THESIS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY

Selective Comminution Applied to Mineral Processing of Critical Metals

LORENA GULDRIS LEON

Department of Industrial and Materials Science

CHALMERS UNIVERSITY OF TECHNOLOGY

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Department of Industrial and Materials Science
Chalmers University of Technology
SE-412 96 Gothenburg
Sweden
Telephone + 46 (0)31-772 1000

Cover:
The cover illustration is a backscattered electron image of particles after a compressive breakage process. The central white particle is a liberated scheelite.

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To Sofia…

To the little one who teaches me and strengthens me day by day.

ABSTRACT

The outstanding properties of tungsten and tantalum make them valuable metals and, in some cases, irreplaceable in applications. Due to the vital economic importance and limited supply, growing interest places these metals on the critical metals risk list. Due to increasing global demand for these metals, there is also a need to develop more efficient extraction processes. The size reduction of particles commonly assesses coarse comminution processes. Nevertheless, does not include mineralogy and element content.

The primary hypothesis of this research is that the utilization of selective comminution could enhance the efficiency of critical metal extraction, rendering it a valuable and cost-effective method in comparison to conventional approaches. As critical metals do not uniformly distribute across distinct size fractions during coarse comminution processes, owing to the influence of mineralogical composition and texture on particle breakage. To underscore the significant role of mineralogy in breakage, a novel testing procedure is proposed, which involves the concentration of critical metals following compressive breakage, aiming to augment the resolution of coarse comminution models.

This study is dedicated to formulating an analytical methodology and test protocols aimed at analysing and characterizing selective comminution possibilities during compression breakage. The research progresses across three key phases. The primary phase involves the comprehensive characterization of rock materials, encompassing mechanical, chemical, and mineralogical analyses. The subsequent phase involves modelling, followed by the third phase, which entails the technical and economic evaluation of material in a theoretical plant distribution case study.

Mechanical characterization includes laboratory-based compressive crushing, encompassing interparticle breakage, while chemical and mineralogical characterization is conducted by evaluating size-fractioned samples post-compression breakage, employing techniques such as scanning electron microscopy (SEM) and geochemical analysis. These tests yield valuable insights into breakage behaviour, mineral composition, and elemental concentration, with implications for early material rejection strategies.

Geochemical analysis is carried out using inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma atomic emission spectroscopy (ICP-AES). Additionally, the production of particles in nano sizes, pressed powder pellets, followed by analysis via Laser Ablation Inductively Coupled Plasma Mass Spectrometry (PPP LA-ICP-MS), offers a cost-effective and suitable means to measure elemental content, circumventing the laborious and costly steps associated with standard techniques such as fused bead and acid digestion.

The data generated through the developed analytical methodology undergoes rigorous analysis and is fitted into a model, which employs a bimodal Weibull distribution for calibration. This concentration model excels in simulating critical metal concentrations based on compression ratios, and it can forecast rare metal concentrations in relation to particle size distributions following compression crushing.

Following comprehensive study, analysis, and modelling of mineral composition, a tool is devised that combines technical and economic models, enabling the optimization of production by enhancing product quality, cost-efficiency, profitability, and capacity. The results demonstrate that the proposed model facilitates the determination and enhancement of process capacity and profitability. Utilizing the technical and economic model offers the prospect of elevating profitability by reconfiguring mass flows based on the mineral composition of the rock, aligning with plant distribution objectives.

In conclusion, the implementation of the developed analytical method enhances the assessment of coarse mineral liberation characteristics, offering fundamental insights into how various ore materials tend to break post-compressive crushing, concerning mineral and elemental distributions. Armed with this information, it becomes possible to propose, refine, and assess the cost of process adjustments, such as machine parameters, plant design, and early material rejection strategies, tailored to the specific properties of each ore.

Keywords: compressive crushing, selective comminution, analytical method, critical metals, minerals, elements.

APPENDED PUBLICATIONS

The following papers are appended to this thesis:

Paper A: Leon, L.G.; Bengtsson, M.; Evertsson, M. Analysis of the concentration in rare metal ores during compression crushing. Miner. Eng. 2018, 120, 7–18. https://doi.org/10.1016/j.mineng.2018.01.041

Paper B: Guldris Leon, L., Hogmalm, K., Bengtsson, M. (2020). Understanding Mineral Liberation during Crushing Using Grade-by-Size Analysis - A Case Study of the Penuota Sn-Ta Mineralization, Spain. Minerals, 10(2). http://dx.doi.org/10.3390/min10020164

Paper C: Guldris Leon, L., Lebrun Thauront, J., Hogmalm, K. et al (2022). Evaluation of Refractory Metal Concentrations in Nano-Particulate Pressed-Powder Pellets Using LA-ICP-MS. Minerals, 12(7). http://dx.doi.org/10.3390/min12070869

Paper D: Guldris Leon, L., Bengtsson, M. (2022). Selective Comminution Applied to Mineral Processing of a Tantalum Ore: A Technical, Economic Analysis. Minerals, 12(8). http://dx.doi.org/10.3390/min12081057

In paper A, Guldris Leon and Bengtsson initiated the idea. The testing was performed by Guldris. Guldris and Bengtsson wrote the paper with Evertsson as a reviewer.

In paper B, Guldris Leon, Hogmalm and Bengtsson initiated the idea and co-wrote the paper with Hogmalm as a reviewer.

In papers C, Guldris Leon and Hogmalm initiated the idea. The testing was performed by Guldris and Thauront. Guldris Leon, Hogmalm, Hulthén, and Malmqvist analysed and discussed the results. Guldris Leon and Thauront co-wrote the paper with Hogmalm as a reviewer.

In paper D, Guldris Leon, and Bengtsson initiated the idea and co-wrote the paper with Bengtsson as a reviewer.

OTHER PUBLICATIONS

Paper 1: Guldris, L., Bengtsson, M., and Evertsson, C. M. *Reduction and fracture analysis of tungsten ore and its use for fundamental liberation modelling*. 12ª Conferencia Internacional de Procesamiento de Minerales (PROCEMIN 2016). 2016. Santiago, Chile.

Paper 2: Guldris, L., Bengtsson, M., and Evertsson, C. M. *Mineralogical characterisation, reduction and liberation analysis of tungsten ore*. Emerging Trends in Minerals Engineering Conference (IOM^3). 2016. London, U.K

Paper 3: Guldris, L., Bengtsson, M., and Evertsson, C. M., *Modelling Reduction and Liberation for Rare Earth Minerals Applications.* International Comminution Symposium (Comminution '16). 2016. Cape Town, South Africa.

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APPENDED PAPERS

Paper A: Analysis of concentration in rare metal ores during compression crushing.

Paper B: Understanding mineral liberation during crushing using grade-by-size analysis - a case study of the Penuota Sn-Ta-Nb mineralization.

Paper C: Evaluation of tungsten and tantalum content through nano-particulate pressed power tablets for LA-ICP-MS.

Paper D: Selective Comminution Applied to Mineral Processing of a Tantalum Ore: A Technical, Economic Analysis.

LIST OF FIGURES

LIST OF ABBREVIATIONS

CRPS	Chalmers Rock Processing Systems
CSS	Close Side setting
BCA	Bulk chemical analysis
EDS	Energy Dispersive Spectroscopy
PPP	Pressed Powder Pellets
ICP-AES	Inductively Coupled Plasma Mass Spectrometry
ICP-MS	Inductively-Coupled Mass Spectrometry
IPB	Interparticle Breakage
SEM	Scanning Electron Microscopy
SPB	Single particle Breakage
QMA	Quantitative Microstructural Analysis
VSI	Vertical Shaft Impactor

b	Bed height	[mm]
s	displacement of the piston	[mm]
s/b	Compression ratio	[–]
$x_{s/b}$	Normalized particle size	[–]
x	Particle size	[mm]
B	Breakage matrix	[–]
f	Feed distribution	[mm]
I	Identity matrix	[–]
p	Product Distribution	[mm]
S	Selection matrix	[–]
α	Calibration constant	[–]
β	Calibration constant	[–]
γ_o	Normalized frequency distribution concentration	[–]
v	Calibration constant	[–]
λ	Calibration constant	[–]
d	Integration interval fraction size	[mm]
c	Integration interval fraction size	[mm]
i	Element index	[–]
m_i	Mass in each fraction	[g]
$m_t\ ot$	Total mass	[g]
p_i	Size fraction	[–]
H_0	Cumulative normalized distribution concentration	[–]
Q	Capacity	[tons]
γ	The proportion of the feed that is split	[–]
C	Production cost	[unit/h]

1

INTRODUCTION

The objective of this chapter is to:

- *Introduce general concepts related to mineral and comminution processing.*
- *Describe the aim of this research.*
- *Describe challenges associated with coarse comminution processes.*
- *Formulate research questions.*
- *Define delimitations of the research.*
- *Provide an outline of the thesis.*

1.1 BACKGROUND

Tungsten and tantalum are valuable and irreplaceable metals with a wide range of applications in modern technology, spanning from industrial to electronic sectors. The significance of these metals is rooted in their unique properties, such as high density, the highest melting point among all metals, and low reactivity. When combined with other materials, they enhance mechanical properties like hardness, toughness, and corrosion resistance. Tungsten and tantalum are raw materials of great economic importance and have a moderate supply risk. Their limited availability of suitable substitute materials, considering the quality and performance they offer, places the extraction of these metals at the top of the "risk list," according to the British Geological (Survey, 2015) immediately following rare earth elements.

Tantalum is a transition metal chemical element with the chemical symbol Ta and atomic number 73. Among its notable properties are high density (16.6g/cm^3), a high melting point (3017°C), a high boiling point (5455°C), and excellent corrosion resistance. Tantalum is typically found in minerals like microlite and the columbite group, often occurring alongside niobium, a metal with similar properties. Tantalum finds principal applications in capacitors for electronic devices, super-alloys in the aerospace industry, and cutting tools (Simandl et al., 2018). Table 1 presents a summary of tantalum properties.

Table 1: Selected properties of Tantalum and Tungsten.

Property	Tantalum	Tungsten
Chemical symbol	Ta	W
Atomic number	73	74
Crystal structure	body-centred cubic (bcc)	body-centred cubic (bcc)
Atomic weight (g/mol)	180.94	183.84
Density (g/cm^3)	16.6	19.3
Melting point (°C)	3017	3422
Boiling point (°C)	5458	5555
Hardness (Mohs)	6.5	7.5

Like all metals, these critical metals are extracted from ores through a series of steps, starting with the reduction of ore particle size through comminution. Comminution is the process of reducing particle size by fracturing the ore through crushing, grinding, milling, or similar methods.

1.1.1 Minerals and Mineral Processing

Mineral processing includes various processes required for the extraction of valuable metals and the production of commercial end products. These minerals are typically found in ores or rocks that are not homogeneous in terms of chemical composition and physical properties. Valuable minerals are often finely disseminated and closely associated with gangue, which holds no commercial value, as depicted in Figure 1.

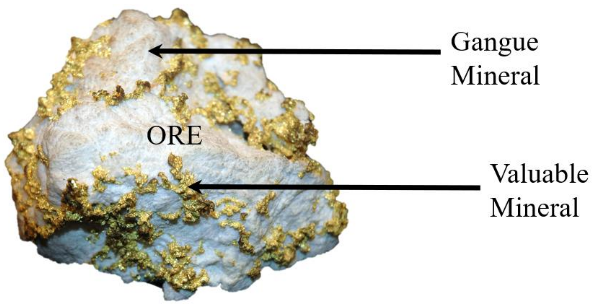

Figure 1: Ore structure (Gold ore). Valuable minerals are finely disseminated and intimately associated with gangue minerals. (Creative Commons Attribution 2.0 Generic courtesy picture of Mother Lode Gold Ore, Grass Valley)

The term content of valuable minerals refers to the ore grade, with valuable minerals generally present in lower percentages compared to gangue minerals. As an example, in copper ores, the valuable mineral content can be approximately in the range 2 to 0.5% (Haldar, 2018). However, in low-grade ores, like those containing tantalum and tungsten, the percentage of valuable minerals typically ranges from 0.1% to 0.03%. To better visualize ore composition, refer to Figure 2, which depicts a square representing an ore (Figures 2A and 2B). The blue squares symbolize gangue minerals, while the green squares represent valuable minerals. Typically, valuable minerals are not concentrated in one specific area (Figures 2B and 2E). Instead, they are finely disseminated and intimately intermixed with gangue minerals (Figures 2D and 2F). Figure 2 illustrates two scenarios: a high-grade ore with a 3% content of valuable minerals (Figures 2A to 2D) and a low-grade ore with 0.3% of valuable mineral content (Figures 2E and 2F). To effectively separate valuable minerals from gangue minerals, it is necessary to reduce the size of the ore particles until the necessary separation is achieved.

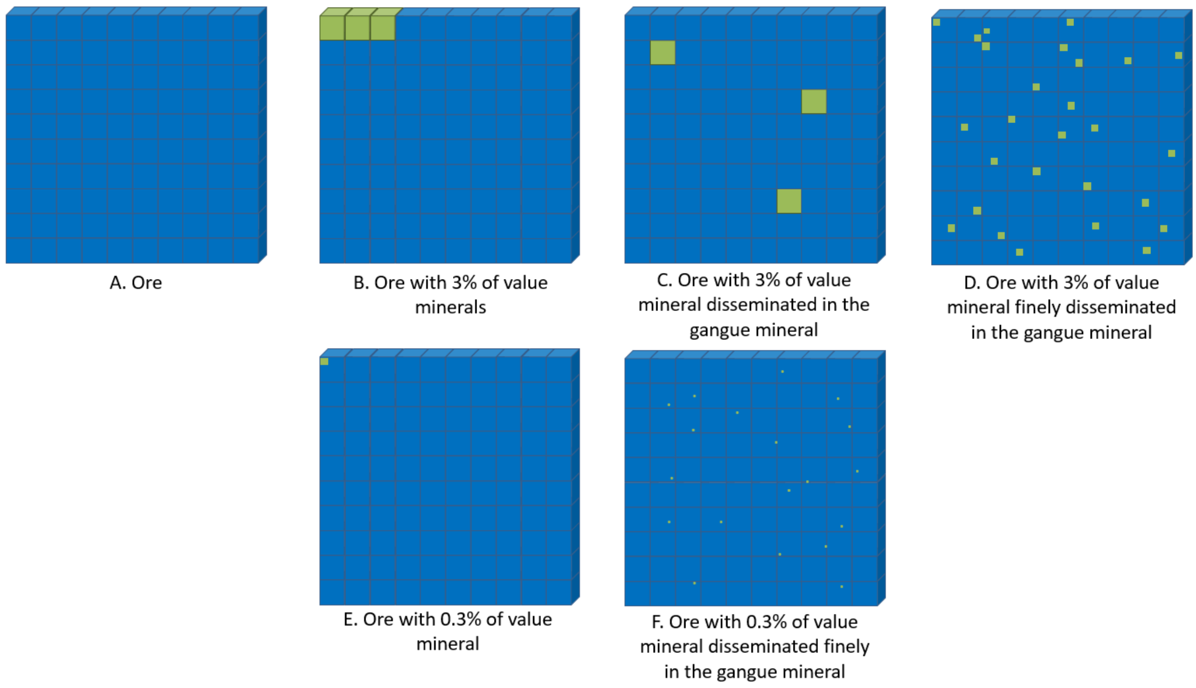

Figure 2: Graphical representation of the relationship between valuable and gangue minerals and the dissemination in the ore.

The primary objective of mineral processing is to liberate and separate valuable minerals from their associated waste gangue minerals. Valuable minerals are extracted through a series of ore processing steps, including size reduction and physical separation. This separation process results in two products: concentrate and tailings. The concentrate is the enriched product containing the majority of valuable minerals, while the tailings consist mainly of gangue minerals that are discarded. The liberation of valuable minerals is achieved by reducing particle size through comminution processes, as illustrated in Figure 3. Reducing particle size is necessary to obtain a mixture of relatively clean and pure particles of either the desired mineral or gangue (Wills & Finch, 2015). Figure 4 presents a flowsheet of the mineral processing process, with a focus on interactions during the comminution stage.

