, applying lower magnetic field intensities in magnetic separation could provide valuable insights by yielding different separation outcomes, allowing for a more comprehensive assessment of the process effectiveness. Other provided suggestions:

- Industrial Scalability: Further testing should be conducted on large-scale applications to assess process viability and economic feasibility.
- Energy Efficiency Improvements: While Test 2 was the most effective, optimization of energy consumption in milling and separation could enhance sustainability.
- Life Cycle Assessment (LCA): A full environmental evaluation should be conducted to compare the ecological footprint of this method against conventional chemical processing techniques.

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