Characterization of Energy Efficiency in Microwave-Assisted Comminution

by

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ABSTRACT

Microwave technology has emerged as a promising tool in the mining and minerals processing industry, offering selective heating capabilities that induce fractures along grain boundaries. This innovative pretreatment method not only targets minerals selectively but also contributes to significant energy savings during subsequent grinding operations.

This thesis focuses on interpreting the multifaceted benefits of microwave treatment, with a particular emphasis on energy efficiency and its impact on downstream processes. Through a case study on copper porphyry ore, crucial parameters such as particle size analysis were linked to notable energy savings in the comminution process, alongside an assessment of mineral liberation and flotation kinetics. Optimization tests were completed to determine the limitations of the pilot-scale microwave tests.

Key observations include the correlation between microwave treatment efficiency and particle size, with relatively larger particles exhibiting better liberation benefits and particle size distribution. XRF analysis showed the elemental content of the sample. QEMSCAN analyses played an integral role in determining the extent of liberation achieved, particularly for particles passing the 2mm sieve size (-2 and +0.045) mm.

A flotation recovery of 10.54 % was realized for the microwave treated sample as opposed to 4.06 % for the reference samples. This study not only provides valuable insights into the application of microwaves in mining and mineral processing but also highlights the potential for further innovation and optimization in this field. The continuous evolution of

microwave technology underscores its growing significance as a sustainable and efficient solution in mineral processing operations.

Keywords: comminution, microwaves, energy, liberation, size reduction, breakage; Copper Porphyry ore

PREFACE

The technical apparatus referred to in chapter 3 of this thesis was designed by me, with the assistance of John Forster (PhD) and Professor Erin Bobicki. The data analysis in chapter 4 and concluding analysis in chapter 5 are my original work, as well as the literature review in chapter 2. Chapter 3 and Chapter 4 of this thesis is part of a collaborative research conference paper which has been accepted by the International Mineral Processing Congress (IMPC) as R. Achina-Obeng, Prof. E. Bobicki, Prof. F. Sadri and J. Forster (PhD) "Assessing the Benefits of Microwave Treatment on Microwave Heating Behaviour, The Particle Size Distribution and Liberation of Copper Ore," for a presentation which takes place in Fall 2024, from September 29 to October 3, 2024 at the National Harbor, Washington, DC. I was responsible for the data collection and analysis as well as the manuscript composition. John Forster (PhD) assisted with the data collection and contributed to manuscript edits. Prof. E. Bobicki and Prof. F. Sadri were the supervisory authors and were involved with concept formation and manuscript review.

DEDICATION

"The More I Learn, The More I Realize How Much I Don't Know."

- Albert Einstein

To my family and friends, whose unwavering support and encouragement have guided me throughout this journey, and to all those who dare to dream, question, and explore, this thesis is dedicated to you. Your faith in me allowed me to achieve this. This thesis is dedicated to Samuel Obeng-Damoah, Helena Ackah-Badu, and Michaelina Alluah-Obeng, my late immediate family.

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I would like to thank the team at Sepro Minerals in Langley BC for allowing me and giving me the opportunity to use their facility for my experiments. I would also like to thank John Forster, whose constant support from the laboratory through meaningful and insightful discussions helped me a great deal.

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Finally, I would like to thank Highland Valley Copper of Teck Resources, Canada, for supplying the ore samples for this research.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABBREVIATIONS

HVC- Highland Valley Copper

MIBC- Methyl Isobutyl Carbinol

MWT- Microwave Treatment

PAX - Potassium Amyl Xanthate

SIPX- Sodium Isopropyl Xanthate

QEMSCAN- Quantitative Evaluation of Minerals by Scanning Electron Microscopy

XRF- X-Ray Fluorescence

SI UNITS

mm- millimetre

% - percentage

μm- microns, also known as micrometres

g- grams

kW- kilowatt

s- seconds

Mins- minutes

Hz- hertz

Mhz- megahertz

v/m - volt per meter

W/m^3- watts per cubic meter

W- watts

Kg/m^3 – kilogram per meter cube

Chapter One

1 Introduction

1.1 Background

Mining is a key industry that supplies raw materials to different sectors worldwide. Mineral processing, which encompasses comminution, sizing, concentration, and dewatering, is essential for extracting valuable minerals from ore. Comminution, the initial stage of mineral processing, involves reducing the size of solid materials such as ore bodies through crushing and grinding. However, traditional comminution methods often consume a significant amount of energy and may not achieve optimal mineral liberation. In response to these challenges, there is an soaring appeal in exploring alternative comminution methods. Microwave-assisted comminution is one such method that leverages selective heating to potentially enhance energy efficiency and mineral liberation, while reducing environmental impacts. Microwave-assisted techniques have been explored in various fields, but their application to comminution in the mining industry and their downstream benefits, especially for copper porphyry ore, is relatively scanty. Microwave treatment demonstrates enhanced efficiency on coarser particle sizes (Hassani et al. 2020; Kingman et al. 2004) With the existing literature, there is a lack of guidance or suggestion on the ideal or optimal particle size of copper porphyry ore that has achieved good downstream benefits. This investigation seeks to bridge this gap by assessing the effectiveness of microwave-assisted comminution of a copper porphyry ore, and to identify the accompanying benefits and optimal parameters for microwave-assisted comminution in mineral processing.

In particular, the aim is to assess whether microwave treatment improves comminution efficiency compared to conventional methods, leading to enhanced mineral liberation and recovery rates.

1.2 Objectives of Research

In the field of mineral processing, there is an ongoing search for methods that can efficiently reduce the size of ore material while conserving energy and promoting environmental sustainability. In view of the growing demand for metals such as copper and the increasing use of low-grade ores, it is necessary to explore innovative methods. Conventional methods for reducing the size of ore material are often associated with significant energy consumption and environmental impacts. As a result, there is a need for alternatives that not only increase energy efficiency, but also benefit downstream economies. This study investigates microwave-assisted comminution, specifically its application in copper porphyry ore. The three key objectives of this study are summarized as follows:

- Impact of Microwave Treatment on Mineral Liberation

The aim of the present study was to evaluate the effect of microwave treatment on mineral liberation in copper porphyry ore. To determine the effectiveness of microwaves in facilitating ore breakage and liberation, there was a comparison of the degree of mineral liberation achieved using microwave-assisted comminution with conventional methods.

- Influence of Microwave Treatment on Particle Size Distributions

In this study, the effects of microwave treatment on the particle size distribution of copper porphyry ore were examined. The objective of the present study is to elucidate the effect of microwaves on the size distribution of comminuted ore particles through a detailed analysis and comparison of particle size distributions obtained from microwave-assisted comminution and traditional grinding methods.

- Optimal Operating Parameters for Microwave-Assisted Comminution

This study aims to identify the optimal operating parameters, including power level and treatment duration, for microwave-assisted comminution of copper porphyry ore. Through systematic experimentation and analysis of various operating conditions, the objective is to establish the most effective parameters for achieving desired outcome in terms of mineral liberation and particle size distribution.

Through the pursuit of these objectives, this research endeavours to contribute valuable insights to the field of mineral processing, shedding light on the efficacy and potential applications of microwave technology in ore comminution.

This study investigates and analyses the use of microwave treatment to enhance energy efficiency and mineral liberation during comminution process. Microwave-assisted comminution represents a special application focusing on a copper porphyry ore. Copper porphyry deposits are known for their complex mineralogy and variability, which present unique challenges in processing. By exploring microwave-assisted comminution, this research is aligned with broader industry trends in the sustainability and efficiency of mining operations.

The innovative and forward-thinking nature of this study emphasizes the potential benefits of reducing energy consumption, minimizing waste, and improving mineral recovery.

1.3 Thesis Outline

This thesis explores the transformative role of microwaves in mining and mineral processing, emphasizing their selective mineral heating and energy efficiency benefits. Microwaves are shown to induce fractures along grain boundaries, conserving energy during subsequent grinding operations.

Chapter 2 reviews various comminution techniques, breakage mechanisms, and energy-saving pretreatment methods, emphasizing microwaves. Additionally, it covers flotation and liberation measurement techniques.

In Chapter 3, the research methodology is outlined, focusing on a case study using copper porphyry ore. The study assesses comminution energy savings from microwave pretreatment, mineral liberation, and flotation kinetics, employing advanced techniques such as XRF and QEMSCAN analysis.