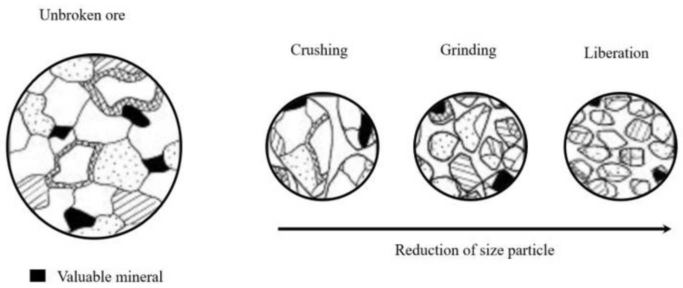

Figure 3. Comminution of an unbroken ore. The particle size reduction is obtained through different comminution processes, such as crushing and milling, until the liberation is obtained.

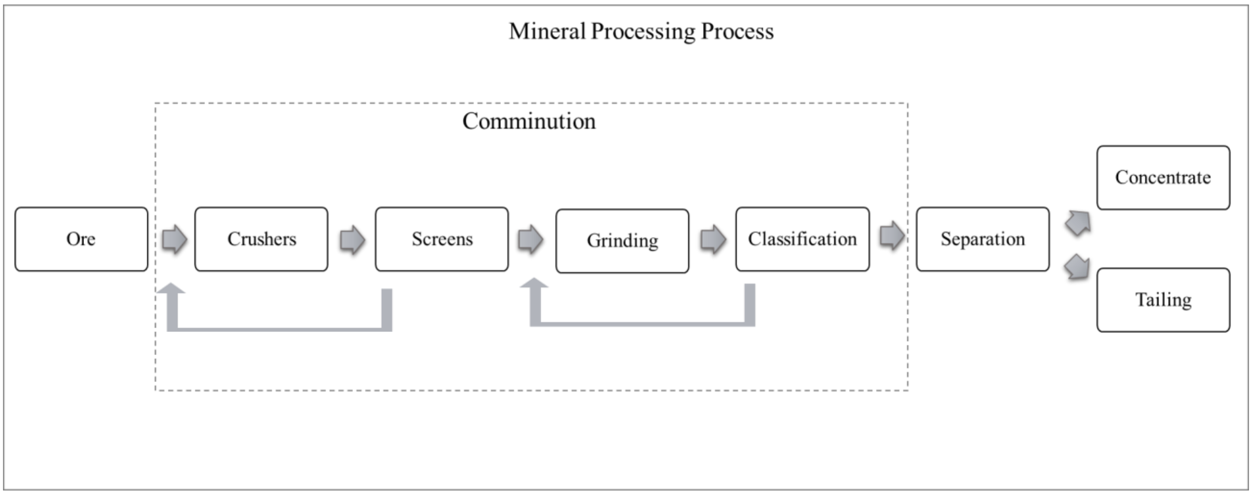

Figure 4. Flowsheet of the mineral processing process. (Wills & Finch, 2015)

Comminution processes serve specific purposes, such as liberating valuable minerals from the ore matrix, increasing the surface area of particles by reducing their size, and facilitating the transportation of particles between processing stages (Fuerstenau & Han, 2003). The comminution circuit comprises crushing, grinding, and initial rejection, as indicated by the dashed box in Figure 4. A comminution plant typically involves two to three crushing stages and one to three milling steps, as depicted in Figure 5. Each stage reduces particle size to a specific degree. When particles reach the desired level of reduction, they are transferred to the next stage to be further reduced until they achieve the required size. Primary crushers are typically jaw or gyratory crushers, while secondary crushers can be of the gyratory, jaw, or cone type. Tertiary crushers commonly include cone crusher. Each step in the process targets particle size reduction. In the primary stage, particle size ranges from 1000 to 500 mm, while at the end of the tertiary stages, particle size typically falls between 22 to 16 mm. Figure 6 illustrates the various process steps and the corresponding particle size reductions. The relationship between the initial particle size (feed) and the final discharge size is referred to as the reduction ratio. High reduction ratio values do not necessarily indicate efficiency (construction, 2015).

The design of the plant typically relies on various factors, such as ore grade, ore type, ore deposit variability, operational scale, and available investment capital.

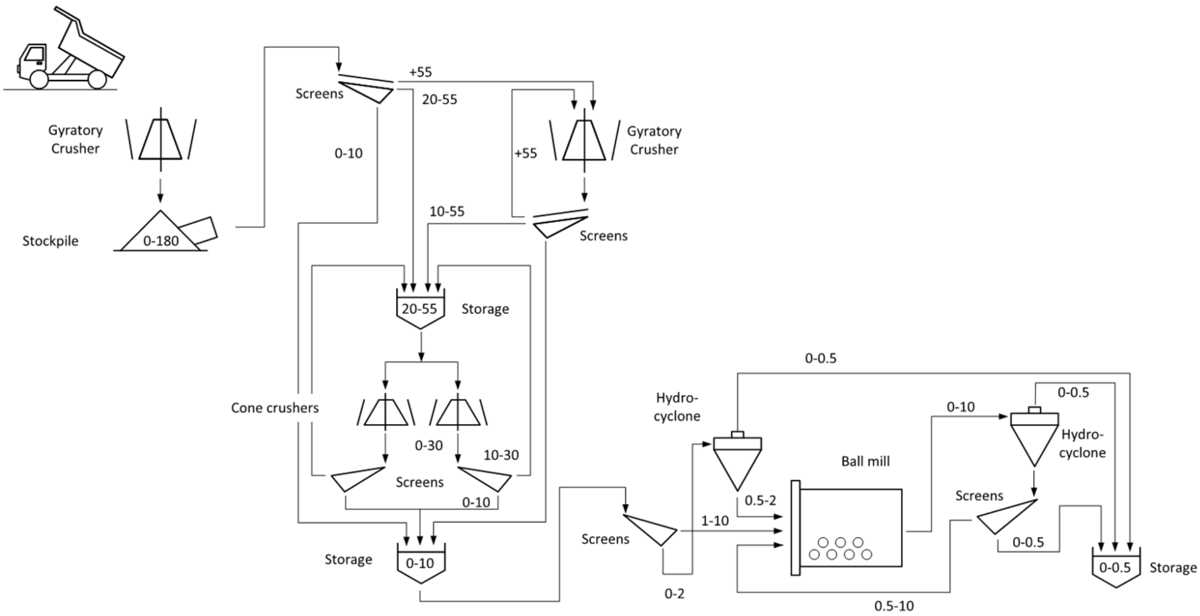

Figure 5. Size reduction circuit for mineral processing of an ore.

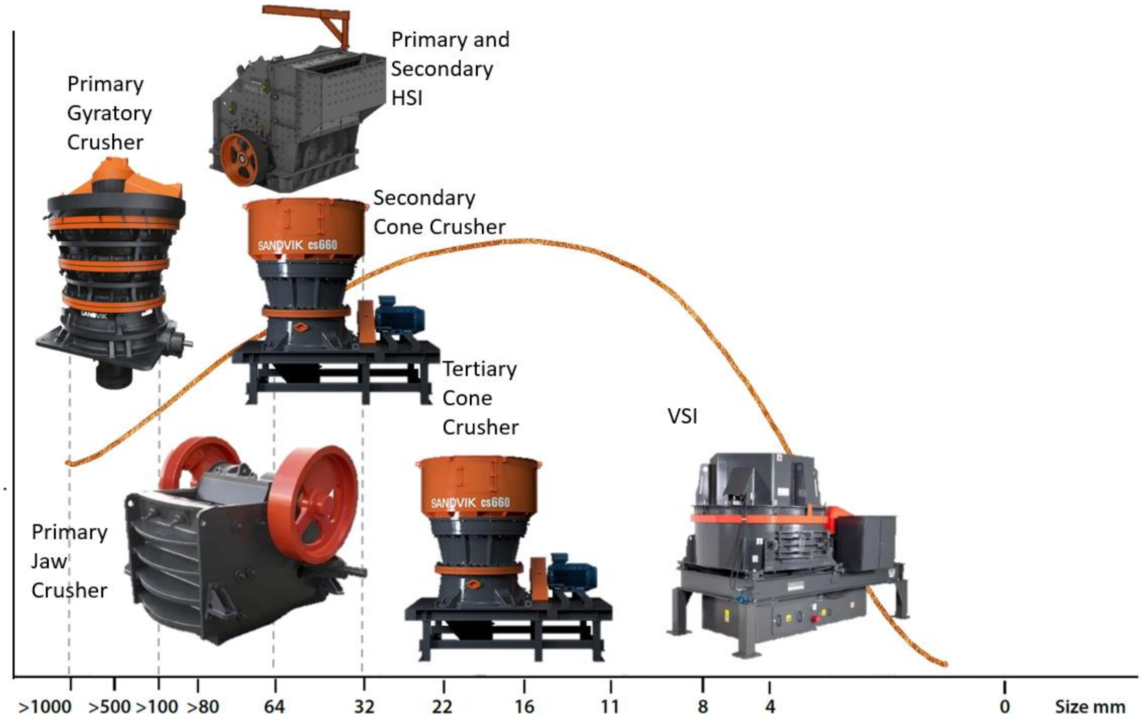

Figure 6. Size reduction process for the crushing stages, from primary to tertiary stage with the associate crusher type, applicable to aggregate industry and mining sector. (Sandvik courtesy pictures of crushers)

1.1.2 Comminution

Comminution is the process of reducing the size of ore particles to liberate valuable minerals from gangue, enabling the subsequent separation process in the following stages, as described in Figure 3 (Wills & Finch, 2015).

Comminution in mineral processing plants occurs as a sequence of crushing (coarse comminution) and milling processes (fine comminution), as illustrated in Figure 4. The comminution of rock materials can be achieved through three different breakage mechanisms: compression, impact, and attrition. Typically, during the crushing stage, particle reduction is achieved through compression or impact forces, whereas in milling, the primary breakage mechanisms involve abrasion and impact. Crushing is generally conducted as a dry process, while milling is typically performed under wet conditions (Wills & Finch, 2015).

The comminution process demands a substantial amount of energy as it necessitates that all particles pass through various equipment and processes to achieve size reduction. Approximately 2% of the total electrical energy generated worldwide is dedicated to the comminution process (Napier-Munn, 2015). Figure 7 provides a comparison of energy requirements across different phases of the process. Notably, grinding demand the highest energy consumption. The energy distribution typically accounts for 80% to 90% during the grinding stage, while the crushing stage consumes approximately 5% to 7% (Jeswiet & Szekeres, 2016; Shi et al., 2009; Wang et al., 2013).

The most significant cost in mining operations is associated with energy expenses, with approximately 70% allocated to the crushing and milling processes (Nava Rosario, 2021; Norgate & Haque, 2010). Notably, fine comminution, which includes grinding and processing, accounts for between 80% to 90% of the total energy consumption, while the crushing stage consumes around 5% to 7% (Jeswiet & Szekeres, 2016; Shi et al., 2009; Wang et al., 2013).

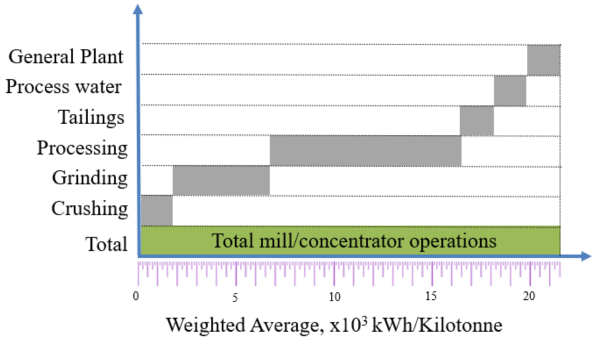

Figure 7. Average energy needs for mill/concentration operations (Jeswiet & Szekeres, 2016).

While the objective of comminution is to reduce the size of particles, there is a risk of producing fine-sized particles that become difficult to treat and may end up in the tailings, a phenomenon known as over-grinding. This can lead to high energy costs (Wills & Finch, 2015). Additionally, over-grinding and the generation of excessively fine particles can render them unsuitable for further processing.

In coarse comminution processes, the assessment of quality and performance is typically based on measuring the particle size distribution, without considering the composition of particles. The evaluation of mineral liberation, chemical composition, and mineralogical composition of particles it is typically conducted at the end of the fine comminution process.

1.2 PROBLEM CLARIFICATION

Comminution processes require significant energy input to achieve the desired size range of ore particles before separation. Every particle must pass through all equipment and stages, including coarse comminution (crushers) and fine comminution (mills). Typically, the quality and performance of coarse comminution processes are evaluated by measuring the particle size distribution. However, it is after the milling stage that the assessment of mineral liberation and composition is typically conducted at the end of the process.

All minerals constituting an ore exhibit a range of characteristics and properties, including grain size, grain shape, intergranular bonding, and mechanical properties like strength, toughness, and brittleness (Mahabadi et al., 2014; Nadan & Engelder, 2009; Nur & Simmons, 1970). These properties can significantly impact how the ore breaks and influences particle size distribution (Hamid et al., 2018; Little et al., 2016; Vizcarra et al., 2010). Therefore, it's crucial to investigate and understand the variations among mineral phases and element concentrations within the ore. The breakage process should be considered when designing industrial comminution processes, with special consideration during the final stages (King & Schneider, 1998; Vagonova et al., 2014).

Recently, a new process approach associated with the phenomenon selective comminution has emerged to enhance the processing efficiency and, consequently, substantially reduce energy consumption. The selective comminution is based on a size-based preferential grading process. Selective comminution relies on the ability of certain ores to distribute metals into specific size fractions (C. Carrasco et al., 2016; Cristian Carrasco et al., 2016). Identifying key factors such as grain shape, texture, grain size, breakage mechanism, and element concentration is essential for recognizing differences in mineral behavior (Hesse et al., 2017).

Figure 8: Required testing methodology that evaluates different ore types, mechanical properties, mineralogical composition and element content.

It is crucial to establish accessible methodologies and models for the analysis, characterization, and prediction of these concepts, integrating them into coarse comminution processes as depicted in Figure 8. Currently, there is limited fundamental information available regarding mineral breakage during coarse comminution and the feasibility of pre-concentration.

Substantial advancements in comminution technologies may arise from a deep, fundamental understanding of liberation characteristics, which can help assess the potential of pre-concentration stages. The development of a methodology to evaluate element distribution can be employed to examine the influence of mechanical parameters on mineral liberation in the early stages of the comminution process.

1.3 RESEARCH AIM

The primary hypothesis of this research is that using selective comminution could be possible to improve the critical metal extraction efficiency, making it a valuable and cost-effective method compared to conventional approaches.

The application of selective comminution techniques, preceded by comprehensive ore characterization and aided by predictive models and advanced analytical methods, will result in a significant enhancement of critical metal extraction efficiency compared to traditional extraction processes. This enhancement will not only improve resource utilization but also demonstrate cost-effectiveness, making it a viable approach for meeting the growing demand for critical metals.

Critical metals are not uniformly distributed across various size fractions during coarse comminution processes. This uneven distribution results from the influence of factors such as mineralogy, texture, grain shape and size, breakage mechanism, and element concentration on particle breakage. Consequently, there is a need for a protocol to enhance coarse comminution processes and models by systematically measuring and integrating ore characteristics.

The objective of this work is to introduce an analytical method, modelling approach, and case analysis to enhance the assessment of coarse mineral liberation characteristics. The method will elucidate how various ores break during compressive crushing in terms of minerals and elements. The analytical method, modelling process, and case analysis of material characterization are illustrated in Figure 9 and Figure 10.

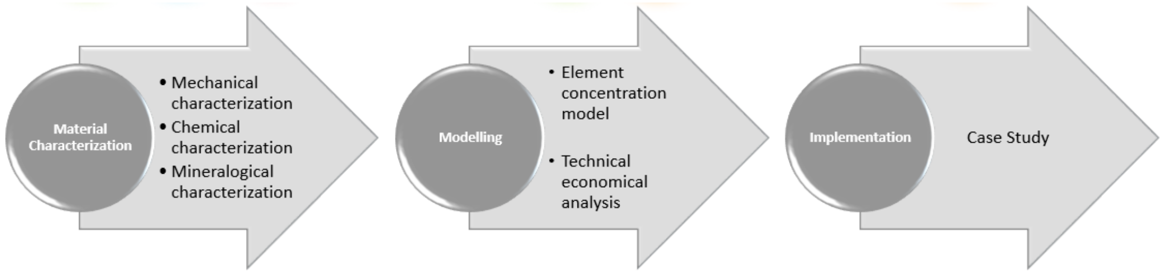

Figure 9: Test procedure.

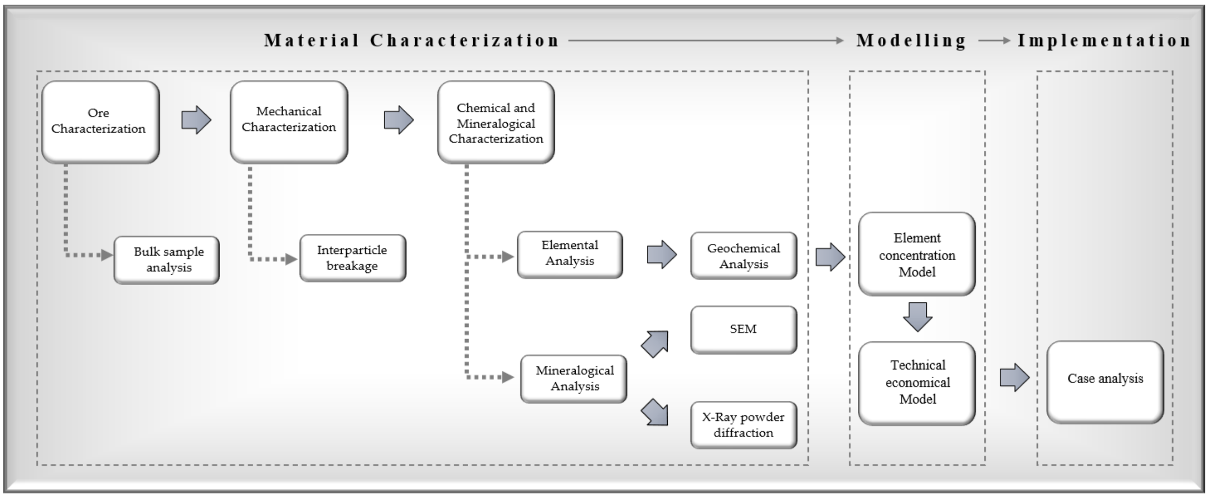

Figure 10. Material characterization and detailed test procedure presented in this thesis.

1.4 RESEARCH SCOPE

This work aims to develop a method capable of analyzing the bulk chemistry of different particle size fractions produced after compression breakage. The specific objective of this method is to test and predict critical metal concentrations as a function of particle size distribution following compression breakage cycles. The subsequent step involves creating a model that can predict critical metal concentrations based on particle size. The final part of the research involves the practical application of these fundamental concepts, analyzing the utilization of selective comminution in both technical and economic terms, with a focus on cost benefits.

The research primarily concentrates on compressive breakage during the tertiary crushing stage, as illustrated in Figure 11. The dashed box represents the research's focal area, where the method developed in Figure 9 and Figure 10 will be implemented as test point samples.

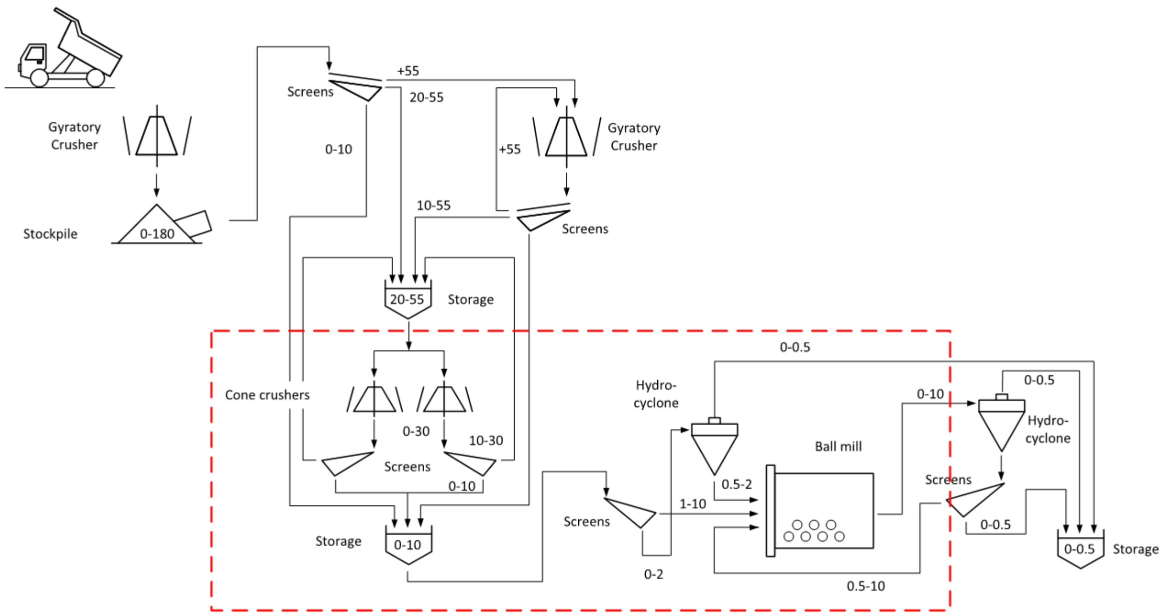

Figure 11. The entire figure represents the size reduction circuit for the mineral processing of ore. This work focuses on tertiary crushing stage and the following ball mill stage, as shown in the dashed box.

1.5 RESEARCH QUESTIONS

The following research questions describe the scope of this thesis:

RQ 1) What protocol would be suitable for identifying the benefits of a selective comminution process?

RQ 2) What properties should be analysed during coarse comminution testing to guide the early selective comminution process? Furthermore, what methods and protocols can guide the coarse comminution process in order to improve the processing of critical metals?

RQ 3) How can element concentration be measured from a process control perspective?

RQ 4) How can a theoretical framework be designed to utilize technical and economical aspects when addressing selective comminution to extract critical minerals?

The research questions outlined above will be explored and answered in the discussion chapter of the thesis. Figure 12 illustrates the connection between the appended papers and these research questions.

	RQ1	RQ2	RQ3	RQ4
Paper A				
Paper B				
Paper C				
Paper D				

Figure 12 illustrates the connection between appended papers and research questions. Larger circles represent a strong relationship.

1.6 DELIMITATIONS

A set of delimitations has been formulated to limit the scope of the research. The delimitations are described below:

- The research is based on compressive breakage.
- The research is based on the study and characterization of critical metal ores. The modelling of concentration was validated for tantalum and tungsten ores.
- This research does not include initial sampling techniques.
- The research does not include milling modelling.
- The research does not include screening and classification modelling.
- This research does not include dynamic process simulations.

1.7 OUTLINE OF THESIS

The thesis is divided into five chapters. A short description of each chapter is provided below.

- Chapter 1. The Introduction section presents research content, problem, scope and research questions.
- Chapter 2. The Frame of Reference section includes the theoretical background and concepts related to the research topic and current testing techniques used in material characterization.
- Chapter 3. The Research Approach section presents the methodology applied in this research.
- Chapter 4. Research Findings and present the core findings of the appended papers.
- Chapter 5. The Discussion Chapter answers and discusses the research questions established and the validity of the results. Findings are summarized and include the scientific contributions, utilization and future research.

2

FRAME OF REFERENCE

This chapter objective is to:

- *Introduce general concepts related to mineral and comminution processing.*

2.1 PARTICLES AND THEIR CHARACTERISTICS

Value minerals are finely disseminated and closely associated with gangue. To separate valuable minerals, it is necessary to unlock or liberate them to achieve separation and subsequent recovery. Comminution processes are designed to reduce particle size and liberate the valuable minerals.

Following the comminution process, the ore is reduced to small particles composed of various minerals. Key characteristics of these particles include their size, shape, structure (single-component or composite), as well as their mineralogical and chemical composition (Fuerstenau & Han, 2003). Additionally, other characteristics define the particles, including density, surface area, reactivity, toxicity, magnetic susceptibility, heat capacity, solubility, and reactivity (Fuerstenau & Han, 2003).

Grain size is closely linked to the size and growth of the minerals within it, making it a critical factor when discussing the liberation and processing of minerals. Another vital concept is mineral associations, which describe how minerals share or are surrounded by boundaries with other minerals. Both grain size and mineral associations have a substantial impact on concepts like liberation and the subsequent processing of minerals (Ahmad Hamid et al., 2019).

Process mineralogy integrates mineral processing with bulk mineralogy, degree of liberation, and the minerals involved to enhance the processing flow sheet for a specific ore (Baum et al., 2004; Henley, 1983). By linking mineral information with comminution, it may be possible to examine and establish optimal conditions for the process. However, these concepts have not yet been efficiently employed to predict and develop optimal liberation of mineral particles during comminution processes (Mwanga, 2016). It's crucial to note that a methodology for size reduction that incorporates mineral and element information during coarse comminution processes is still lacking.

2.2 COMMINUTION

2.2.1 Cone Crushers

For secondary and tertiary crushing stages, the most commonly used equipment is known as a cone crusher. Cone crushers operate on the principle of compressive breakage. During the reduction process in this machine, particles undergo both single particle breakage (SPB) and interparticle breakage (IPB) throughout different stages of the process. Compression of particles is achieved by moving the inner cone (mantle) while the outer cone (concave) remains fixed.

The final particle size can be controlled by adjusting the distance between the mantle and the concave, a parameter known as the closed side setting (CSS). There are various types of cone crushers available on the market, all employing the same principle but with different designs and sizes. The choice of crusher size and chamber design depends on capacity, material, and feed particle distribution requirements. Figure 13 provides an overview of a general cone crusher and its design.

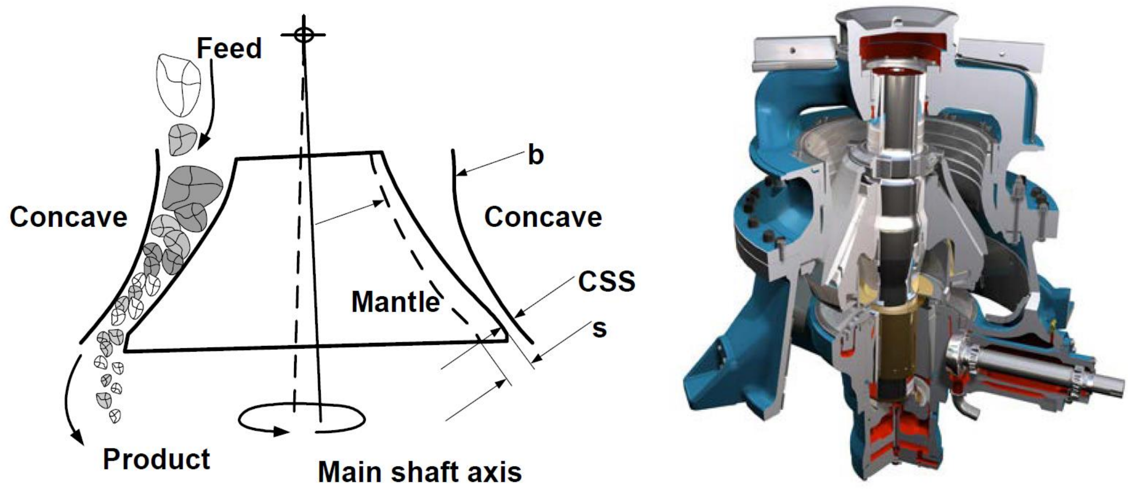

Figure 13. Cone Crusher (Bengtsson et al., 2009; Evertsson, 2000)

2.2.2 Breakage Principles

The breakage of materials is a complex phenomenon resulting from the transmission of energy to separate mineral particles and liberate minerals. Mechanical forces are employed to break down the mineral matrix into distinct mineral phases. Particle fracture occurs when the applied stress surpasses the material's strength, leading to the formation of cracks. The breakage mechanism is closely related to the failure of the particle (Mwanga, 2016; Semsari Parapari, 2020).

There are three main comminution principles of industrial interest:

- Impact breakage
- Attrition
- Compressive breakage

The three principles are shown in Figure 14. In the sub-sections below, the different mechanisms will be described.

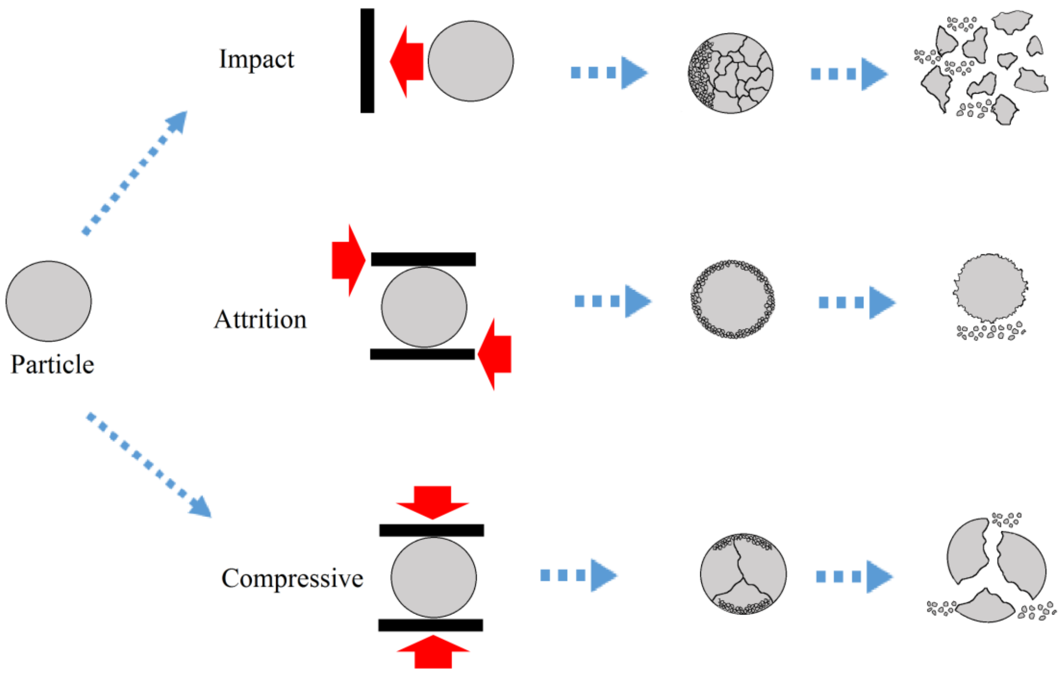

Figure 14. Particle breakage mechanism forces present in the particles and the resultant distribution of broken particles.

2.2.2.1 Impact breakage

As the name suggests, impact breakage occurs due to the generation of intense stress during an impact. In this case, acceleration transfers energy to particles in the form of kinetic energy. Fragmentation takes place when particles impact walls or other particles, leading to multiple fractures. The impact breakage process involves a sequence of steps, starting with the fracture of the original particle and followed by sequential fracturing of already fragmented particles. This process continues until the energy is dissipated (Tavares & King, 1998). Impact breakage is considered the most efficient method in terms of energy and particle reduction. Figure 14 illustrates the basic principle of this type of breakage and the resulting particle size distribution.