Chapter 4 solidifies the benefits of microwaves for comminution energy savings and downstream processes, emphasizing the correlation between microwave efficiency and particle size. The discussion highlights the downstream process advantages, ensuring a promising trajectory for microwave applications in mining and mineral processing.

Chapter 5 presents conclusions, recommendations, and limitations, proposing improvements for maximizing microwave use in mineral processing. These recommendations stem from the findings in previous chapters and suggest avenues for future research and development.

Chapter Two

2 Literature Review

Mineral processing is a postmining operation which is aimed at achieving liberation and separation of mineral of interest. The mineral of interest is separated from the gangue material from the ore body. The variations in the physical and chemical properties of the mineral of interest and the gangue material is what helps in the separation process by employing appropriate methods. Mineral processing undergoes four main steps, and these are comminution, sizing, concentration and dewatering (Haldar 2018). However, the current research primarily concentrates on comminution and beneficiation, with a specific emphasis on flotation.

Mineral beneficiation is a process wherein valuable components of an ore are concentrated through physical separation methods. It involves several stages, including crushing, grinding, and concentration, with the aim of increasing the grade (concentration) of valuable constituents in the original ore. Typical beneficiation processes encompass sample preparation, comminution, size classification, and concentration (Haldar 2017).

Comminution is important as it liberates the mineral of interest in an ore body for recovery, it makes it easier to transport ore by means of conveyor belts for further mineral processing.

The microwave treatment provides a strategic approach to not only contribute to waste reduction and minimize energy consumption but also result in cost efficiencies, heightened mineral recovery, and an overall reduction in the carbon footprint associated with mining operations (Chen et al. 2024; Hassani et al. 2020) . The central focus of this thesis is the exploration and enhancement of microwave-assisted comminution techniques tailored specifically for copper porphyry ore.

Research studies conducted by Adewuyi et al. (2020) on community energy consumption in Canada between 1990 to 2015 revealed a high energy consumption from oil, coal and natural gas sources amounting to an average of 70.71 PJ of power with the indicated years. The statistics from the research studies are depicted in the Figure 2-1 illustrates the trend in energy consumption attributed to comminution in Canadian mining companies from 1990 to 2015.

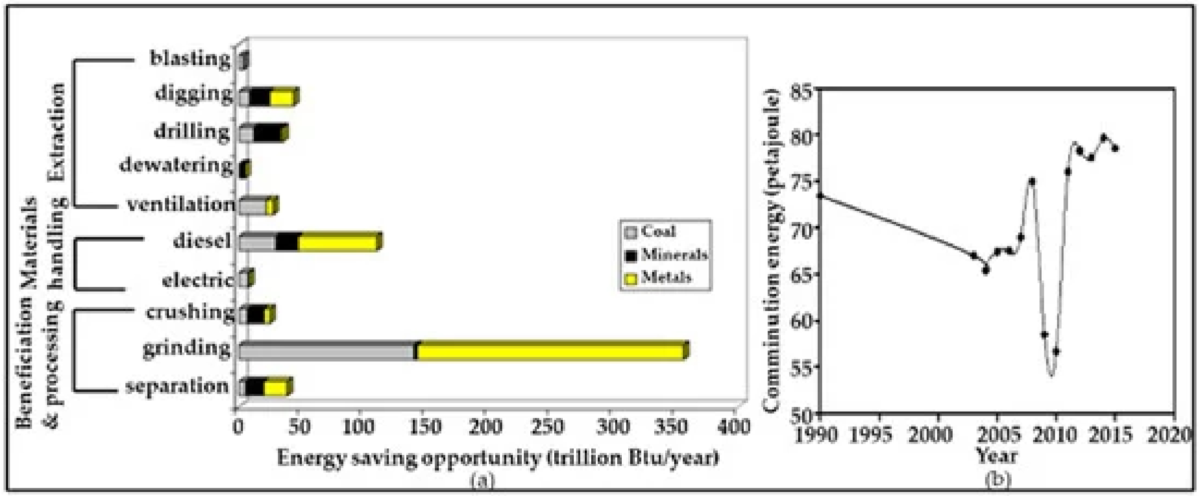


Figure 2-1 Energy consumption in Canadian mining companies due to comminution from 1990-2015 (Y. Wang, E. Forssberg, M. Svensson 2017)

To mitigate the substantial power consumption associated with comminution processes, researchers have historically advocated for optimization strategies such as adjusting the mill diameter to ratio, as posited by Kanda and Kotake (2007), and manipulating the mill load ratio and filling, as proposed by Yin et al. (2017) . These conventional methodologies seek to refine the efficiency of grinding operations. In recent decades, a novel paradigm has emerged, championed by Somani et al. (2017), advocating for a distinctive approach: the pre-treatment of ores prior to grinding. This avant-garde methodology involves a meticulously designed series of interventions aimed at inducing trans and intergranular cracks in the ore body. The fundamental objective is to expedite the liberation of minerals during the subsequent grinding phase, thereby curtailing the overall effort and energy demands entailed in the process. This alternative strategy reflects a progressive shift in addressing the challenges posed by high energy consumption in comminution, offering a nuanced perspective on optimization in the realm of ore processing.

Energy efficiency with conventional grinding circuits is considerably low (Kolev et al. 2021). To achieve mineral liberation, size reduction and or comminution must take place. Comminution in mineral processing consumes the most energy of all the processes in a mineral processing plant. For mine sustainability and cost effectiveness, many approaches have been employed to enhance energy efficiency including various pretreatment methods for ores such as thermal (oven, microwave, or radiofrequency), chemical additive, electric, magnetic, and bio-milling before comminution. However, pre-treatment by microwave radiation has shown very promising prospects such as better liberation and grind with past research (Bobicki et al. 2020; Hassani et al. 2020; Ding et al. 2017). Microwave pre-treatment studies from past research have been discussed with some deductions made on how to maximize its effects towards the efficiency of comminution energy. The employment of microwave assisted comminution of ore is geared towards the efficient use of comminution energy and to ensure enhanced mineral liberation and hence recovery processes.

2.1 Pre-treatment of Ore

Pre-treatment of ores is usually performed on ores prior to comminution. The primary purpose of pre-treating ores is to generate intergranular cracks to make grinding easier consequently resulting in less expenditure of grinding energy. Various pre-treatment procedures of ores before comminution have been investigated to decrease the energy required for comminution.

Pre-treatment of ore by chemical additives is said to be a very convenient method of pre-treatment before grinding. The chemical additive method for instance has been employed at the industrial level mainly in cement production. Usually, there are two types of chemicals used to produce cement. The first type is used for surface structure modification of the particle to enhance grinding while the second is a strength enhancer. These chemical additives serve as grinding aids normally reacting with ore molecules on the ore surface. These reactions cause local stresses which results in fragmentation at the grain boundaries. This allows for enhanced size distribution and reduction in the clustering of particles (Chipakwe et al. 2020).

The microwave pre-treatment results exhibited some encouraging progresses at pilot scale mining applications in 2017. Conventionally, size reduction takes place because of the exertion of mechanical stress. The greater the number of fractures created in a particle, the easier it is to break it. Usually, minerals have distinct microwave absorption characteristics as such different minerals within the same sample have contrasting levels of heating when subjected to microwaves. The variation in heating of these different minerals in a particular sample causes thermal stresses within particles which is likely to create fracture which normally occurs along grain boundaries with enhanced grinding and liberation being the resultant effects (Bobicki et al. 2018).

Ore pre-treatment by electrical means relies on the principle of passing high voltage electrical pulses through a rock matrix to create fragmentations. There is expansion and explosion of rock grains in the rock matrix when high voltage electrical pulses are passed through it. The lack of uniformity in the expansion of mineral grains result in the generation of micro cracks (Somani et al. 2017).

Reports showed that magnetic, radiofrequency, and bio-milling methods have not led to much promising effects in minimizing comminution energy. With respect to minimizing comminution energy, safety, cost effectiveness and advantages to downstream processes, microwave and electrical pre-treatment methods may be focused for applications in the mining industry (Adewuyi et al. 2020). Figure 2-2 shows the different pre-treatment methods and their categorization.

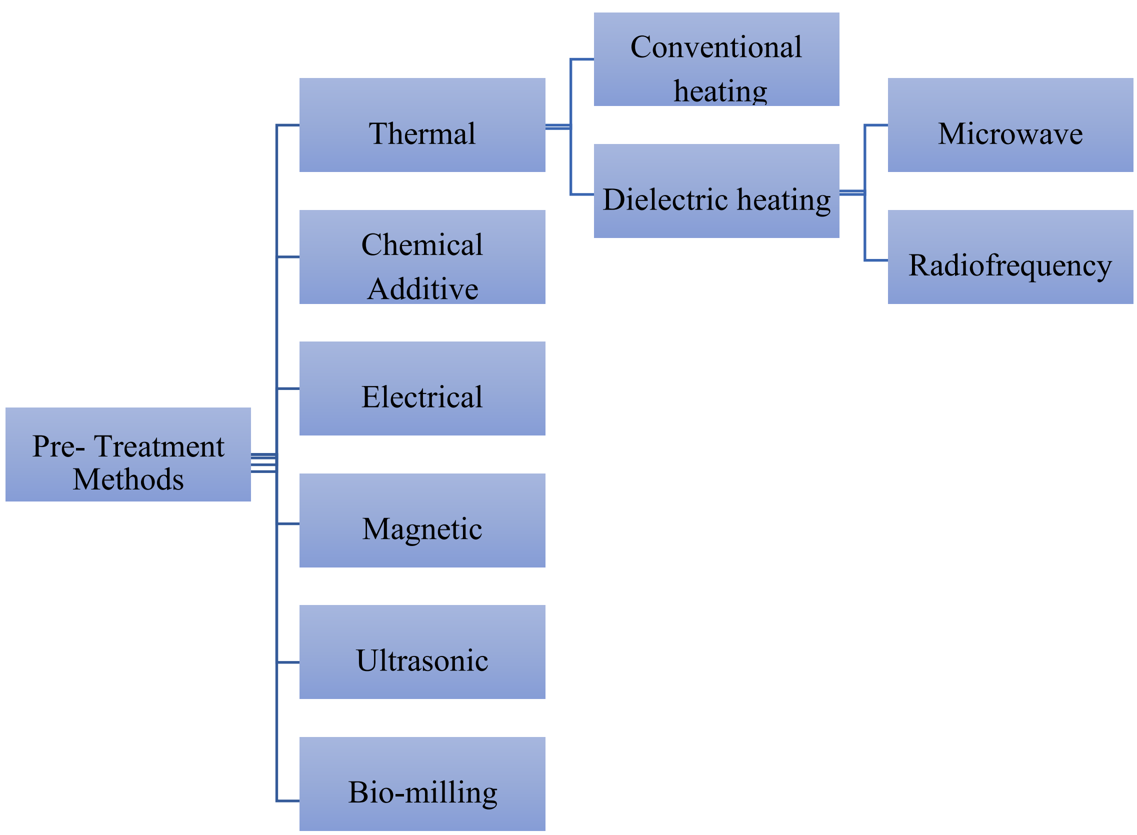


Figure 2-2 Classification of pre-treatment methods (Adewuyi et al. 2020)

2.1.1 Thermal Treatment

Thermal ore treatment encompasses the utilization of heat by virtue of convection and conduction to induce granular cracks in ore bodies to aid with liberation at latter stages of mineral processing. During heating, the rise in temperature of the ore body over a period interferes with the physical properties of the ore which causes misalignment, fractures, and loss of the structural integrity of its particles. According to Adewuyi et al. (2020) there are two approaches of applying thermal treatment to ores. These methods include conventional heating using furnaces and electromagnetic heating using either radiofrequencies or microwaves.

2.1.2 Treatment Using Furnace

The conventional methodology for ore pre-treatment entails the utilization of a furnace employing thermal heating, wherein the entire ore undergoes heating at diverse temperatures to induce granular cracks. Regrettably, this method is marked by heightened energy consumption (Adewuyi et al. 2020). Its inefficiency is highlighted by the indiscriminate heating of the entire rock ore, lacking the precision required for targeting specific elements within the rocks. This lack of specificity translates into escalated energy usage, thereby elevating operational costs (Hamuyuni et al. 2021). An alternative and more efficacious approach would involve refining the pre-treatment process to selectively apply heat to distinct elements within the ore, which is employed using electromagnetic heating, consequently resulting in a substantial reduction in energy consumption and fostering economic savings.

2.1.3 Treatment Using Electromagnetic Heating

The utilization of electromagnetic heating, specifically in the forms of microwave and radio frequency heating, falls under the category of dielectric heating and has been widely employed across diverse industries for its precision and targeted heating capabilities. This heating method involves various mechanisms such as dipolar polarization, interfacial polarization, and conduction due to heat (Ramaswamy and Tang 2008). Within this framework, minerals within the ore body possessing dielectric properties respond to electromagnetic heating.

In simpler terms, when minerals with adequate electrical properties within the ore body encounter an electromagnetic field, rapid oscillation of electrons occurs, resulting in material heating due to the generated electrical resistance. This not only facilitates a faster and more efficient heating process but also mitigates the risk of excessive energy consumption and reduces overall operational costs for mining endeavours. Notably, research studies feature the considerable improvement in mineral recovery achieved through microwave-assisted pre-treatment (Sun et al. 2016; Schmuhl et al. 2011)

2.1.4 Electric Treatment

Electric treatment involves the application of high-voltage electrical pulses to fragment rocks into sizes conducive to grinding. The initiation of these pulses prompts the evaporation of moisture within the ore body, leading to controlled explosions that further break down the rocks into smaller dimensions. While this method exhibits praiseworthy characteristics, it is not without its challenges. These include the diminished conductivity of ores and minerals, safety hazards associated with managing high-voltage power, and notable technological constraints concerning the design, development, and commercialization of this approach, as articulated by (Singh et al. 2019).

2.1.5 Chemical Additives Treatment

Chemical additive is one of the most popular ore pre-treatment methods as it can be applied on a large industrial scale, these are chemicals are added before comminution to (El-Shall and Somasundaran 1984; Mishra et al. 2017). Due to their nature and stage of addition during the comminution process, they are referred to as grinding additives. These chemicals aids in the flow property modifications, particle de-agglomeration, frothing minimization, and surface softening Singh et al. (2019) . The effects of additives on grinding have been studies by various researchers in the mining industry. The areas analysed includes the effect on enhanced grinding, impact on selective grinding, impact on efficiency as well as cost and long-term effect on flotation and filtration.

Although chemical additives have the potential to reduce energy consumption by half, a commercial scale application is still not practical because of cost involved in acquiring and applying these additives in the comminution process, product contamination due to the chemical additives added and lastly safety and environmental issues pertaining to the pollution of water bodies and other natural resources around mine sites.

2.1.6 Microwave History & Theory

The exploration of microwaves in ore treatment has a rich research history dating back to the 1960s, exemplified by the pioneering work of Puschner (1966). Microwave heating operates within two predominant frequency bands: 915 MHz, tailored for industrial applications, and 2450 MHz, strategically employed in expansive commercial endeavours.

2.1.7 Microwave Heating Mechanism

It is crucial to emphasize that microwave energy differs from thermal energy. Microwave heating occurs when charged particles within a material attempt to reorient themselves in response to the electromagnetic field. The degree to which a substance absorbs microwave energy is contingent on the arrangement and characteristics of its atoms, as well as the energy levels of its electrons (Sun et al. 2016).

Given that many materials lack magnetic properties, there exist two fundamental mechanisms for microwave heating: dipole rotation and ionic conduction. Dipole rotation occurs when molecules within the material are not synchronized with the microwave field and subsequently rotate to reposition themselves. This rotational motion results in friction, leading to the generation of heat. On the other hand, ionic conduction serves as the prevailing mechanism for heating minerals. (Tranquilla 2000). The motion of charged atoms within the microwave field produces heat and facilitates the transfer of energy. A rise in sample temperature directly constitutes to an increase in the transfer of energy.