The extent of size reduction is controlled by the amount of energy transferred to the particle. Therefore, the breakage can be regulated by adjusting the speed. Equipment that operates based on this principle is known as impact crushers. These crushers are designed to reduce particle size, but during the process, they often generate fine particles due to the abrasive forces involved, which can sometimes be necessary but are generally undesirable (Evertsson, 2000). Equipment that employs this principle includes the vertical shaft impactor (VSI), hammer mill, and impact mill.

2.2.2.2 Attrition

Attrition forces arise due to shear failure resulting from the interaction between particles. This type of breakage typically occurs when tangential loads or the application of stress, not high enough to cause fracture, act on large particles (Napier-Munn et al., 2005). Attrition breakage results in the production of fine particles from the surface of larger particles. It mainly generates fine material and can be attributed to impact, compression, shear forces, or a combination of these factors (Anticoi Sudzuki, 2019). Figure 14 illustrates the fundamental principle of attrition in breakage and the resulting particle size distribution.

2.2.2.3 Compressive breakage

Compressive breakage involves the crushing of rock material through the application of compression forces between two surfaces. In this type of breakage, the contact forces between particles increase tensile stresses within them. When these tensile stresses surpass a critical value, cracks form and propagate, ultimately breaking the rock into smaller pieces. The extent of size reduction can be regulated by controlling compression through force and input energy. Examples of crushing equipment that utilize compressive breakage principles for particle size reduction include jaw crushers, gyratory crushers, cone crushers, and roller crushers. Compressive forces can be applied either individually or in a repetitive process.

As a result of compressive forces, particles undergo breakage, leading to the formation of particles that can be categorized into two classes based on size. Larger fragments are produced due to fractures and cracking caused by tensile stress, while fine particles are generated through shear stresses at contact points. The principle of compressive breakage and the forces involved are illustrated in Figure 14 (Evertsson, 2000).

2.3 TESTING TECHNIQUES

2.3.1 Mechanical Test Methods and Modelling

Commonly used comminution methods include mechanical rock tests, particle breakage tests, as well as bench-scale grindability tests.

A mechanical rock test is typically conducted through compressive loading to determine rock strength by calculating the applied load and displacement over time. Typically, samples consist of drill core sections, single particles, multiple particles, or slices of a drill core, as illustrated in Figure 15 (Mwanga, 2016). The most common test method is a compression test, where the sample is pressed between two parallel surfaces until it fails, and the yield of the maximum load is used to calculate the compressive strength. These methods are advantageous because they are straightforward to apply and require relatively small quantities of material. Since rocks are not homogeneous, it is recommended to repeat the tests multiple times to ensure the repeatability of the obtained values. These tests not only provide information about tensile strength but also allow for the analysis of the size of resulting particles, enabling the determination of particle size distribution after compression breakage.

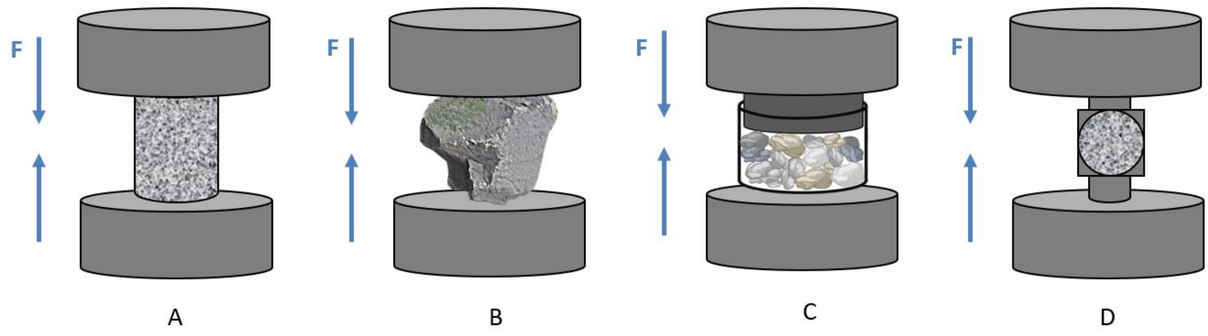

Figure 15: Rock mechanical test applied to different testing samples. A: Drill core sample. B: Single particle (single particle breakage). C: Bed breakage (interparticle breakage). D: Slices from drill cores (Brazilian test).

The second group of tests falls under the category of particle breakage tests. Within this category, various test types exist, including the simple drop weight test, twin pendulum test, split Hopkinson pressure bar test, and rotary single impact tester.

In the drop weight test, a known weight is released onto a sample of the ore from a specific height to determine the potential energy used. After the impact, the rock fractures, and the resulting particles are collected and analyzed for size to determine the breakage size distribution. While the test shares the same fundamental principles, advanced equipment can also detect impact waves. This test can be conducted using different types of samples, including drill core sections, single particles, or multiple particles, as illustrated in Figure 16. An extended version of this test has been developed, incorporating well-established instruments for ultra-fast cell devices.

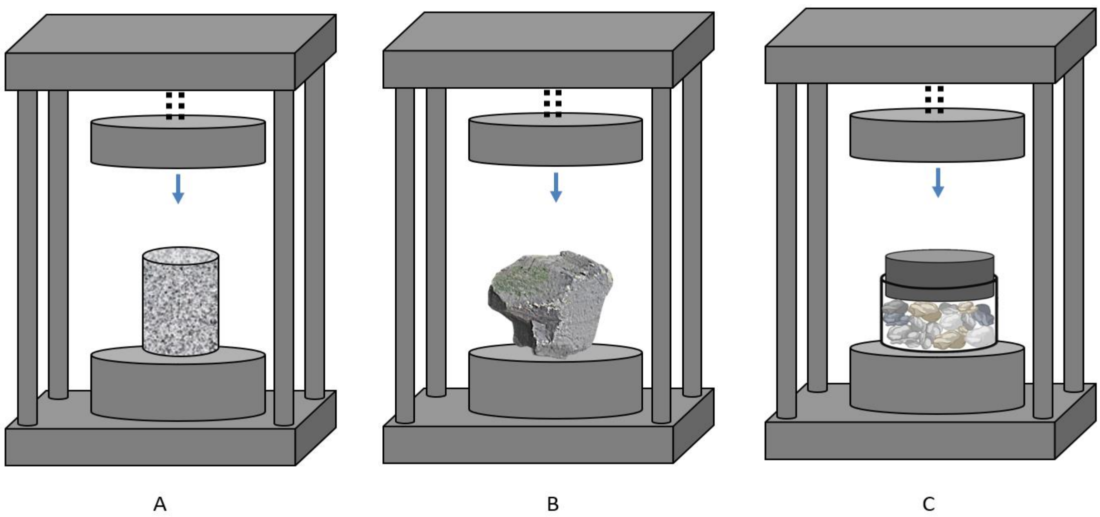

Figure 16: Drop weight test. A: Drill core sample. B: Single particle (single particle breakage). C: Multi-particle (interparticle breakage).

In the twin pendulum test, two pendulum hammers are released from a specific deflection angle and height to impact and fracture a single particle, as illustrated in Figure 17. This test, also known as the Bond test, enables the calculation of the crushing work index, considering the density of the rock and the impact energy for the deflection angle.

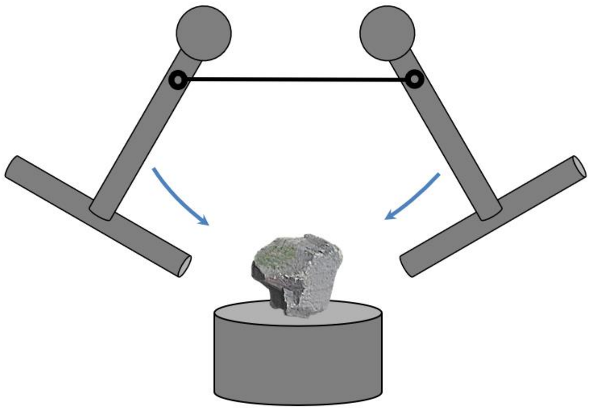

Figure 17: Twin pendulum test.

The Split Hopkinson Pressure Bar Test involves a collision between a single-particle sample and two horizontally mounted steel bars, an incident bar and a transmitted bar. A loading system applies stress, and deformation waves are recorded to gather data on load displacement and energy. The final test in the particle breakage group is the Rotary Single Impact Tester, where particles are accelerated against a plate by gravitational forces, resulting in collisions between the particles and the impact surface. To ensure reliability and confirm that values are not influenced by particle heterogeneities, these tests need to be repeated (Mwanga, 2016).

The third category of tests pertains to bench-scale grindability tests, with the Bond test being a prominent example. These tests are designed to assess the grindability of materials. The material is subjected to milling under standardized conditions related to the size of the mill, operating speed, and distribution of ball mills. Following this process, the particles' size distribution is analyzed, and a work index is calculated. Unlike the other two categories, which assess the impact and compression breakage of particles, this group focuses on the impact and attrition forces affecting the material. While conducting these tests requires a larger quantity of material, repeated testing is not necessary (Mwanga, 2016).

All three types of tests are applicable to comminution test work, covering both coarse (e.g., cone crushers) and fine comminution (e.g., high-pressure grinding rolls and mills).

Various studies, knowledge, and models in coarse comminution have sought to predict particle size distribution following crushing. Over the years, significant attention has been directed towards particle size distribution as a key factor in assessing the quality and performance of comminution processes. Coarse comminution models are capable of predicting particle size distribution, capacity, and energy consumption. These models trace their roots back to (Whiten, 1972)Whiten's empirical model (1972), wherein he introduced a population balance model for predicting particle size.

Evertsson (2000) contributed to this field by presenting a model for rock breakage in cone crushers. His work was grounded in rock breakage characterization through a piston and die test. This research led to the development of a laboratory test procedure for calibrating crusher models, incorporating both single and inter-particle breakage. Subsequently, Bengtsson (2009) introduced the the bimodal model to model size distribution for energy based crushing (VSI). These compressive breakage models provide insights into predicting particle size distribution.

However, it's worth noting that existing models have limitations stemming from the intrinsic physical properties of the ore, such as element/mineral concentration or mineral liberation. Bengtsson's work offers the potential for utilizing empirical models to predict these characteristics.

2.3.2 Chemical and Mineralogical Characterization

2.3.2.1 Chemical Characterization

To understand the element content in a mineral, a chemical characterization needs to be carried out. This analysis includes determining the content of major and minor rock-forming elemental components as well as trace elements. Such an analysis provides information about the elements present in a sample and their respective proportions relative to the total elemental concentration.

The most commonly used methods for chemical characterization include:

- X-ray fluorescence spectroscopy (XRF)
- Dissolution and atomic absorption spectroscopy (AAS)
- Inductively coupled plasma optical emission spectrometry (ICP-OES)
- Inductively coupled plasma mass spectrometry (ICP-MS)
- Electron probe micro-analysis (EPMA) with an energy-dispersive spectrometer (EDS)
- Electron probe micro-analysis (EPMA) with wavelength-dispersive spectrometer (WDS)
- Laser ablation inductively coupled plasma-mass spectrometry (LA-ICP-MS)

These methods are used to analyze and determine the chemical composition of ores and samples in mineral processing (Ahmad Hamid et al., 2019).

Typically, bulk chemical analysis is carried out by fusing beads and performing acid digestion of samples, followed by inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS). However, it's important to note that using ICP-AES and ICP-MS tests can be time-consuming, expensive, and pose certain challenges. As a result, the preparation and issues related to the dissolution of refractory minerals offer a more accurate approach to obtaining results.

Laser ablation inductively coupled plasma-mass spectrometry (LA-ICP-MS) is currently being utilized across various research fields and has the potential to streamline bulk analysis of minerals like rocks, ores, and sediments in a cost-effective and straightforward manner. LA-ICP-MS offers several advantages, including direct solid sample analysis, the ability to analyze multiple elements, and a rapid experimental procedure (Liu et al., 2013; Mukherjee et al., 2014). It eliminates the need for pre-treatment processes such as acid decomposition, which can lead to contamination or loss of analytes during sample preparation (Garbe-Schönberg & Müller, 2014; Ito et al., 2009; Liu et al., 2013; Miliszkiewicz et al., 2015).

There are four primary techniques for laser ablation analysis of geological samples: synthesizing minerals, selecting homogeneous natural minerals, vitrifying pulverized samples, and manufacturing pressed powder pellets (PPP) (Garbe-Schönberg & Müller, 2014). However, not all of these techniques are suitable for rocks, minerals, and elements. Nano-particulate PPP has been previously evaluated and studied for wet-milling protocols in aqueous suspension by Garbe-Schönberg and Müller, 2014, demonstrating exceptional measurement reproducibility and simplicity (Garbe-Schönberg & Müller, 2014).

2.3.2.2 Mineralogical characterization

This analysis provides information about the minerals present in a sample, including details on grain size distribution, grain morphology and associations, and the relative abundance of minerals in the sample under investigation. There are two tests that can provide this information: X-ray diffraction (XRD) and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS).

X-ray diffraction (XRD) analysis is used to assess the minerals present in a sample and their proportions by analyzing a pulverized fraction of the bulk sample. XRD is a well-established standard method with significant importance in research and investigations of mineral deposits and their products. It is typically the starting point for mineral characterization. This method is suitable for studying materials with slight variations in mineralogy or mineral chemistry and is also appropriate for coarse-grained samples where performing thin-section analysis is not possible. One of its advantages is its ease of use and rapid results with simple sample preparation (Cook, 2000). X-ray diffraction, a non-destructive technique, is pivotal in mineral characterization, finding broad utility across multiple domains. It offers invaluable insights into crystal structure, size, and orientation, facilitating precise phase identification. Moreover, it enables determination of lattice parameters, evaluation of dislocation density, and quantification of residual stress and strain within materials. Additionally, it aids in studying phase transformations and thermal expansion coefficients, contributing to a comprehensive understanding of mineral properties.(Ali et al., 2022)

Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) allows for the determination of quantitative information about nearly all elements in the periodic table, including trace element concentrations at the ppm level. This method is destructive and requires a larger volume of the analyzed sample. Over the past decade, this technique has been investigated and applied in various areas and for different surface forms (Cook, 2000).

There are various analytical techniques available for examining the minerals present in an ore. These techniques are often performed using optical or electron microscopes. In many cases, these analyses are quantitative, meaning they can identify the minerals in the sample, their associations, and structures without quantifying them. However, advanced microscopic techniques now allow for both identification and quantification of minerals in ores, providing detailed and quantitative information about mineralogy and textures. Standard methods for mineral characterization include the Scanning Electron Microscope-based Automated System (SEM) and Electron Microprobe Analysis (EMPA).

The Scanning Electron Microscope-based Automated System (SEM) is a widely used automated mineralogical system employed in process mineralogy and mineral characterization. This method offers detailed identification of individual minerals by analyzing samples in situ, such as thin polished sections or sample mounts. SEM equipment can provide essential mineralogical information, including modal mineralogy, grain size, mineral associations, and mineral types (Cook, 2000; Gottlieb et al., 2000; Jones, 1970). Globally recognized automated and quantitative software products for SEM analysis include Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN) and Mineral Liberation Analyzer (MLA).

The examination of samples using scanning electron microscopy (SEM) facilitates the detailed identification of individual minerals. This can be done either *in situ* within a thin polished section prepared from a rock sample or within a sample mount created from concentrates or other processing products. SEM also generates optical images that can be further processed and analyzed using image analysis techniques. This allows for the characterization of mineral size, morphology, habit, and associations.

2.4 LIBERATION OF VALUE MINERALS AND LIBERATION MODELS

As previously explained, the objective of comminution is to liberate or release valuable minerals from the gangue minerals as coarsely as possible to facilitate separation in the subsequent processes.

Liberation is measured by the percentage of minerals occurring as free particles in the ore relative to the total content. This concept is known as the degree of liberation. Complete liberation between mineral and gangue particles is rarely achieved through physical methods, even when the ore is reduced to the desired grain size. The properties of grains and grain boundaries influence the difficulty of achieving complete liberation.

Depending on the process and separation techniques employed, a high degree of liberation is not always necessary to achieve high recovery values. Generally, gravity separation processes do not require particles with a high degree of liberation. Separation is possible as long as there is a difference in apparent density between the locked particles and free gangue particles. The same principle applies to magnetic separation, where the magnetic susceptibility difference between gangue and valuable minerals allows for effective separation (Wills & Finch, 2015).

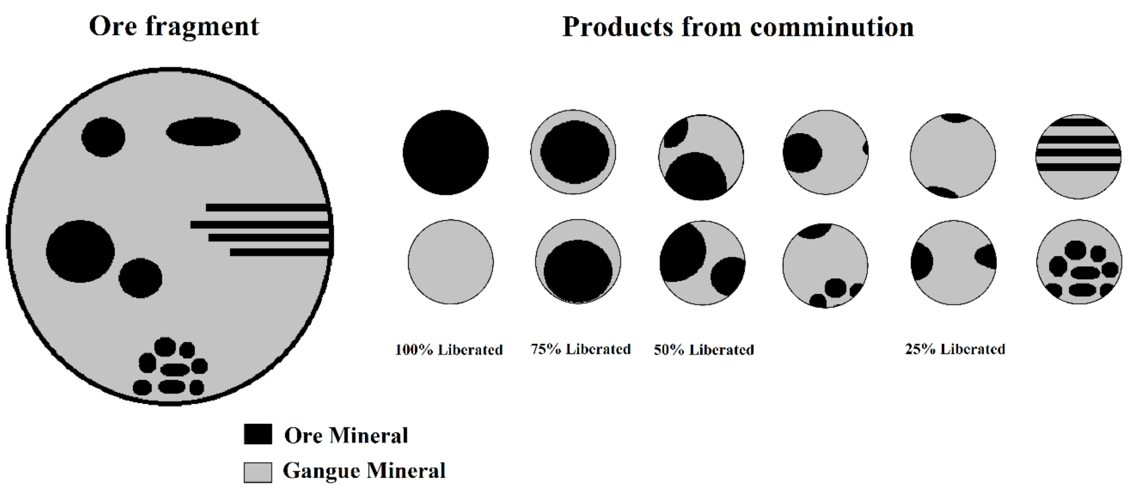

Figure 18. Ore particles. (Wills & Finch, 2015)

Figure 18 illustrates an ore particle with two distinct sections. One section is rich in the valuable mineral (black area), while the other section contains a significant amount of the valuable mineral but is tightly bound to the gangue mineral. As this particle undergoes the comminution process, it will result in liberated mineral fragments and gangue particles, as well as composite particles with varying degrees of valuable minerals. (Wills & Finch, 2015).

Mineral liberation, attributed to Gaudin (1939), involves two primary types: comminution and detachment (Gaudin, 1939). Comminution-based liberation occurs through a random fracture mechanism, where cracks propagate within mineral grains without regard for the various phases present in the ore. When comminution-based liberation occurs, particles within a single-sized class break down into a predictable unimodal distribution of finer particles. Importantly, this type of liberation keeps the compositions of all particles constant, regardless of factors such as ore texture, grain boundaries, or mineral properties.

The second type of liberation is detachment or non-random breakage. In this type of breakage, fractures or crack propagation occur along the grain boundaries, enabling the preferential liberation of mineral phases, often referred to as intergranular fracture. Liberation through non-random breakage leads to varying compositions in different size fractions, which allows for selective comminution techniques. While there are various definitions of non-random breakage, they all result in variations in the mineral composition of different size fractions.

There are two forms of non-random breakage in particle bed breakage: interfacial breakage (grain/phase boundary fracture) and preferential breakage. In interfacial breakage, fractures occur along different grain or phase boundaries, allowing for the preferential liberation of various phases. In the second case, preferential breakage happens when there are differences in the characteristics of phases, resulting in varying rates of breakage based on the soft mineral and hard mineral content of the ore. Soft minerals will break more readily than hard minerals under the same force, leading to different concentrations in the composition of the fine and coarse fractions (Fandrich et al., 1997; Hesse et al., 2017; Mariano et al., 2016; Mariano, 2016).

Mineral liberation is typically measured in the final step of the comminution process when all particles have been reduced to a specific size. Several liberation models attempt to quantify the degree of liberation by predicting how a particle behaves in a separation process, with a primary focus on fine comminution and considering random fracture.

These liberation models, along with their mathematical equations, are rooted in the work of (Gaudin, 1939) and evaluate breakage mechanisms based on grain structure and bonding. Gaudin originally described two types of mineral liberation: comminution and detachment. Subsequent researchers like (Andrews & Mika, 1975; Barbery, 1987; King & Schneider, 1998) continued this research and developed models that aimed to predict the fraction of particles of a specific size containing less than a predetermined fraction of a particular mineral.

These models have laid the foundation for contemporary research, but they do come with certain unrealistic assumptions about mineral grain structure. Subsequent researchers, such as (Wei & Gay, 1999) (Gay, 2004) and (Gay & Latti, 2006), introduced multi-mineral systems and developed models that apply to various breakage mechanisms. Their studies suggest that liberation analysis and modelling are valuable tools for determining the optimal particle size for comminution units, particularly for fine particles. They established connections between liberation distribution, ore texture, and the dispersion rate function.

Other studies, like the one conducted by (Fandrich et al., 1997), explored how mineral liberation is affected by particle bed breakage. Liberation analysis and modelling prove beneficial in determining the ideal grind size for comminution units, especially for fine particles, and they provide insights into the relationship between liberation distribution, ore texture, and dispersion rate function.

In addition to liberation analysis and prediction, it's crucial to understand other fundamental concepts, such as fracture processes, mineral concentration, breakage mechanisms, and the behavior of various minerals (Fandrich et al., 1997). Researchers have identified different types of ore breakage, including random and selective breakage. In random breakage, fractures occur randomly without considering the different phases present in the ore. Conversely, in selective or non-random breakage, fractures are preferential, leading to different concentrations in the composition of particles of varying sizes (Mariano, 2016) summarized perspectives on random and non-random breakage in his work. More recent studies by (Vizcarra et al., 2010) and (Ozcan & Benzer, 2013) evaluated mineral liberation using different breakage mechanisms.

Mineral liberation is primarily observed in finer-sized particles, making all liberation models applicable to fine comminution processes. However, during coarse comminution, liberation is thought to be less significant. Still, the mineral concentrations in different particle sizes can influence liberation performance in later stages. A recent study by C. Carrasco et al. (C. Carrasco et al., 2016) introduced a coarse liberation model based on preferential grade by size response. This model ranks the response parameter of the ore as a function of particle size distribution and size reduction process using the Monte Carlo method. Another study by (Bazin et al., 1994) suggests that certain ores preferentially distribute metal into specific size fractions, supporting the development of a mineral concentration model based on a third-order polynomial fit. In recent research conducted by (Hesse et al., 2017), selective breakage was investigated using quantitative microstructural analysis (QMA), offering a detailed analysis of issues related to selective breakage, such as mineral geometry and orientation within the structure.

2.5 SELECTIVE COMMINUTION

Selective comminution allows the separation of the material with low valuable mineral content at early stages in the comminution processes by taking advantage of the differences in mineral behaviour of an ore, for example, the differences in the comminution behaviour of the mineral that conforms the ore.

Depending on the processed material and its physical and chemical properties, it might be possible to conduct an early separation, such as a pre-concentration of valuable minerals or by valueless gangue rejection at coarse sizes at the earliest stage of the process flow (Boundy et al., 2017; Bru & Parvaz, 2018; Hesse, 2018; Hesse et al., 2015; Hesse et al., 2017; Lieberwirth et al., 2020; Ni et al., 2022; Waters et al., 2017). The separation may be possible by adding a classification step and producing a pre-concentrate with the valuable mineral high content. An early separation means that not all materials must follow all processes and machines. By adding a pre-classification stage during an early stage of the process, it is possible to reduce the handling and further size reduction of waste material, which means reducing mass flow rate in some parts of the process, thereby reducing the amount of water and energy required during the entire process (Ballantyne, Hilden, et al., 2012; Ballantyne, Powell, et al., 2012; De Kretser et al., 2009; Franks et al., 2015; Johnson, 2018).

Depending on the processed material and its physical and chemical properties, it may be feasible to implement early separation techniques, such as pre-concentration of valuable minerals or the rejection of low-value gangue materials in the coarser size fractions at the initial stages of the process flow. Achieving this separation often involves introducing a classification step and generating a pre-concentrate with a high content of valuable minerals. Early separation allows certain materials to bypass subsequent processing steps and machinery.

Incorporating a pre-classification stage at the early phase of the process offers several benefits, including the reduction of material handling. This reduction in mass flow rate in specific parts of the process leads to decreased overall water and energy consumption throughout the entire process.

Selective comminution represents a promising approach in the processing of low-grade ores. Successfully implementing selective comminution requires a deep understanding of the mineral composition and ore properties. Material properties play a crucial role in this process, but it's equally important to employ appropriate technologies, design machinery accordingly, and optimize operational parameters (Hesse et al., 2017).

Some ores exhibit a tendency to distribute metal minerals into specific size fractions, allowing for the early rejection of low-grade materials (Bamber et al., 2008; Bowman & Bearman, 2014; Bru & Parvaz, 2018; Burns & Grimes, 1986; C. Carrasco et al., 2016; Cristian Carrasco et al., 2016; Hesse et al., 2017). Selective comminution relies on understanding the distinct properties and behaviors of the minerals within the ore. Recognizing how compressional breakage impacts mineral liberation and the concentration of valuable minerals is a crucial initial step in identifying ores suitable for pre-concentration.

The presence of various minerals in an ore, each with distinct breakage rates or behaviors (such as hard and soft minerals), can result in differences in the grindability of the ore and its constituent minerals. Even minor stress intensities and shear forces have been shown to be adequate for liberating soft mineral associations or breaking relatively weak boundaries. Consequently, it's possible to obtain a mineral product with a high metal content in the finer fractions (Kwade & Schwedes, 2002). Soft minerals tend to achieve liberation more quickly under the same stress conditions, particularly through mild abrasion or attrition forces (Tong et al., 2013). In summary, minerals with differing breakage behaviors may preferentially distribute into finer fractions, while others may remain in coarser fractions, leading to variations in grade among the fractions (Lieberwirth & Kühnel, 2021).

Selective comminution typically requires that the content of valuable minerals varies significantly in specific size fractions. To determine suitable separation criteria, one can analyze the variation in mineral content within each size fraction (Fandrich et al., 1997). There are various methods for evaluating whether selective comminution is applicable to a particular ore type or process. The most common approach involves examining how the distribution of valuable and waste minerals is influenced by particle size. This method necessitates the integration of cumulative particle size distribution data to distinguish between the waste and valuable components.

2.6 TECHNICAL ECONOMICAL MODEL

In mineral processing, it is common to encounter operations that generate profits lower than their production costs, particularly when dealing with low-grade ores. Moreover, the complex conditions faced by various mining companies often lead to significant reductions in production efficiency. Numerous factors contribute to the overall cost of the process, including natural elements and the entire technological chain involved in mining and processing (Vagonova & Volosheniuk, 2012; Voloshyna & Kostakova, 2017). Today, the evaluation and reduction of mining costs have become even more crucial due to the increased demand for critical metals and stricter environmental regulations (Huang et al., 2008).

Technical analysis in mineral processing encompasses a wide range of information and considerations related to materials and plant operations. This includes aspects like particle size distribution, optimizing plant yield, assessing plant efficiency, designing process flowsheets, evaluating mineral liberation, and analyzing equipment performance. Other factors that fall under technical analysis include manipulating washability data, analyzing partition curves, maximizing plant efficiency, evaluating flowsheets, simulating mineral liberation during comminution, simulating classification processes, and assessing the performance of key plant components. This involves various stages like crushing, screening, classifying, washing, dewatering, and drying.

Conversely, economic analysis involves the assessment of both investment and operational costs associated with equipment, installation, maintenance, and equipment replacements (Huang et al., 2008).

2.7 REFLECTIONS ON THE FRAME OF REFERENCES

In the Frame Reference chapter, various concepts related to the research topic have been established. Currently, numerous methods are used to measure and test mechanical properties, both before and after coarse comminution, as well as to measure elemental and chemical properties of ores after fine comminution processes. It's evident that these methods are interconnected, as previously discussed in the Introduction chapter. Upon reviewing these related concepts, the necessity of measuring and integrating ore characteristics into coarse comminution processes and models remains evident.

3

SCIENTIFIC APPROACH

This chapter objective is to:

- *Explain the research approach used in this thesis.*

This work has been conducted at the Chalmers Rock Processing Systems (CRPS) research group within the Department of Industrial and Materials Science at Chalmers University of Technology. The research focuses on equipment and processes for producing crushed rock materials, primarily for application in the mining and aggregate industry (Asbjörnsson, 2013; Bengtsson et al., 2009; Evertsson, 2000; Hulthén, 2010; Lee, 2012; Quist, 2017; Svedensten, 2004). The scientific approach adopted in this research is characterized as a problem-oriented research methodology. In problem-oriented research, the methods chosen to address the problem are based on the nature of the problem itself, rather than being driven by specific tools or techniques. This approach has been described by (Evertsson, 2000). Figure 19 illustrates the schematic diagram depicting the various steps involved in the problem-oriented research methodology.

Research involves conducting a comprehensive investigation to uncover new information or achieve a higher level of understanding. This quest for new knowledge and deeper comprehension is typically driven by the presence of a problem that demands a solution. In such cases, existing knowledge falls short, necessitating a more profound exploration. A problem-oriented research methodology enables the systematic application of the research process, often involving multiple iterations at various activity levels.

In accordance with a problem-oriented research methodology, the initial step involves the identification of a knowledge gap, which becomes evident through the presence of a problem that requires resolution to yield improvements or benefits. Importantly, the term "problem" does not exclusively denote a malfunction; it can also pertain to opportunities for enhancement, increased productivity, or streamlining processes to make them more efficient and quicker. Subsequently, the next step entails an examination of the real-world circumstances and physical principles associated with the specific problem. Various forms of observation serve this purpose, including literature reviews, experiments, observations, and data analyses. Literature reviews serve as the foundation for understanding existing knowledge within the research area, while experiments can shed light on the nature of the problem. Data acquisition or experimental findings can be evaluated and employed in constructing a theory or model. (Hulthén, 2010; Quist, 2017)

Experiments have played a crucial role in this project, serving as a vital means to clarify and evaluate the underlying processes. All observations related to the problem have been grounded in experimental tests and interactive data collection. Through the establishment of a well-structured series of experiments and the subsequent analysis of the acquired data, it became feasible to comprehend the physical principles influencing the process and various interactions. This understanding ultimately facilitated the development of a new and improved testing methodology.

As a result, various methods and models are evaluated and developed. These models encapsulate the observed phenomena through mathematical formulations based on physical measurements and behaviors discovered during the research. The goal is to select the most suitable method and model based on the nature of the problem. This involves an iterative process where several potential methods are tested to identify the most effective one. This iterative approach allows for the exploration of new interactions that may lead to potential improvements.