The primary factors influencing the extent of heat generation within the exposed material are the complex permittivity (ε) and complex permeability (μ). Complex permittivity is associated with the oscillating electric field component of microwaves, signifying the material's capacity to absorb and retain electric energy potential. Similarly, complex permeability is tied to the oscillating magnetic field component of microwaves, indicating how the material responds to the applied alternating magnetic field component. Key parameters that govern microwave heating encompass the complex permittivity (as indicated in Equation 1), power density (defined by Equation 2), heating rate (expressed in Equation 3), and penetration depth—referring to the distance at which the power density decreases to 1/e of its surface value (outlined in Equation 4) (Chaouki et al. 2020).

$$\varepsilon = \varepsilon_0 \cdot (\varepsilon_r' - j\varepsilon_r'') \qquad \text{(Eq.1)}$$

$$P_{av} = 2\pi f \varepsilon_0 \varepsilon_r'' E^2 + 2\pi f \mu_0 \mu_r'' H^2 \qquad \text{(Eq.2)}$$

$$\frac{dT}{dt} = \frac{2\pi f \varepsilon_0 \varepsilon_r'' E^2}{(C_p \rho)} \qquad \text{(Eq.3)}$$

$$D_p = \frac{\lambda_0}{2\pi\sqrt{2\varepsilon_r'}} \left\{ \sqrt{\left[1 + \left(\frac{\varepsilon_r''}{\varepsilon_r'}\right)^2\right]} - 1 \right\}^{-\frac{1}{2}} \qquad \text{(Eq.4)}$$

In Eq. 1, ε_0 is the permittivity of free space. The average power density (Eq. 2) is the sum of the electric and magnetic losses, where E is the root mean square electric field intensity (V/m); f is the frequency (Hz); μ_0 is the permeability of free space ($2\pi \times 10^{-7}$ H/m); μ_r'' the relative magnetic loss factor; and H is the root mean square of the magnetic field. The heating rate (Eq. 3) is directly proportional to the power density, (P_{av}) and inversely proportional to the specific heat capacity, (C_p) and density of the workload (ρ). Eq. 4 is used to compute the distance at which the microwave power is reduced to 1/e of its surface value, where λ_0 is the wavelength of the incident radiation (Chaouki et al. 2020).

2.1.8 Microwave Pre-treatment

The use of microwaves as an ore pre-treatment method has been investigated in quite a few applications and research carried out over the years (Adewuyi et al. 2020; Somani et al. 2017; Sun et al. 2016). Nevertheless, there has been a substantial and rising involvement in the size reduction of ore in mineral processing. There is evidence to suggest that liberation is the basic benefit of microwave pre-treatment of ore. With the depletion of high-grade ores, it has become eminent to process low grade ores. This however increases cost in mineral processing or recovery. Since these low-grade ores would have to be grinded to very fine sizes before their minerals can be liberated, this may result in the production of slimes and consequently loss of valuable minerals to tailings due to process limitations. Microwaves are a form of electromagnetic radiation with wavelengths from 1 m (300 MHz) to 1 mm (300 GHz). These waves create microfractures in the ore, reducing the energy needed for grinding (Haque 1999).

According to reports by Amankwah et al., fragmentation of the ore by microwave pre-treatment is usually successful when there are at least two components, with distinct dissimilar microwave absorption properties. The variances in strength are linked to the differential thermal expansion of the constituents of the ore, which had many variants of hyperactive, active, and inactive microwave absorbers (Amankwah et al. 2005). Different heating rates are detected in minerals when exposed to a microwave field because of thermal selectivity of materials. Stress induction as well as the formation of cracks between different mineral particles by microwaves improves liberation and increases comminution efficiency (Singh et al. 2019). There has been the introduction of microwave treatment processes in mineral processing especially with microwave-assisted comminution to enhance energy efficiency of comminution which according to Radziszewski has an efficiency of less than 1% (Radziszewski 2013).

According to (Cisternas et al. 2022; Adewuyi et al. 2020) comminution in the mineral processing process consumes 50%-70% of the total energy used and contributes largely to the total carbon emission. Consequently, the usage of other comminution process using ball mills have indicated highly inefficient outcomes because of noise, mechanical and heat losses with efficiencies reaching the highest of only 1%-2%. As a result of these high energy consumptions, it is crucial to devise more efficient and effective comminution approaches to save cost, energy and reduce global carbon emissions. Microwave heating has emerged as a highly advantageous alternative to traditional ore heating methods in mineral processing, as underscored by Haque's insights in 1999. The delineated advantages encapsulate the merits that differentiate microwave heating from conventional approaches. These advantages encompass expeditious energy transfer, discriminative material heating based on dielectric properties, non-contact application of heat, volumetric heating ensuring uniform temperature distribution, and prompt commencement and cessation of the heating process. The application of microwave-assisted heating to rock ores specifically enables targeted and efficient heating, thereby mitigating energy consumption and reducing the overall carbon footprint associated with mineral processing operations. The nuanced benefits offered by microwave heating underscore its potential to revolutionize and optimize ore processing methodologies on an industrial scale.

2.1.9 Methods to Characterize the Effects of Microwave Treatment on Comminution

A prior knowledge of the mineralogy of ore materials minimizes variability and enhances the reliability and repeatability of microwave treatment. This can be projected to explain the uneven microwave treatment of ore both at the bench and pilot scales. If there is a thorough understanding on the effect of ore mineralogy with regards to microwave treatment and energy consumption, it will be paramount for a more consistent and efficient assessment. Permittivity tests can be conducted to have a better understanding of the manner the ore will respond to microwave treatment. The effect of microwave heating on sample factors such as volume, surface area, size and shape will be studied to know which benefits most from microwave pretreatment. A basic understanding of how microwave energy interacts with minerals is important for projections and the use of the technology on a large scale for mineral processing.

Presently, the world is geared towards reduction in energy consumption and enhancing sustainability. Most industries have embraced this, and the mining and mineral processing industry is not left out. Several approaches have been explored by the mining and mineral processing industry to enhance energy efficiency, such as decarbonization methods and the use of electric cars, different pretreatment methods prior to comminution, with an example being the use of microwaves. There however remains a critical knowledge gap concerning the optimal utilization of microwave pretreatment. Existing research has shown promising results (Bobicki et al. 2014;

Ding et al. 2017; Hassani et al. 2020; Kobusheshe 2010) suggesting that microwave pretreatment has the potential to significantly reduce energy consumption during comminution and enhance mineral liberation. However, a thorough understanding of the factors influencing the effectiveness of microwave pretreatment, quantitative characterization of its benefits and its scalability for industrial applications, remains limited in the literature. This research and thesis seek to bridge this knowledge gap by systematically investigating the impact of microwave pretreatment on energy efficiency in comminution and the liberation of valuable minerals. By optimizing microwave treatment parameters, conducting comparative analyses with conventional methods, this study aims to provide a holistic understanding of the benefits and challenges associated with microwave-assisted comminution. Such insights are crucial for the mining industry's efforts to reduce energy consumption and improve overall process efficiency, especially when processing low-grade ores.

2.2 Microwave Treatment of Copper Porphyry Ore

The recent strides in technology and automation for ore sorting are essential to tackle the difficulties posed by hard rock mineral processing. Barren materials introduced during mineral processing, whether intentionally or unintentionally, can lead to substantial energy consumption and material losses in the form of tailings. Utilizing conventional methods for processing such ores proves costly and inefficient, underscoring the importance of incorporating ore sorting techniques to eliminate undesired materials prior to beneficiation.

Research by Ballantyne and Powell (2014) underscores that ore grade significantly influences comminution energy requirements, grind size, and ore competency. Higher ore grades contribute to more efficient processing. Batchelor et al. (2016b) emphasize the advantages of early elimination of barren materials from low-grade ores, resulting in heightened metal production, decreased processing and waste handling, and a diminished plant carbon footprint.

The ore sorting process typically involves stages like ore preparation, presentation, excitation, discrimination, and separation. The selection of a particular approach is contingent on factors such as energy consumption and the cost associated with the separation process (Batchelor et al. 2016b). Researchers have explored various excitation-discrimination methods, including optical, thermal, nuclear, electrical, and magnetic approaches (Batchelor et al. 2016a; Lessard et al. 2016; Murphy et al. 2012).

However, the existing approaches have proven ineffective for discriminating low-grade copper porphyry, which constitutes a significant portion of global copper production and serves as a primary source of gold, molybdenite, and silver (Batchelor et al. 2016a).. This inefficiency presents a notable challenge, emphasizing the need for further research and innovation in ore sorting technologies, specifically tailored to address the complexities of low-grade copper porphyry. Developing sorting methods that cater to its unique characteristics could significantly enhance the efficiency of mineral processing in this context.

In summary, while ore sorting technologies have demonstrated success in various mineral processing scenarios, the challenge of discriminating low-grade copper porphyry remains a pivotal area for ongoing research and development in the realm of ore sorting and mineral extraction.

The utilization of microwave technology for treating copper porphyry has recently gained considerable traction and has become a focal point of interest among researchers exploring various ore grades. This innovative method has demonstrated efficacy in diminishing ore competency and promoting liberation by exploiting the targeted volume heating of specific mineral components within the ore. A distinctive advantage of this approach is its ability to swiftly heat materials with high microwave absorption rates, such as copper, iron, and nickel, as opposed to the comparatively slower heating of rock-forming minerals like micas and quartz (Kobusheshe 2010). Employing high-density microwave power, the method induces controlled thermal cracking along the grains of the mineral composition. This thermal treatment leads to the development of both inter-granular and trans-granular fractures, which results in the efficiency of the process.