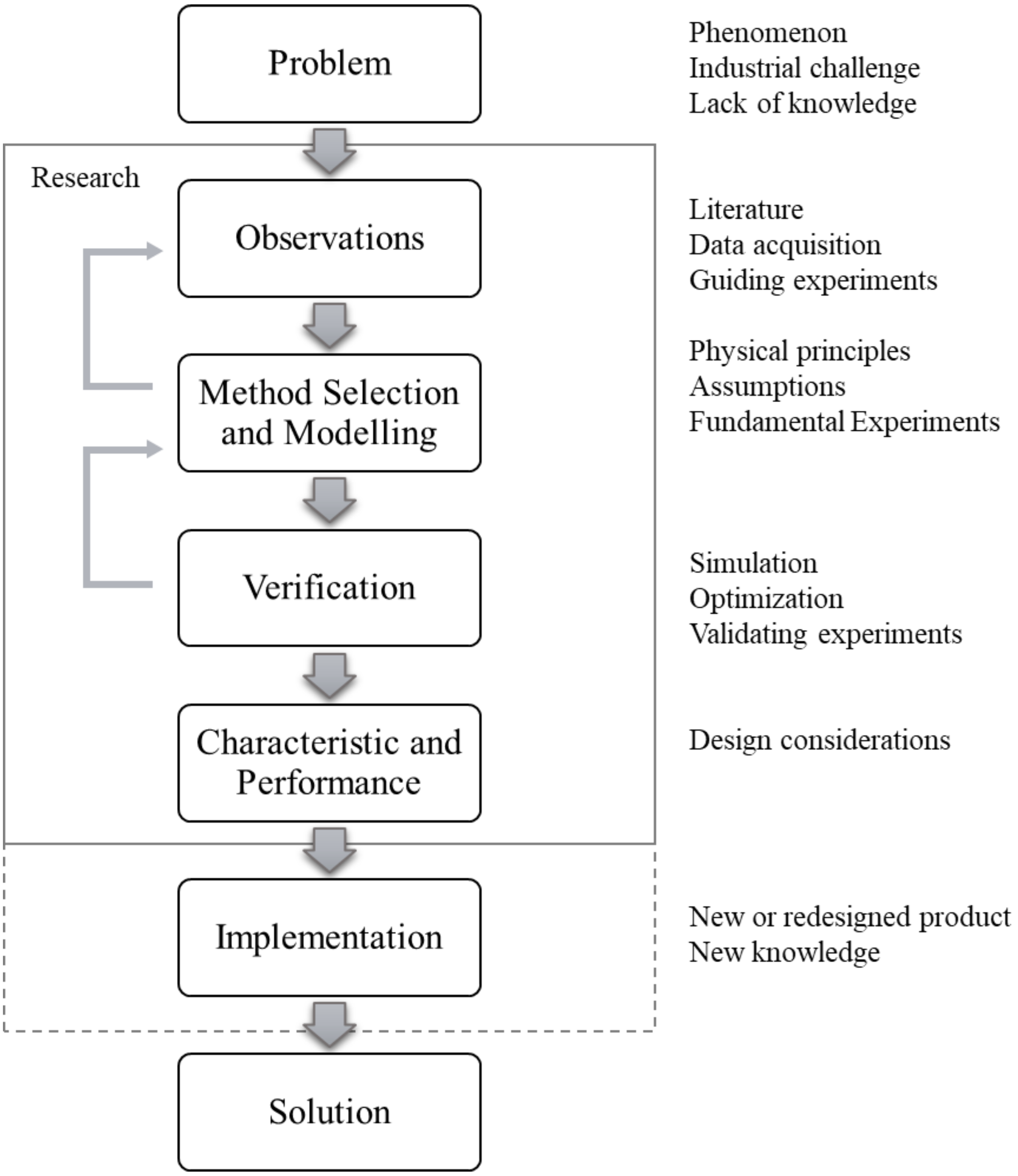

Figure 19. Structure of problem-based research approach (Evertsson, 2000).

The results obtained through these methods and models are then tested and verified, often through case studies or real-world industrial applications. Once a validated model is in place, simulations can be performed to gain additional insights and knowledge, with the aim of enhancing performance. It's worth noting that, in this particular project, simulations were not conducted, which is considered a limitation and could be addressed in future project work.

It is important to emphasize that research is an evolutionary process (Hulthén, 2010), meaning that the research aims and problems were not fixed from the outset but evolved and adapted to new needs and understandings over the course of the five-year research. Initial hypotheses that were confirmed served as the foundation for developing new hypotheses. Through this iterative process of exploration, new knowledge is generated and can be implemented, ultimately leading to the resolution of the original problem (Bengtsson, 2009; Lee, 2012).

4

RESEARCH FINDINGS

The objective of this chapter is to:

- *Provide the core findings from the appended papers.*

4.1 SUMMARY OF THE APPENDED PAPERS'

The summary presented in this chapter concentrates on the results and critical contribution shown in Figure 20 and Figure 21 (Detail procedure). The papers answer some research questions and contribute to relevant information about and evaluation of the research topic.

A summary of the contributions of each paper is listed below:

In Paper A, a mathematical model is developed by which to predict the element concentration in different size fractions after compression breakage.

Paper B explains the development of a test protocol with which to characterize the grade by size variation during compression breakage.

Paper C explains the development of a test protocol capable of measuring the concentration of refractory elements.

Paper D presents the development of a technical, economical analysis capable of simulating a crushing plant performance by predicting yield, product quality and process cost.

The foundation of this research lies in the quest to develop an efficient method for producing critical metals through a process known as selective comminution. This study draws upon the findings of four distinct papers, each contributing to improving the extraction of critical metals. To assess and improve the extraction of critical metals, this endeavor demands three essential components: energy efficiency, cost-effectiveness, and process optimization.

Achieving these objectives has necessitated a synthesis of measurement perspectives and process perspectives. The measurement perspectives involve ore characterization, enabling the determination of both mechanical and chemical properties. This critical data is acquired through the application of precise testing protocols, an area thoroughly explored in articles A, B, and C.

In addition to measurement, the research incorporates the development of models and predictions, as featured in papers A and D. These predictive tools serve to enhance the understanding of critical metal extraction processes, by making possible the prediction, control and optimization.

Finally, the research embraces a process perspective, acknowledging that efficient metal extraction involves more than just measurements and predictions. It involves the careful planning of various factors to create an efficient and effective production process.

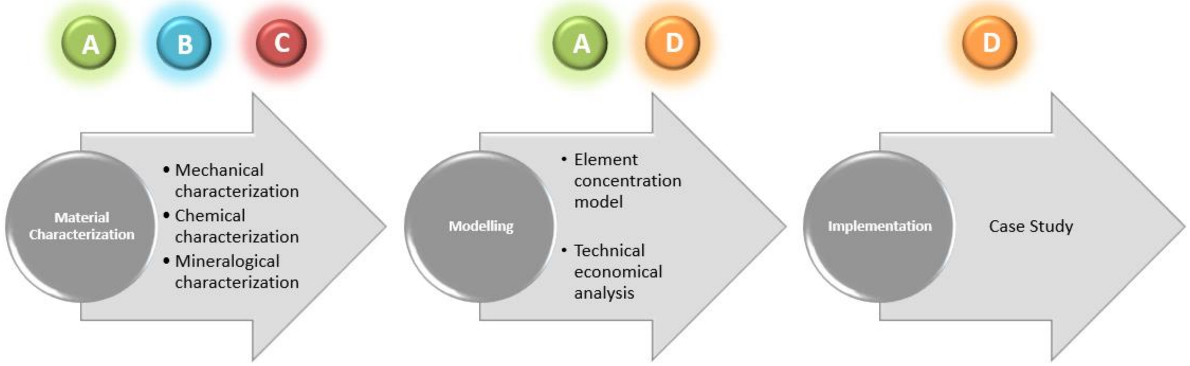

Figure 20: Test procedure presented in this thesis related to the papers appended.

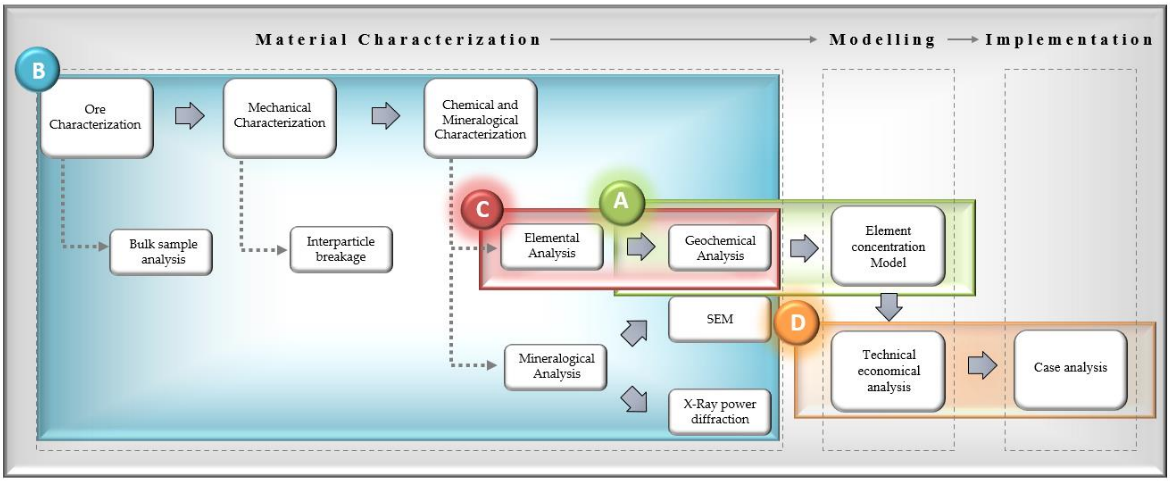

Figure 21: Material characterization and detailed test procedure presented in this thesis related to the papers appended.

4.2 CORE FINDINGS FROM THE APPENDED PAPERS

4.2.1 Paper A

Title: Analysis of concentration in rare metal ores during compression crushing.

Background and challenge

The existing models for coarse comminution are capable of predicting size reduction, size distribution, capacity, and energy consumption. However, there is a need to enhance the resolution of crusher models by considering or incorporating new factors that have not been previously addressed. One crucial factor is the concentration of elements and minerals within the ore.

Through the characterization of different ores, it has been observed that the distribution of individual elements is neither homogeneous nor consistent across various size fractions. This observation opens new possibilities for modelling the distribution of critical elements following compressive breakage.

Currently, coarse comminution models primarily focus on predicting size distribution post-comminution. However, there is a gap in understanding what happens to critical metals and how they are distributed among size fractions during comminution. This knowledge gap also translates into a lack of models capable of predicting the concentration of critical metals in the context of coarse comminution. Addressing this gap would be a valuable contribution to the field.

Key contribution

Paper A introduces a mathematical model that predicts the concentration of critical metals as a function of particle size distribution, decoupling it from mass considerations after a compression breakage process. Paper A establishes a framework for analyzing geochemical data using a cumulative distribution function for normalized values. This approach allows for the modelling of normalized concentration behavior concerning particle size distribution through a bimodal Weibull distribution, effectively eliminating the need to consider mass relationships. It also helps mitigate discrepancies due to material variations and sampling errors.

The key contribution of Paper A is the elemental concentration analysis. By simulating absolute elemental concentrations, this model enhances the resolution of coarse comminution models. However, to fully leverage its potential, this elemental concentration model needs to be integrated with other components within crushing models. Typically, coarse comminution models can predict process data (output) based on known operating parameters (input). These predictions are often limited to energy consumption, capacity, and particle size distribution. The incorporation of material properties for predicting element concentration represents a significant advancement in modelling capabilities.

The methodology introduced in Paper A for analyzing geochemical data, involving the use of a cumulative distribution function for normalized values, is a significant contribution. It allows for the modelling of normalized concentration behavior with respect to particle size distribution, and this behavior can be effectively captured using a bimodal Weibull distribution. Importantly, this approach eliminates the need to consider mass relationships, providing a more accurate representation of the concentration distribution. It also helps mitigate discrepancies arising from material variations and sampling errors.

Figure 23 in Paper A presents a flowsheet of the elemental concentration model, demonstrating how this model can be integrated into coarse comminution models. By simulating absolute elemental concentrations, this model unlocks new possibilities and applications, while also enhancing the resolution of coarse comminution models. However, to fully harness its potential, it should be integrated with other modelling components within crushing models. Coarse comminution models typically predict process data (output) based on known operating parameters (input), However, these predictions often focus on energy consumption, capacity, and particle size distribution. The inclusion of material properties for predicting element concentration enables an increased model resolution as shown in Figure 22.

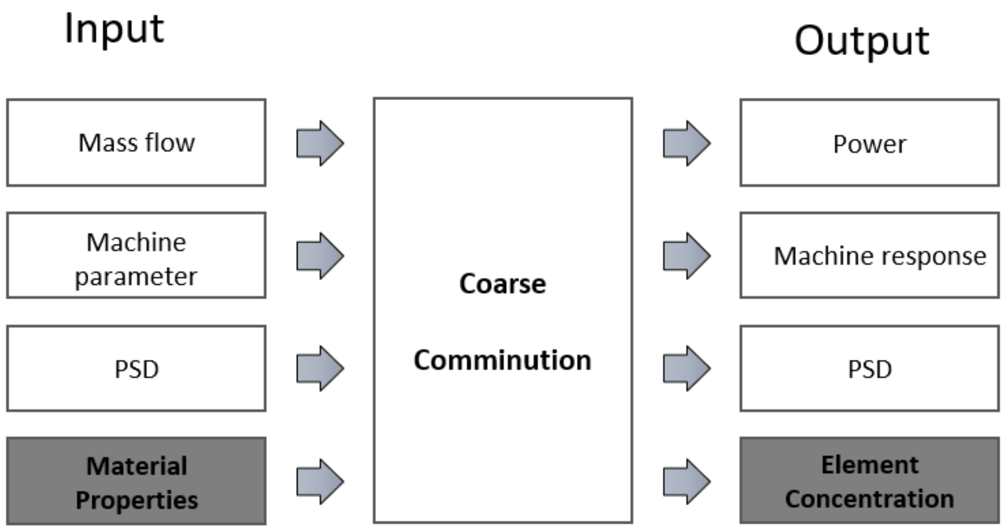

Figure 22. Predictive modelling parameters for coarse comminution. The grey areas show the new addition to the model.

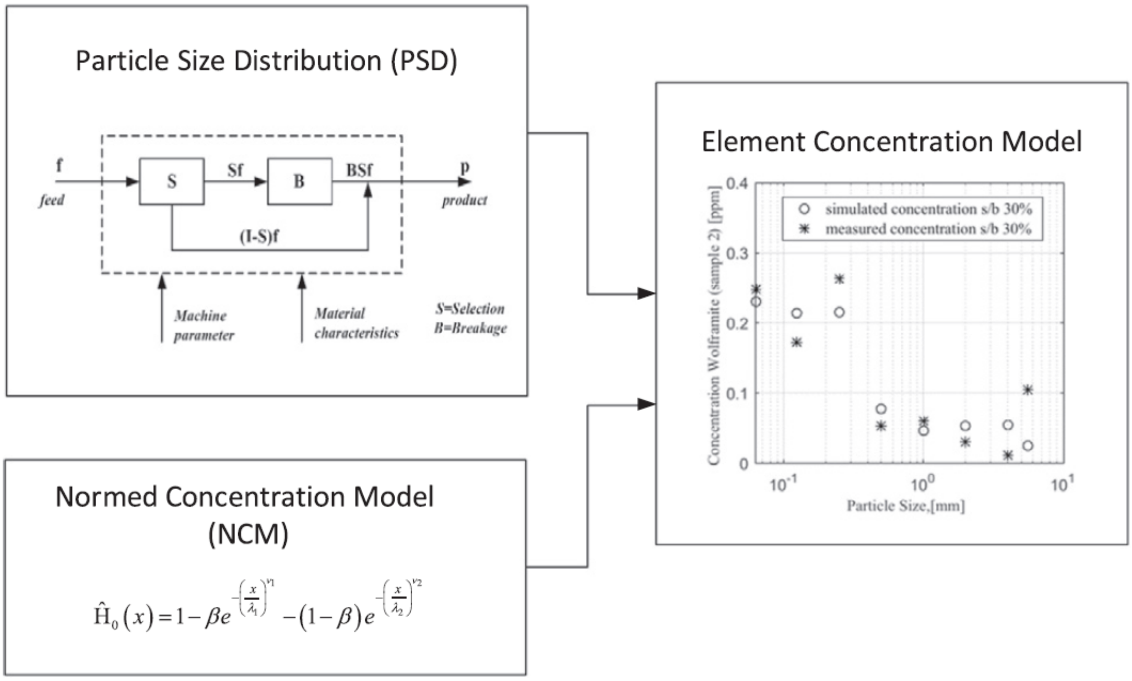

Figure 23: The flowsheet for calculating the elemental concentration.

Discussion

Positive review

In Paper A, the development of a mathematical model for predicting element distribution in coarse comminution is a valuable contribution with the potential to streamline testing procedures. Once a thorough understanding of ore behavior has been established and validated through testing, the mathematical model can serve as a predictive tool. This can significantly reduce the need for extensive and time-consuming testing, as the model allows for the prediction of behaviors and requirements based on established knowledge. This approach not only saves time and resources but also provides a more efficient and cost-effective means of managing and optimizing coarse comminution processes.

Critical review

The modelling presented in Paper A requires the support of material testing and physical measurements of the ore, which a model can never replace. Ores vary in behavior, and it's essential to conduct in-depth studies and gather data through physical testing to gain knowledge before applying the model.

Furthermore, the model presented in Paper A can simulate absolute elemental concentrations. However, it should be integrated with information from other modelling components in the crusher model to provide a more comprehensive understanding of the process and offer improvements during the crushing stage.

4.2.2 Paper B

Title: Understanding mineral liberation during crushing using grade-by-size analysis - a case study of the Penuota Sn-Ta-Nb mineralization.

Background and challenge

Particle size distribution is the sole evaluated property during coarse comminution. The breakage of particles is affected by mineralogy and texture. Nevertheless, there is a lack of knowledge about the breakage of various minerals and even less information about the distribution of critical metals.

This paper also addresses the results obtained regarding minerals, minerals association concerning critical metal minerals, fracture of the phases, and liberation of the particles in different size fractions.

Key contribution

The development of a grade-by-size methodology applicable to coarse comminution is achieved by combining well-known test procedures and techniques. This paper encompasses several properties that must be observed and measured to assess element distribution. These properties include the mechanical characterization via interparticle breakage (IPB) of the ore, which involves laboratory compressive breakage. Following this, there is a mineralogical and chemical characterization of the sample post-IPB.

Paper B delves into the possibility of developing a test protocol for characterizing the grade-by-size variation during compression breakage. This test protocol evaluates scenarios for early waste material rejection by assessing material behavior after a series of compressive breakages. Figure 23 illustrates a flowsheet of the elemental concentration model.

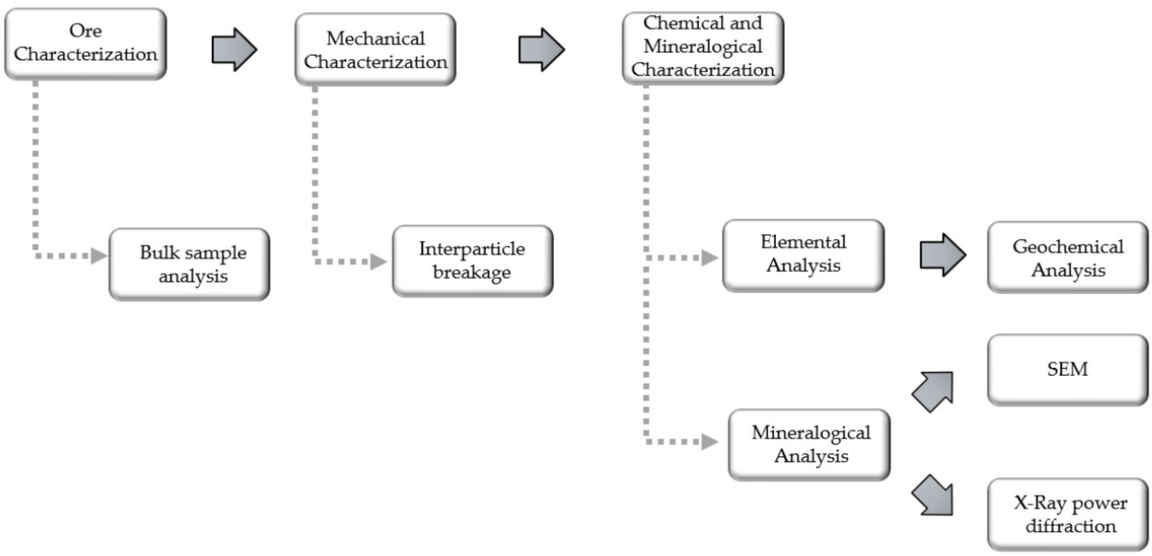

Figure 24: Method for analyzing the mineral and element composition in compression breakage.

Discussion

Positive review

Paper B has analyzed element distribution during coarse comminution, which opens up new opportunities for ore processing. A simple and economical method that provides this information can create possibilities at various stages of the process. This can be especially beneficial in the planning, design, and operation of early process steps. It could also prove helpful in already functional processing plants where selective comminution might be applied. Furthermore, it could aid in the rearrangement of the operations circuit.

Critical review

While Paper B suggests an excellent and economical approach to acquiring knowledge about ores, it's important to recognize that each ore is unique and behaves differently. Therefore, a comprehensive understanding of the ore through physical testing is necessary. However, it's worth noting that some ores exhibit a homogeneous distribution after breakage, resulting in no accumulation of elements in different fractions. In such cases, applying a grade-by-size approach may be impossible, and the testing methodology proposed in Paper B may not be applicable or beneficial for all ores and processes.

4.2.3 Paper C

Title: Evaluation of tungsten and tantalum content through nano-particulate pressed power tablets for LA-ICP-MS.

Background and challenge

Refractory metals, such as tungsten and tantalum, pose analytical challenges when it comes to measuring their chemical composition. Analyzing these metals necessitates a fused bead and acid digestion of samples, followed by the use of inductively coupled plasma mass spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS). These techniques are time-consuming, complex, and expensive.

Key contribution

A novel analytical approach was developed for measuring refractory metals, involving the production of nano-particulate pressed powder tablets (PPP) followed by LA-ICP-MS analysis. Paper C assesses the feasibility of measuring the geochemical composition of ore using this method. It provides the same information as bulk chemical analysis, covering major, minor, and trace elements, while requiring less time, effort, and cost. This technique simplifies the process by eliminating the need for fused bead and acid digestion methods and inductively coupled plasma mass spectrometry (ICP-AES), without omitting valuable information.

Figure 24 schematically illustrates the sample processing and measurement protocol employed in the study. Paper C primarily focuses on creating powder pressed pellets through nano-grinding and evaluates various methods for drying the suspension sample, including freeze-drying (FD), evaporation, vacuum-driven sterile filter (VDSF), and vacuum-driven with a membrane filter (VDMF). Additionally, it explores the advantages of using binders during pellet production. Lastly, it assesses and compares the behavior of pellets made with or without a binder, considering different homogenization times between the binder and ore powder (1 min, 5 min, and 10 min). This protocol offers a simple, fast, and cost-effective means of measuring refractory elements.

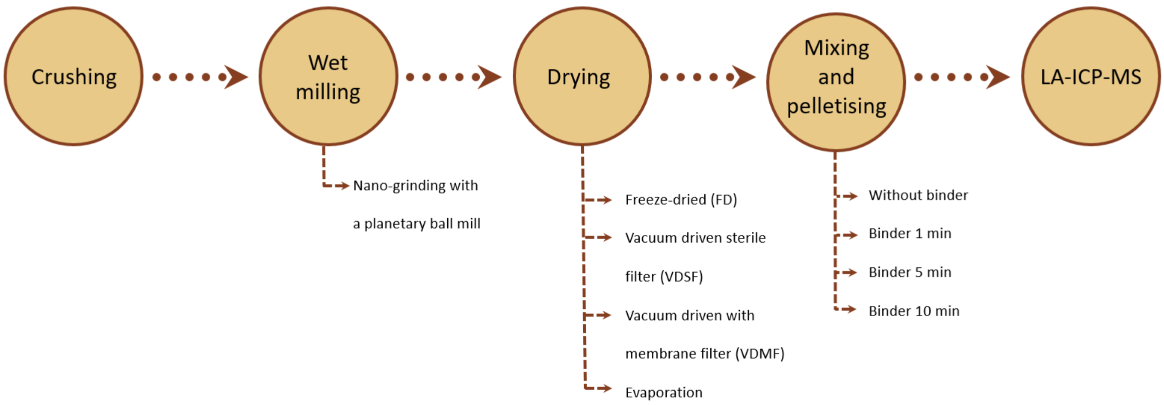

Figure 25: Flow chart of sample processing and measurement protocol process.

Discussion

Positive review

Paper C presents an efficient testing method for PPP LA-ICP-MS, indicating its potential as a straightforward alternative to ICP-MS and ICP-AES techniques. Moreover, this method provides equivalent information about the content of refractory elements through a simple, fast, and cost-effective process, eliminating the need for expensive and challenging techniques like fused bead and acid digestion of samples.

Critical review

While the method presented in Paper C offers simplicity, the value results obtained show higher errors than standard methods. There is a compromise between accuracy, speed, and cost-effectiveness in this technique. This doesn't mean it may not be the right solution; it simply may not be suitable for all cases, particularly when high accuracy is required.

4.2.4 Paper D

Title: Selective Comminution Applied to Mineral Processing of a Tantalum Ore: A Technical, Economic Analysis*.*

Background and challenge

Techno-economic analysis plays an essential role in plant design and operations; however, there is limited work, insufficient research, and a lack of tools to understand the effect of economic factors and criteria on the optimality of flowsheets. Furthermore, there is minimal knowledge and few tools available for evaluating the process in terms of equipment, capacity, material distribution, and associated costs. This limitation hinders our ability to optimize how a crushing plant should be controlled for efficient process utilization.

Key contribution

Paper D describes the methodology that combines a technical and economic model to evaluate the possibility of rearranging mass flow based on the mineral composition of the rock, thereby improving profit. Paper D suggests a schematic protocol for the dominant parameters in a crushing plant that use selective comminution to influence product quality, including yield, product quality, and process cost.

The primary benefit of Paper D is the methodology used to calculate cost parameters as a function of mass flow for each piece of equipment, allowing for the calculation of a plant's production cost. The techno-economic analysis focuses on predicting the yield of the plant configuration, product quality, and process cost. Two cases are analyzed from both technical and economic perspectives, each with different plant configurations.

The two configurations are based on an existing plant (Case 1) and a proposed change in the process using selective comminution techniques to achieve early separation and classification (Case 2). The resulting comparison of production costs for both cases is presented in Figure 25.

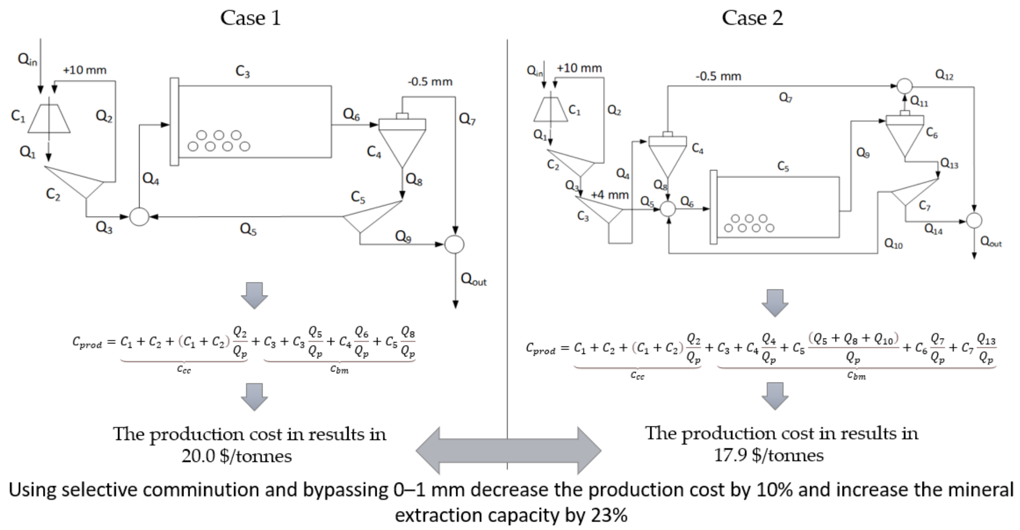

Figure 26: Comparison cases of a configuration of a plant, cost calculations, and result of the production cost.

Discussion

Positive review

In Paper D, a proposed framework indicates and assists in establishing the best practices for evaluating plant quality, capacity, and arrangement to achieve optimal economic plant performance.

Reducing the amount of material in the ball mill will decrease the required energy and associated costs. This paper introduces a tool capable of demonstrating the cost-benefit of using selective comminution. Paper D enables the evaluation of selective comminution in a mineral processing plant.

Paper D describes an open tool that can be applied to any extraction plan without limitations in terms of the type of mineral and size fractions, among others. It can also be used to simulate or analyze other dynamic processes in a crushing plant to optimize not only capacity and cost but also yield and product quality.

Critical review

The tool presented in Paper D benefits from additional sources of information, such as the incorporation of supplementary modelling and simulation components. Examples of these components include classification and screening models.

A case study is provided for the extraction of tantalum ore, which serves as an illustration for critical metals with low grades. However, in certain cases or with different types of ores, selective comminution may not yield economic benefits for flow division.

Furthermore, a preliminary analysis of the material is essential, encompassing factors like rock type, mineral composition, size reduction characteristics, and enrichment fractions, among others. Therefore, this model should be used in conjunction with prior studies, analyses, assessments, and models, all of which need to be acquired and understood before implementing this tool.

5

DISCUSSIONS AND CONCLUSIONS

This chapter objective is to:

- *Present the most critical findings presented in this thesis.*
- *Answer and discuss the research questions stated in Chapter 1.*
- *Present the scientific contribution and claims proposed in this thesis.*
- *Discuss finding crucial to future work.*

The primary hypothesis of this research is that using selective comminution could be possible to improve the critical metal extraction efficiency, making it a valuable and cost-effective method compared to conventional approaches. The application of selective comminution techniques, preceded by comprehensive ore characterization and aided by predictive models and advanced analytical methods, result in a significant enhancement of critical metal extraction efficiency compared to traditional extraction processes. This enhancement will not only improve resource utilization but also demonstrate cost-effectiveness, making it a viable approach for meeting the growing demand for critical metals.

Critical metals are not uniformly distributed across various size fractions during coarse comminution processes. This uneven distribution results from the influence of factors such as mineralogy, texture, grain shape and size, breakage mechanism, and element concentration on particle breakage. Consequently, there is a need for a protocol to enhance coarse comminution processes and models by systematically measuring and integrating ore characteristics.

5.1 GENERAL

The research results are geared towards introducing an analytical method to enhance the assessment of coarse mineral and elemental characteristics by analyzing how different ore materials break after undergoing compressive crushing, particularly in terms of minerals and elemental distribution. The protocol encompasses the mechanical characterization of particles post-compressive breakage, along with mineralogical and elemental analysis. This procedure facilitates the measurement of elemental concentrations and mineral identification within various size fractions following a series of compressive crushing stages.

Additionally, we have developed a model capable of analyzing critical metal concentrations as a function of particle size distribution. The assessment of element concentrations across different size fractions opens the possibility of employing selective comminution during ore processing. When used judiciously, selective comminution has the potential to reduce production costs and enhance processing capacity.

Furthermore, we have conducted a technical and economic analysis to estimate the feasibility of improving profitability by optimizing mass flow, considering the mineral composition of the rock.

Some general findings are formulated below:

- All ores exhibit unique characteristics and behave differently during and after compression. The test methodology developed in this study enables the interpretation, evaluation, and comparison of other ores with varying behaviors and properties by analyzing similar parameters. Having a standardized protocol that facilitates the assessment of ore behavior in both mechanical and chemical terms allows for the comparison of how different ores perform. This, in turn, aids in the identification of the most effective processing techniques.