2.3 Permittivity and Dielectric Properties of Materials

The relative dielectric constant (real permittivity) quantifies a material's capacity to become polarized when exposed to an electric field. Conversely, the relative dielectric loss factor (imaginary permittivity), determines the efficiency with which microwave energy can be transformed into heat within the material (Pickles 2009). A material possessing a high relative imaginary permittivity will exhibit a greater propensity for absorbing energy compared to a material with a lower relative imaginary permittivity. The rate at which power is absorbed into the sample is contingent not only on the sample's volume and dimensions but also on factors such as the location of hotspots, particle size distribution (PSD), and mineral composition (Shadi et al. 2023). Ore fragments that closely match the penetration depth tend to exhibit a favourable response to microwave heating (Sun et al. 2016). Several factors influence the dielectric characteristics of a substance, and these encompass temperature, moisture level, frequency, as well as the material's density and structure (Parkhomenko 2012). Based on the dielectric attributes of the material, microwaves can be absorbed, reflected, or transmitted. When the imaginary permittivity is exceptionally elevated, the material tends to exhibit reflection. Conversely, materials characterized by exceptionally low dielectric properties remain transparent to microwaves, indicating minimal or no interaction with them (examples include quartz and alumina).

2.4 Penetration Depth of Ores

The distance by which an electromagnetic radiation such as microwaves travel into a material before being absorbed is known as the penetration depth of that material. It is a critical parameter that determines how effectively microwaves can interact with and heat an ore material. Microwave absorbing materials which have a penetration depth which is close to the particle size respond better to microwave heating (Sun et al. 2016). Minerals or metals that have higher electrical conductivities also have greater penetration depth ratio which results in a greater influence on how responsive the ore is to microwaves thereby absorbing the microwave energy more efficiently. This ensures a uniform within the ore matrix which leads to obtaining efficient microwave heating processing outcomes. Higher permeability and greater penetration depths facilitates efficient microwave treatment of an ore material (Lin et al. 2022).

2.5 Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC)

Under controlled temperature conditions, TGA/DSC are analytical techniques employed to determine the thermal properties and behaviour of materials. To measure the change in any physical or chemical changes associated with phase transformations, decomposition or loss of moisture, in relation to change in temperature with time, TGA/DSC measurements are taken (Sarbishei and Tafaghodi Khajavi 2019). These measurements are important to determine the effects of microwave treatment on ores and can help in enhancing the optimization of the microwave heating process.

2.6 Comminution Process

Comminution refers to the process whereby a solid material especially an ore body is reduced in size into small fragments, particulate sizes or pulverized. Comminution refers to both crushing and grinding, essentially describing the breakdown of ore particles. It is the first stage of mineral processing, usually starts with the crushing circuit, where ore from the run-off- mine pad (ROM pad) is introduced into a primary crusher which could either be a jaw or gyratory crusher (Wills et al. 2016). After it has been crushed and reduced to the required mesh size, it is then conveyed, mostly by conveyor belts into a secondary crusher for further size reduction before it is eventually sent to the milling circuits where comminution continues to increase the surface area of the ore in order to enhance liberation of the mineral of interest and further mineral beneficiation processes (Klein et al. 2018; Bouchard et al. 2018).

Minerals found in ore bodies are embedded together with gangue materials. For these minerals of interest to be made free from the gangue materials there must be size reduction and this process is known as comminution (Wills et al. 2016).

The first step in mineral processing is normally comminution in mineral beneficiation plants. Comminution involves crushing and grinding processes. Crushing reduces the particle size of run-of-mine ore (usually as large as 1370 mm), and these are reduced during crushing to 100-200 mm with primary crushers. Dependent on the type of ore and specified feed particle size for milling, secondary crushers may or may not be needed. Grinding is carried out until the mineral and gangue materials are significantly produced as separated particles. The primary purpose for comminution in ore processing is to liberate minerals for successful separation in downstream processing (Wills et al. 2016).

Comminution is known to be the most energy consuming process in mining. The mining energy value chain starts with comminution through to smelting then finally refining. Comminution is the main constituent of the mining energy value chain. For instance, there is an expectation of 95.5 % increment of mining energy from what was consumed in 2013 for the copper mining industry with a projection of 41.1 terawatt-hours (TWh) to be consumed in the year 2025 (Jeswiet and Szekeres 2016). At a copper mine site, the apportioning of energy is 3 – 5 % for blasting, 5 – 7 % for crushing, and 80 – 90 % for grinding (Jeswiet and Szekeres 2016).

An enhancement in the energy performance of comminution processes represent significant overall energy savings. The main comminution methods are crushing, and grinding, however, there are other technologies that can affect energy usage. These include continuous mining, ore sorting, rock weakening and size classification technologies (Klein et al. 2018).

2.6.1 Crushing

Crushing is achieved by compression of the ore against a rigid surface or by impact against a surface in an inflexibly controlled motion of crusher parts. In mineral processing, the first stage of comminution is crushing. Normally, crushing is a dry process which takes place in many steps. Feed for primary crushing is usually ore from the run-of-mine. The primary purpose of crushing in mineral processing is to produce amenable feed size for milling. There are primary, secondary, and tertiary crushers. Primary crushers encompass jaw and gyratory crushers, while secondary crusher include cone, roll and impact crushers (Haldar 2018; Wills et al. 2016). Table 2-1 outlines the range of feed particle sizes suitable for various types of crushers and the corresponding expected product sizes. Additionally, it provides information on the expected throughput and the stage of crushing for each piece of equipment.

Table 2-1 Summary of crushers

Crusher	Particle size	Expected Throughput (tph)	Crushing stage
Jaw	Feed: Up to 1.5 m	Up to 725	Primary
Gyratory	Feed: Up to 1.5 m	Up to 5000	Primary
Cone	Feed: Up to 15 cm	1100-3000	Primary grinding

2.6.2 Grinding/Milling

Milling otherwise known as grinding is achieved by abrasion and impact of the ore material which is usually because of the unrestricted movement of independent grinding media such as balls, rods, or pebbles. This is contrasted with grinding which is accomplished by abrasion and impact of the ore by the free motion of unconnected media which may include rods, balls, or pebbles. Generally, the last stage in the process of comminution is milling. During milling, ore particles are reduced in size by the mixture of impact and abrasion, it can be dry or with water (Wolosiewicz-Glab et al. 2018). Usually, the grinding process is carried out after crushing, the process may be dry or wet. Minerals being crystals tend to break into infinite numbers of sizes and shapes every time they are introduced to energy (Tromans 2008).

Grinding is usually done in cylindrical tumbling mills. The main differences between cylindrical tumbling mills lie in the ratio of diameter to the length of the cylinder and the type of grinding media used. Grinding mills can be grouped into these main types; rod mills, Autogenous & Semi-Autogenous Grinding (SAG) mills and ball mills. Rod mills use rods as grinding media, size reduction occurs by friction and attrition between steel rods and ore particles. Mill feed size can be as large as 50mm with a product size as fine as 300 µm. Rod mills are the preferred choice for sticky and coarse-grained ores which may clog crushers. Due to the controlled size reduction, they are mostly operated in open circuits. The grinding media of an AG mill is the ore itself. SAG mills make use of steel balls with forces of impact and abrasion between grinding balls and ore particles, this result in finer particles. SAG/AG mills feed size can be as large as 200 mm and product feed size of 0.1 mm. Ball mills are usually secondary grinding mills. Ball mills have feed size ranging from 5-15 mm and produces a product size of under 300 µm for separation or extraction (Anderson et al. 2015).

Particle size reduction by high pressure grinding rolls (HPGR) is by compressing and crushing the feed between two counter rotating, parallel rollers with a small gap between them. Compared to tumbling mills, HPGR achieves a size reduction by means of inter-particle compression within the particle bed rather than contact breakdown of coarse particles between rolling surfaces (van der Meer and Gruendken 2010). Table 2-2 presents the range of feed particle sizes suitable for different types of milling machines along with the corresponding expected product sizes. It also includes details on the expected throughput, the grinding stage, and the specific energy consumption measured in kilowatt-hours per ton for each piece of equipment.

Table 2-2 Summary of grinding mills

Grinding mill	Particle size	Expected Throughput (tph)	Specific Energy (kWh/t)	Grinding stage
HPGR	Feed: 95 mm	200-7200	1-3	Primary grinding/
AG/SAG	Feed: 100-200 mm	>2000	3-30	Primary grinding/
Rod	Feed: 19-50 mm	500	1.5-3.5	Primary grinding
Ball	Feed: 6-15 mm	100-1000	5-20	Primary/ Secondary

The challenge of size reduction normally arises when we ought to prevent oversizing and under sizing as, if not controlled may result in too fine or coarse material and would be bad for mineral liberation and recovery (Mwanga et al. 2014). Thus, size control is very important when it comes to mineral processing. The main grinding methods which are normally used are by tumbling, stirring and vibrations. These methods can be seen demonstrated in the figures below.

2.7 Breakage Mechanisms

The mechanism of breakage is referred to as the action of physical quantities, by means of force or energy, which when applied to a material introduces mechanical stress which in turn causes weakening or breakage of the material. Many terminologies have been used by researchers to describe the mode of breakage mechanisms. The popular terms used in literature are compression, impact, shear, attrition, and abrasion (Semsari Parapari et al. 2020).

Compression between two surfaces of a material causes the transformation of potential energy into elastic energy (Quist and Evertsson 2016). With brittle materials, elastic energy is possessed in the crystal lattice till the attainment of its peak level where micro-cracks are formed (Anderson 2017). In some instances, some materials have already existing micro-cracks in their natural state. The stress produced by compression is stored up in the tips of these cracks. As a result of the merging of the numerous cracks produced, fracturing takes place (Kranz 1983). Figure 2-3 depicts the breakdown of particles through compression between two surfaces moving against each other.

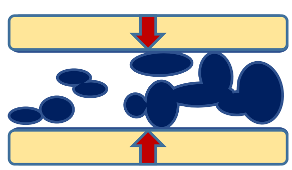


Figure 2-3 Illustration of compression of particles between two surfaces.

According to Kelly and Spottiswood, impact is the fastest form of breakage mechanism (Kelly and Spottiswood 1982). The extent of the impact breakage mechanism and its practicality is unpredictable. Nevertheless, with the aid of technology, simulation, and modelling, this can be known to an extent by means of machine geometry and function (Semsari Parapari et al. 2020).

Figure 2-4 depicts the breakdown of a particle as it is impacted by a hard surface, resulting in size reduction due to the force exerted on the particle.

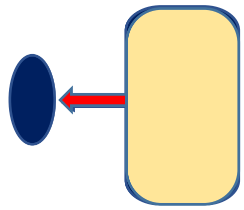


Figure 2-4 Illustration of impact of particle between two surfaces.

The major force that acts parallel to the surface of the material it is being applied to is known as the shear force. It tends to act parallel with the material and the machine or equipment in motion (Reem 2011). Figure 2-5 Illustration of shear force acting parallel with the particle and equipment. Figure 2-5 shows how a particle breaks apart when it's on a hard surface. Another surface presses against it, causing it to break because of the force. This kind of breakdown is similar to what happens when a jaw crusher breaks down particles.

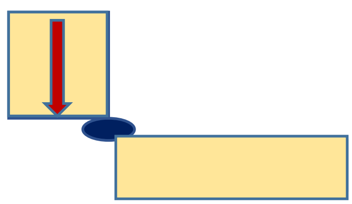


Figure 2-5 Illustration of shear force acting parallel with the particle and equipment.

Last but not the least, we have attrition and abrasion. Attrition refers to how a material is broken down by means of friction. Abrasion occurs because of attrition.   However, in some instances, attrition has been defined when materials collide between two other materials or media whereas abrasion is said to be when materials are rub against each other. Usually, both terms are closely related to the shear mechanism of breakage (Hennart et al. 2009).

Figure 2-6 illustrates how particles break down when two hard surfaces move against each other. The forces in action here are attrition and abrasion.

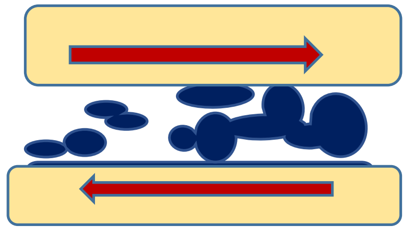


Figure 2-6 Illustration of attrition and abrasion forces with particles by means of friction.

2.8 Energy Use in Comminution (mining)

In recent times, there has been an exhaustion of high-grade ores and as such an increase in the extraction of low-grade ores. This however has two major implications especially for mineral processing which are processing exponential increase of tonnages of ore and the need to produce finer particles during comminution. These consequently results in the consumption of more energy (Awuah-Offei 2018). Comminution of ore has low energy efficiency even when it is considered in relation to the global use of electricity (Deniz 2013; Jeswiet and Szekeres 2016; Holmberg et al. 2017; Anderson et al. 2015; Baawuah et al. 2020). Ideally, to have a reduction with the amount of energy used for comminution is to basically avoid comminution where appropriate, however this is not quite practical with mineral extractive processes and cannot be relied on. Thus, the application of appropriate and efficient comminution technology is commendable (Reichert et al. 2015). The selection of an appropriate technology and grinding machine is usually dependent on the properties of the feed material, the specified final product, and the efficient production of the required products while consuming minimum amount of energy (Semsari Parapari et al. 2020). Table 2-3 shows the total net energies (E) required to reduce a particle from an infinite feed size to a product of size *x*.

Table 2-3 The theories Of Rittinger, Kick and Bond (Hukki 1961)

Theory	Equation	Description
Rittinger	$E = K_R \ ln\ ln\ (S_p - S_F)$ E ($J.kg^{-1}$) = The energy required per mass of feed K_R = Rittinger's constant	The net energy required in a certain process of comminution is proportional to the new surface formed.
Kick	$E = K_k \ ln\ ln\ \left(\frac{d_1}{d_2}\right)$ E ($J.kg^{-1}$) = The energy required per mass of feed K_k = Kick's constant	The net energy required in a certain process of comminution is proportional to the weight or size of the materials or ore worked on.
Bond	$E = W_i\left(\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}}\right)$ E ($J.kg^{-1}$) = The energy required per mass of feed W_i = Bond work index work	The net energy required in a certain process of comminution is proportional to the length of the freshly formed cracks which induces breakage

With comminution, the relation between energy and particle size reduction cannot be overlooked (Ballantyne et al. 2015). In the 19th century, Rittinger reported that the energy required for breaking down a material is proportional to the new surface area formed. Some years after this report, Kick also reported that the energy used in comminution is proportional to the weight and size of the material. However, during the 20th century Bond also proposed that the energy required for comminution is proportional to the length of new cracks produced and further explained that these cracks are what induces disintegration (Hukki 1961).

Later in the 1960s, Hukki established that these earlier comminution theories proposed may be dependent of each other but rather within a specific size range (Hukki 1961). According to Ballantyne et al. (2015) from experimental research, Tavares and King in 1998 showed that Kick's proposal was reliable especially for large sizes of materials where the surface energy is not significant relative to the internal energy needed for breakage.

2.9 Flotation

Sangita Mondal et al. (2021) defined froth flotation as a metallurgical process of extraction of minerals in a state pure from its ore. The process utilizes a unique ore separation technique that is mainly sued in mineral processing to enhance ore recovery (Piotr Pawliszak et al. 2024). Froth flotation has been used in mineral processing for decades (B.K. Gorain et al. 2000). Air bubbles are forced into tanks using aerators and agitators which attach to surfaces of cells which are facilitated and raised up in tanks. Copper oxide minerals are essential resources for copper production, these come in the form of malachite, cuprite, chrysocolla and azurite. Ore separation and recovery using froth flotation seizes vantage of the wet qualities at the surface of various material particles (Piotr Pawliszak et al. 2024) .

In a mineral beneficiation plant of copper, comminution is closely followed by froth flotation (Pearse 2005). Particle size is key when it comes to froth flotation, grinding is essential to achieve efficient mineral liberation. With grinding expending more energy at the processing plant, it is essential that the grinding process is optimized for energy efficiency while ensuring cost effectiveness at the same time (Ran et al. 2019). For this research, the flotation kinetics is considered, to study the relationship between the recovery of minerals and flotation time. Thus, recovery as a function of time with respect to microwave treated and reference materials with varying particle size ranges (200 and 250) µm.

The rate of flotation is dependent on bubble-particle attachment and transportation of particles from pulp to froth (Sahu et al. 2022).This study is intended to aid in the optimization of flotation process by enhancing flotation kinetics and efficiency.