- Ores are composed of various minerals, each of which exhibits distinct characteristics such as strength, hardness, and brightness. These specific properties govern the behavior and breakage patterns of each mineral. When different types of ores contain minerals with similar behaviors, they may exhibit comparable breakage patterns and mineral accumulation in various fractions. Minerals that are prone to easy breakage typically result in finer particle sizes and tend to accumulate in the finer size fractions.

- The distribution of critical metals following a series of compressive breakage processes has been assessed. It has been observed that critical elements do not exhibit uniform distribution across various size fractions during coarse comminution processes. This observation opens the door to more effective and informed processing techniques, such as selective comminution or circuit operation rearrangements.

- By generating nano-particulate pressed powder pellets followed by analysis using Laser Ablation Inductively Coupled Plasma Mass Spectrometry (PPP LA-ICP-MS), it is feasible to measure the content of refractory elements. This method offers a simple alternative to ICP-MS and ICP-AES techniques, providing the same information about refractory element content through a fast and cost-effective process. However, it's essential to find a balance between accuracy, speed, and cost-effectiveness when considering this technique. While it may not provide the highest accuracy, it can still be a suitable solution for many cases, especially those where ultra-precision is not necessary.

- After a series of compressive breakages, the cumulative distribution function for normalized values can be used to represent the distribution of critical metals. Normalized frequency distribution eliminates the need to consider mass relationships and helps minimize discrepancies arising from material variations and sampling errors.

- Different types of ores exhibit varying mechanical and chemical behaviors, yet they often share similar properties that can be analyzed and modeled for their critical metal content. A mathematical model has been proposed to predict the concentration of critical metals as a function of particle size distribution following a compression breakage process. This modelling approach offers the potential to streamline testing procedures but does not imply the replacement of material testing and physical measurements of the ore, which remain essential.

- Previously, mass flow, machine parameters, and particle size distribution were utilized as inputs to the model, predicting power, machine response, and final particle size distribution. The incorporation of the concentration model has notably enhanced the resolution of the comminution models, as material properties can now serve as an input parameter to yield element concentration as an output. However, it is worth noting that the modelling of critical metals could be further refined by integrating it with information provided by other modelling components within the crusher model, thereby potentially increasing the overall resolution.

- Selective comminution can enhance the mineral processing of critical metals by introducing a pre-classification stage at an early comminution phase. This preliminary classification step has the potential to reduce the final product size, subsequently leading to reductions in energy consumption and production costs.

- Incorporating a theoretical framework that considers both mass flow and operating costs has significantly improved the analysis, optimization, and evaluation of process performance. This approach enables a more thorough assessment of structural design in terms of equipment and processes.

5.2 ANSWERS TO RESEARCH QUESTIONS

The following answers are given to the research questions stated in Chapter 1 of this thesis:

RQ 1) What protocol would be suitable for identifying the benefits of a selective comminution process?

In most cases, materials undergo size reduction during both coarse and fine comminution processes. However, the evaluation of valuable mineral composition typically occurs at the end of the process, even after fine comminution when particles have been reduced to micro or nano sizes.

The liberation of ore minerals depends on factors like rock texture and differences in size and mechanical properties between valuable minerals and gangue minerals. These factors can lead to specific particle sizes during breakage, influenced by the relative composition of these minerals. For example, the comparison between gneiss and granite reveals anisotropic behavior in gneiss, potentially resulting in different mineral compositions in fines.

After rock comminution, critical metals are not uniformly distributed among different size fractions due to the varying breakage patterns influenced by mineralogy and texture. As ore is broken into different particles, their behavior during breakage depends on the initial rock texture and their specific properties. Consequently, different fractions may exhibit distinct compositions. The remaining material often consists of locked or semi-liberated critical metal particles, enhancing the grade of specific fractions. This variability in mineral composition within coarse comminution necessitates a testing procedure that quantitatively measures the composition of valuable elements in each fraction, enabling classification of high and low-grade fractions.

Given the uneven distribution of critical metals across different size fractions, the development of a protocol capable of evaluating mineral and element distribution during and after compression breakage becomes crucial. Such a protocol would provide comprehensive insights into the grade-by-size distribution of critical metals.

Selective comminution involves the possibility of preconcentration by segregating and discarding material with low or negligible mineral content at an early process stage, often by incorporating a classification step.

RQ 2) What properties should be analysed during coarse comminution testing to guide the early selective comminution process? Furthermore, what methods and protocols can guide the coarse comminution process in order to improve the processing of critical metals?

Traditionally, the only material property analyzed during coarse comminution processes has been particle size distribution. However, it is now evident that during the reduction stage, particles undergo fracture, leading to varying distributions of valuable metals among different size fractions.

To characterize the grade-by-size variation during compression breakage, it becomes essential to evaluate specific properties related to element and mineral distribution. This evaluation encompasses mechanical, mineralogical, and chemical characterizations of the sample.

The mechanical characterization involves examining how particles break and the resulting size distribution. But first, it's crucial to understand what transpires within these particles at a chemical level. Achieving this necessitates an analysis of the minerals present during fracture at different sizes and their elemental content.

While several methods can enhance coarse comminution, not all offer relevant and cost-effective information. For instance, the protocol must evaluate mineral and element content across various size fractions. Techniques like QEMSCAN provide similar data but often require a substantial number of samples, making them economically unfeasible.

Conversely, an extended analytical protocol has been introduced, combining a well-established mechanical test for interparticle breakage with an enhanced assessment of coarse mineral liberation characteristics. As previously established, the liberation of ore minerals depends on rock texture and the differences in size and mechanical properties between valuable minerals and gangue minerals. These differences may result in distinct grain sizes during comminution.

By analyzing the bulk chemistry of different grain size fractions generated after compression testing and creating element-by-size diagrams, it becomes possible to assess an ore's liberation characteristics. This information can prove invaluable in optimizing the processing of critical metals.

RQ 3) How can element concentration be measured from a process control perspective?

Various methods are available to measure element concentration, but not all of them offer a fast and cost-effective approach. Techniques like ICP-MS involve complex procedures that are time-consuming, expensive, and challenging. Moreover, ICP-MS tests are typically conducted in specialized laboratories, leading to long waiting times and high testing costs. From a process control perspective, a more rapid, straightforward, and cost-effective method is desirable for plant operations.

Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) in pressed powder pellets provides a quantitative approach to measuring element concentration that is less expensive and easier to execute. This method can be applied to refractory ores with lower concentrations, which can be particularly challenging to analyze using conventional techniques. This novel approach has demonstrated its safety, cost-effectiveness, and speed while delivering data quality comparable to traditional methods.

PP-LA-ICP-MS serves as a suitable technique for integration into mining processes due to its simplicity. The LA-ICP-MS sample processing and measurement protocol consist of five main steps. First, it involves sampling and reducing particles to a size smaller than <75 µm through dry milling, following standard specifications for chemical tests. Next, wet milling is employed to further reduce particle sizes to the nano range, utilizing a high-power planetary ball mill. The third step focuses on drying the sample suspension, preferably achieved through evaporation by heating the suspension in an oven at 60 °C for 30 minutes or until no liquid remains. The fourth step involves pellet pressing, followed by a chemical characterization of the pellets using LA-ICP-MS, with subsequent evaluation of the results.

RQ 4) How can a theoretical framework be designed to utilize technical and economical aspects when addressing selective comminution to extract critical minerals?

Understanding the distribution of critical metals enables early-stage preconcentration, achieved by segregating materials with a high metal content and discarding lower-value components. This reduction in material volume leads to decreased energy consumption during fine comminution, resulting in lowered process costs.

A comprehensive theoretical framework has been developed to assess the economic benefits of early material rejection in crushing plants. This framework considers economic and technical aspects, incorporating mass flow and energy requirements at different process stages. Key factors such as cost and capacity are vital considerations when analyzing, optimizing, and evaluating structural designs concerning operating expenses and mass flow.

An industrial case study has illustrated opportunities for process enhancement. By utilizing the proposed methodology for material analysis and characterization, it was observed that critical metal concentration was notably higher in finer particle sizes (0-1 mm fraction). To optimize critical metal processing, it is advisable to screen and classify fractions with higher critical metal content before the milling stage. Neglecting to separate the fine fractions may introduce risks of over-grinding and excessive energy consumption during milling. Size separation can be achieved by introducing a hydro-cyclone before the ball mill. This classifier allows the finest material (smaller than 1 mm) to be directed to separation processes like flotation, while larger particles (above 1 mm) proceed to the milling process for size reduction.

The implementation of selective comminution and reduced mass flow into the mill can yield significant energy savings. In the industrial case study, bypassing the 0-1 mm fraction resulted in a 10% reduction in production costs and a 23% increase in process capacity.

Mining companies can employ the proposed methodology at various stages of process flow planning for economic analysis. Moreover, the protocol can evaluate process performance, facilitating the assessment of selective comminution techniques.

5.3 CONTRIBUTIONS AND CLAIMS

Particle size distribution is insufficient to analyse and model what happens during coarse comminution processes regarding minerals thought which to breakage and element concentration. There is a lack of knowledge regarding minerals, elements and their distribution in different size fractions during comminution. All ores are different and behave differently, and it is necessary to make an in-depth study to gain knowledge of the ore. The mineralogy and texture affect the breakage of particles, and critical metals are not evenly distributed in different size fractions during comminution processes.

In this thesis, a grade by size methodology was developed applicable to coarse comminution. The method covers a series of properties that must be observed and measured to evaluate element and mineral distribution. The properties covered include a mechanical characterization by interparticle breakage (IPB) of the ore, followed by a mineralogical and chemical characterization of the sample after compressive breakage. This method was followed by a mathematical model capable of predicting element distribution during coarse comminution.

Parallel to this method, a protocol was developed to perform an elemental characterization by producing nano-particulate pressed powder pellets (PPP) followed by an analysis using Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS).

With these two protocols, it is possible to evaluate the mineral and element distribution of critical metals for a subsequent evaluation of early rejection of waste material by assessing material behaviour after compressive breakage.

Selective comminution is followed by a theoretical framework that predicts yield, product quality and process cost. Finally, the theoretical analysis makes evaluating the structural design regarding operating expenses and mass flow possible.

5.4 UTILIZATION

Critical metals usually are found in low grade ores which can present a content of 0.03%, which means that it is an expensive process. The low quantity of the value metals requires a creative and selective process focused on the properties of the ore. Therefore, a test methodology has been developed by which evaluate ore properties and the optimal process to reduce cost and energy while simultaneously increasing the profit of the process.

The methodology and models developed are tools capable of evaluating and measuring ore characteristics and processing. By using the tool developed is possible to propose, evaluate and optimize plant setups to maximize production, decrease waste and increase profit by using selective comminution.

We are currently going through a situation by which energy reduction during any process is highly appreciated. With this tool, it is possible to address and evaluate the process and possibilities for improvement. In other words, the tool developed will help decrease the energy needed during the process directly related to reducing waste and cost.

The summary presented in this chapter concentrates on results, contribution and utilization, as shown in Figure 27.

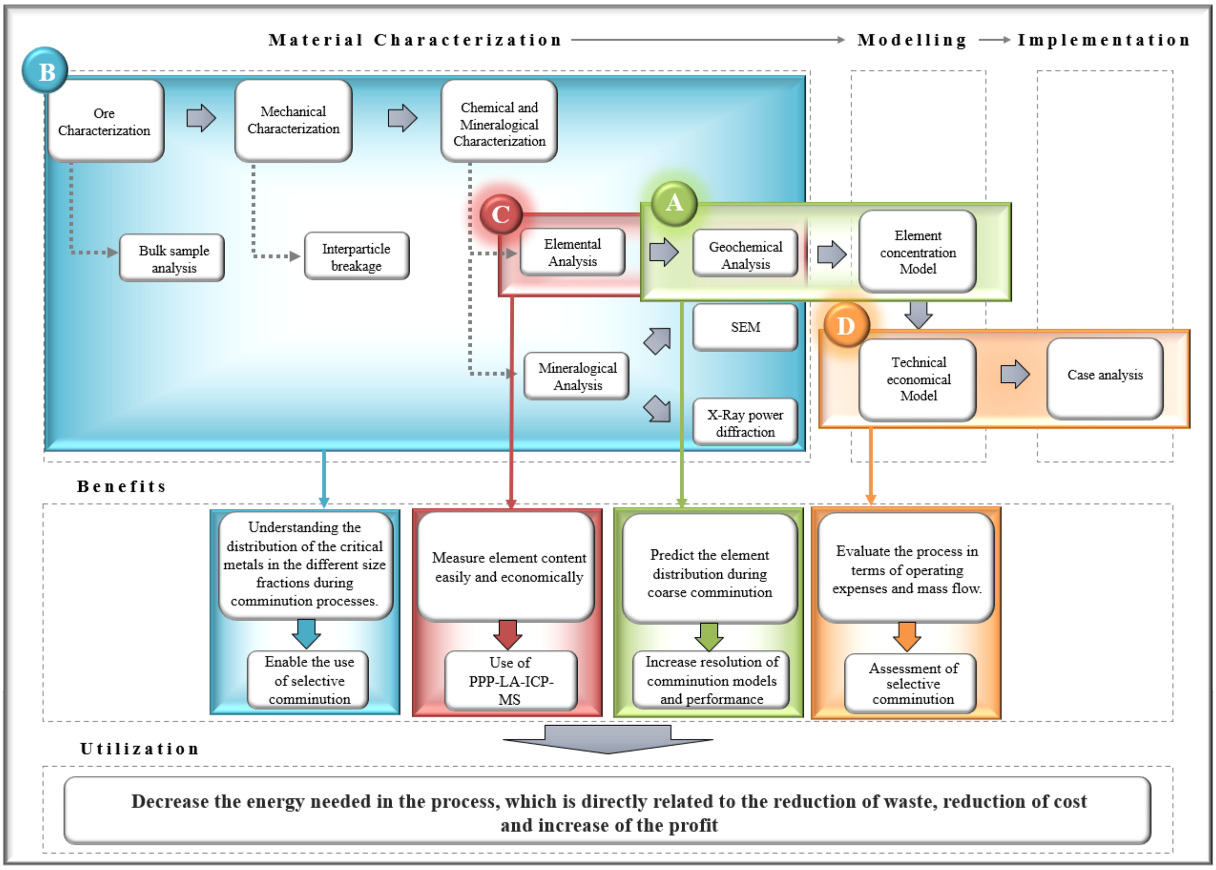

Figure 27: Material characterization and detailed test procedure presented in this thesis related to the appended papers and their utilization and benefits.

5.5 VISION FOR FURTHER DEVELOPMENT

The results presented in this thesis have been dedicated to developing a test protocol capable of measuring additional characteristics. However, considerable work remains to be accomplished.

- Plant validation is needed for future work. Working with actual data and real cases will increase research findings. By analysing real cases and actual data, it will be possible to validate the usage, capacities, and information acquired to complete the protocol.

- The concentration model was validated for low grade ores of tantalum and tungsten. The next step could include validation of other types of ores and possibly applying this model to high grade ores.

- All experiments and characterizations were performed with previously acquired samples. Nonetheless, it is crucial to include sampling techniques and their inference regarding the type of sampling, the origin of the sample, and other previous factors that were not evaluated.

- The production of nano-particulate pressed powder pellets, followed by an analysis by LA-ICP-MS, has provided efficient and accurate data, avoiding the common problems and limitations of standard geochemical methods. However, the testing of the LA-ICP-MS might be improved by using other equipment.

- The use of bed breakage parameters needs to be evaluated during fine comminution. Some researchers have been evaluating milling stages using bead breakage; nevertheless, more research is required in this area.

- Appropriate technology to achieve selective comminution needs to be addressed and evaluated. For example, using sensors during comminution could potentially benefit the sorting and separation process.

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