The floatability of copper porphyry ore has been reported by (Sahyoun et al. 2005; Batchelor et al. 2016b; Gholami et al. 2020) to have shown improvement after microwave pretreatment. Microwave pre-treatment of a copper porphyry ore resulted in a 30 µm increase in particle size during grinding while still achieving the same copper recovery rate as the untreated material (Batchelor et al. 2016b). Microwave pretreatment enhanced the ability to float pentlandite when it existed in fine particles and improved the floatability of pyrrhotite when it was in coarser particle form. However, it had no impact on the recovery of pentlandite during the flotation process. (Da Silva and Waters 2018). The surface of chalcopyrite that underwent microwave pretreatment exhibited reduced roughness, a change attributed to the occurrence of surface oxidation and the creation of iron dioxide. (Barani et al. 2019). Flotation kinetics were directly linked to the surface roughness, meaning that microwave-pretreated samples necessitated an extended conditioning period within the cell. This observation carries significance for future studies involving high-power microwave treatment before conducting downstream flotation experiments. Changes in the surface properties of minerals following microwave treatment have been proven to influence subsequent processes in downstream operations. Sulphide ores, in particular, exhibit swift responses to heating, leading to the creation of oxidized surfaces. (Da Silva and Waters 2018). In addition to fracturing, microwave heating induces phase transformations at mineral surface grain boundaries, thereby modifying surface properties. These alterations can impact processes such as flotation. (Barani et al. 2019; Da Silva and Waters 2018). Nevertheless, numerous investigations have demonstrated enhanced flotation performance following microwave treatment.

According to a report by Kaya et al. (2020), the occurrence of phase transformations at mineral surface grain boundaries, leading to alterations in surface properties, necessitates careful consideration when applying microwave energy to mineral samples prior to comminution. This caution is particularly important given the critical role of surface chemistry in downstream processes. In this context, the economic comparison between the negative impact of microwave treatment on flotation and the advantages of improved liberation and energy efficiency due to microwave treatment was explored. The study revealed that the benefits of reduced grinding energy consumption and increased throughput could easily be nullified by a decrease in flotation recovery.

2.9.1 The Process of Froth Flotation in Copper

According to (Qicheng Feng et al. 2022; Ahmad Hassanzadeh et al. 2020; M. L. Shengo et al. 2019) froth flotation is a very complex yet widely applied technique utilized in separating raw ore materials in many extractive industries, the copper oxide minerals are no exception. The process utilizes the variations in the exterior qualities of the precious material and the gangue material. In the process of copper ore extraction, three complex processes are involved, these include solids, liquids, and gas (air bubbles called froth). Due to the effective surface hydrophobicity, high amalgamation rate, refined particle proportions and complicated material structure of copper oxide minerals, it is extremely difficult to effectively extract the mineral using other processes. After the valuable ore has been extracted from the rest of the waste rock, the process of froth flotation is then utilized to purposely concentrate and detach the precious ore from the gangue material. In the copper extraction approach, flotation is primarily used as an antecedent for hydrometallurgical or pyrometallurgical treatments as identified by Peterson Mutembei Kugeria et al. (2018). Due to the varying complexity and composition of copper ores, which can be in the form of carbonates, basic phosphates, oxides, or hydrates, it can contain a lot of impurities, hence needs an effective processing technique to extract the valuable mineral.

During the processing of copper oxide minerals, the fresh ore is exposed to crushing and grinding which has a substantial impact on the flotation procedure. Due to the ionic bonds on the surface of copper ores, it results in high levels of hydrophilicity. Concurrently, these behaviour of the ore makes it problematic for collector reagents to react with ore surface due to the presence of hydration shells. Sulfidization direct flotation and methods are then used where the reagents are added to alter the chemical properties of ore before the reactors are added to help extract from the sulfidized mineral surface.

The first process involved before the flotation process is the crushing of ore into powdery form. The suspense of the powdery ore is conveyed into a large tank and combined with water to form a solution. After the solution has been thoroughly mixed, collectors and froth stabilizers are added in increments. The collectors react with the fine particles of the solution. A rotating paddle called an impeller agitates the suspended solution. As a result of this agitation, an oily froth is formed on the exterior of the suspended solution which contains the ore particles in the form of bubbles. These particles move to the surface of the solution as they are lighter than the impurities within the mixture. Finally, the bubbles on the surface are skimmed off and passed through a dryer for ore recovery.

2.9.2 Flotation of Copper Porphyry Ore

Copper predominantly exists in the Earth's crust in the form of copper-iron-sulphide and copper sulphide-minerals. Porphyry copper deposits stand out as the most significant sources of global copper, contributing to 60% of the annual copper production (Aikawa et al. 2022). To meet the escalating global demand for copper, mining industries are actively exploring effective and efficient approaches to extract copper from increasingly deeper sources, considering its finite and depleting nature. Extracted ore from copper porphyry deposits is typically stockpiled after being retrieved from the Sulphide lode, and conventional froth flotation methods are employed for recovery. Due to the oxidic nature of these ores, concentration methods such as froth flotation or heap leaching are preferred to optimize cost-effectiveness.

Froth flotation, characterized as a physio-chemical process, exploits the electrochemical properties of mineral surfaces, lying between hydrophobic and hydrophilic states, induced either naturally or artificially by chemical reagents (Ndoro 2017). This separation technique involves the participation of three main states—solid, liquid, and gas. The process requires the attachment of air pockets to a mineral suspension known as flotation pulp. To facilitate this attachment, particles must be sufficiently small to adhere and be transported by the air pockets to the surface, subsequently accumulating in the froth layer.

In the pursuit of effective separation and concentration, the use of reagents plays a pivotal role in the froth flotation process. Therefore, in industrial plants, precise measurement and addition of reagents are crucial for optimal mineral recovery and the prevention of unwarranted losses to tailings. These reagents can be classified into frother, collectors, and modifiers.

2.9.3 Limitations of Flotation

The copper industry is experiencing challenges processing low-grade copper ores especially where the ore has high levels of pyrite content (Richard Li Jie Lee et al. 2022; J. Yianatos and P. Vallejos 2024). At present, a number of difficulties are being met by copper processing plants, the most prominent one is the decreasing copper head grade during processing which is indirectly correlated with the high demand of the ore resulting on the exertion of significant pressure on processing plants to preserve the required flotation occurrences for copper production (Yongjun Peng et al. 2003; Maedeh Tayebi-Khorami et al. 2018). Another challenge found by (Saeed Farrokhpay et al. 2018) indicates that copper contains different clays such as kaolinite and bentonite which affects the overall ore recovery after metallurgical processes.

Another major challenge faced by mining industries during the processing of copper ores is the high volumes consumed, raising sustainability issues particularly in areas where access to water is quite limited (Sayed Janishar Anzoom et al. 2024).

During the recovery phases of particles in flotation, there are three main stages involved. These include particle collection using bubbles formed in the pulverize phase, bubble-particle aggregate transfer and lastly the recovery of the particles at the froth phase. The effectiveness of the first step during the flotation process has a dire consequence on the amount of ore to be recovered after the process. The bubble element collision effectiveness depends on the particle sizes, the rhythm of motion of the hydrophilicity of these bubbles (Ahmad Hassanzadeh et al. 2016)

Lastly, the high energy dispensed during froth flotation of copper ore porphyry due to a decline in the feed grades has had a major impact, increasing milling energy consumption by 50% –100% when the copper ore grades fed to the processing plants decreases below 1% (J. Yianatos and P. Vallejos 2024).

2.10 Liberation of Ore

Mineral liberation is ultimately the most important part of efficient processing of minerals. Excessive grinding which is not cost effective in its sense may negatively impact ore liberation and in the same vein, poor grinding kinetics causes inefficient liberation and hence losses. Hence an in-depth knowledge about ore liberation is important. It is the process by which valuable mineral grains or particles are made distinct from invaluable particles. Reliant on which separation method is being used, there are some factors that must be taken into consideration to ensure efficient liberation. Flotation for instance, is dependent on the surface properties and as such a greater exposed mineral surface is needed compared to leaching and gravity separation. Whereas gravity separation is dependent on the differences in the specific gravity of the mineral particles and the gangue, flotation is highly dependent on the particle sizes. The finer the sizes the more liberation takes place and vice versa. It should however be noted that, complete liberation does not translate to full recovery. To ensure efficient recovery, the grinding circuit must produce an ideal size range for optimum performance (Bradshaw et al. 2019; Sousa et al. 2018).

2.10.1 Liberation Measurement Techniques

The initial point to consider when measuring liberation is the differences with the chemical composition or grade and particle size of the mineral of interest. A prior knowledge about the variation in grade and particle size of the mineral grains usually helps in determining the degree at which liberation is adequate or not. Critical evaluation of the mineral grain distribution in the particles that make up the stream to be separated is important for the measurement of mineral liberation. Characterizing the mineral grain distribution are mainly categorized into; two-dimensional (2-D) measurements obtained from polished sections of ore particles and three-dimensional (3-D) measurements obtained from whole ore particles by either physical measurement or nonintrusive methods such as 3-D tomography. Image analysis methods using optical and Scanning Electron Microscope with Energy Dispersive X-Ray Spectrometer (SEM-EDS) platforms are usually used and the most accepted of the technologies. Automated image analysis instruments such as (e.g., Quantitative Evaluation of Minerals by Scanning Electron Microscopy [QEMSCAN], Mineral Liberation Analyzer [MLA], Mineralogic, and TESCAN Integrated Mineral Analyzer [TIMA-X]) are mostly used on site at mineral processing plants (Bradshaw et al. 2019). In this present study, QEMSCAN analysis was utilized.

2.10.2 Categories of Ore Liberation

An ore is said to be liberated if more than 90% of the particles are composed of the mineral of interest. When between 30% and 90% of the particles are composed of the mineral of interest it is referred to as middling. When less than 30% of the particles are composed of the mineral of interest, it is said to be locked per a report by Bradshaw et al. (2019). However, in this present study, the QEMSCAN analysis conducted by XPS grouped the minerals of interest in liberation classes based on their area percent value as follows: locked (<30%), low grade middling (30–50%), medium grade middling (50–80%), high grade middling (80–95%), liberated (>95%) and free (100%). This is to emphasize that dependent on the particular equipment and techniques used for the mineral characterization, different studies and analysis may classify liberation differently.

Chapter Three

3 Methodology

This chapter presents an overview of the methodology adopted for conducting the research. The methods and techniques employed in data collection, analysis, and interpretation are discussed in detail. The chapter is organized to provide a clear understanding of how the research was executed, ensuring transparency and reproducibility. To minimize excessive grinding of ore materials to ensure efficient liberation, microwave pretreatment was utilized, and a suitable size range chosen for a separation process after QEMSCAN analysis is done. Figure 3-1 illustrates a flowchart depicting the various methodologies utilized and the workflow. Further elaboration on these methods is provided in this chapter.

Figure 3-1 shows a flowchart illustrating the methodologies employed and the workflow.

3.1 Materials and Reagents

The test material was a copper porphyry ore from Highland Valley Copper (HVC) in the Province of British Columbia in Canada. The copper bearing minerals were chalcopyrite, chalcocite, bornite, as well as some oxide copper minerals such as malachite and cuprite. The main iron bearing minerals were pyrite and marcasite. The molybdenite form was molybdenite. The significant amounts of non-sulfide gangue minerals were quartz, chlorite, illite and biotite. Reagents used for the flotation process are, Lime - [Ca $(OH)_2$] (10 %) (10 g/t), Potassium amyl xanthate (PAX) - (0.5 %) (1 g/t), Sodium Isopropyl Xanthate (SIPX) - (0.5 %) (1 g/t), Methyl Isobutyl Carbinol (MIBC) (12 g /t), Pine oil (3 g /t) and Fuel oil (3 g/t). Table 3-1 shows the Minerals found in the copper porphyry ore and their respective chemical formulae.

Table 3-1 Minerals found in copper porphyry ore and their chemical formulae

Mineral	Chemical Formula
Chalcopyrite	$CuFeS_2$
Chalcocite	Cu_2S
Bornite	Cu_5FeS_4
Malachite	$Cu_2CO_3(OH)_2$
Cuprite	Cu_2O
Pyrite	FeS_2
Marcasite	FeS_2
Molybdenite	MoS_2
Chlorite	$(Mg, Fe)_3(Si, Al)_4O_{10}(OH)_2(Mg, Fe)_3(OH)_6$
Illite	$(K\ H_3O)\ (Al, Mg, Fe)_2\ (Si\ Al)_4\ O_{10}\ [(OH)_2\ (H_2O)]$
Biotite	$K\ (Mg, Fe)_3\ AlSi_3\ O_{10}(F, OH)_2$

Using Quantitative Evaluation of Materials by Scanning Electron Microscopy (QEMSCAN), the mineralogy of both the reference and microwave-treated materials was characterized, allowing for a detailed assessment of mineral liberation and distribution. The mineralogical data presented in Table 3-2 were obtained from QEMSCAN analyses conducted by Expert Process Solutions (XPS). QEMSCAN is an advanced analytical tool that provides detailed mineralogical information by scanning a polished sample surface with an electron beam, mapping the mineral phases present based on their unique chemical compositions and crystal structures.

Table 3-2 Assay Results of Mineral Elements in Reference and Microwave-Treated Material

	Assay of mineral elements	**Reference material**	**Microwave treated material**
QEMSCAN Measured Chemical Assay (%)	Aluminium (Al)	8.12	7.83
	Calcium (Ca)	1.62	1.75
	Copper (Cu)	0.18	0.23
	Iron (Fe)	1.71	1.86
	Magnesium (Mg)	0.22	0.23
	Sulphur (S)	0.16	0.19
	Silicon (Si)	33.20	33.30

3.2 Sample Preparation

A CR-201, Terminator Jaw Crusher, by TM Engineering Limited, Canada was used for crushing. The HVC (Highland Valley Copper) ore samples were crushed and screened into particle size fractions of (-6.7 and +4.75) mm, from which 10 kg of material was taken out using the coning and quartering method for further analysis (microwave treatment and grinding). Afterwards, the sample was rotary split into 200 g charges. Microwave treatment was performed on half (5 kg) while the other half (5 kg) was stored and used for reference purposes. After microwave treatment, both the reference and microwave treated samples were further crushed into particle size fractions below 2 mm for flotation test experiments conducted at Sepro Minerals lab. (-6.7 and +4.75) mm and (-3.35 and +1.7) mm size fraction were used for time grind tests conducted at the University of Alberta. Particle Size Analysis of the samples (MWT and reference) were conducted for size fractions (below 2 mm, for both the (-6.7 and +4.75) mm and (-3.35 and +1.7) mm samples after the grind tests.

3.3 Microwave Treatment

3.3.1 Bench-scale Microwave Heating Behaviour Tests

A BP-211 laboratory microwave system with 2450 MHz, 3.2 kW manufactured by Microwave Research and Applications, USA was used for the bench-scale experiments. The reason for this test is to study the effect of particle size, sample mass, and power input on the microwave heating behaviour of the samples.

Microwave heating curves give first-hand knowledge on how material responds to microwaves upon exposure. The curves were also useful in determining microwave exposure times of ore samples before grinding tests are conducted. Usually, a plot of temperature against heating time gives a microwave heating behaviour curve.

A quartz crucible containing 50 g of sample, with a size fraction ranging from (-3.35 and +1.7) mm, was placed inside the reaction chamber of the laboratory bench-scale microwave system. The choice of a particle size of 3.35 mms was based on the typical feed size used in ball mill grinding tests. The test was conducted with an 8-minute residence time. Following each test, the quartz crucible was promptly removed from the microwave system using thongs to ensure safety, and a thermocouple probe was inserted into the centre of the sample to record the maximum temperature. Afterward, the sample was left to cool for approximately an hour, and its mass was measured and recorded.

3.3.2 Bench-scale Microwave Pre-treatment Tests

The microwave-assisted comminution investigation focused on a particle size fraction of material passing through a 2-mm sieve using the bench-scale microwave. The treatment conditions included 3200 watts of power, a frequency of 2450 megahertz MHz, and an 8-minute residence time for both 50 g and 200 g charges of sample, respectively, for the (-3.35 and +1.7) mm (-3.35 and +1.7) mm size range. Additionally, a 200 g charge for 8 minutes, also at 3200 watts power and 2.450 GHz frequency, was applied for the (-6.7 and +4.75) mm size range.

For both the (-6.7 and +4.75) mm and (-3.35 and +1.7) mm size ranges, five 200 g samples from the microwave-treated samples were combined into one charge to serve as feed material for grinding experiments conducted at the University of Alberta. Table 3-3 outlines the experimental conditions for microwave-assisted comminution conducted at the bench scale. It provides details such as power, frequency, residence time, size fraction of the sample, mass of the sample, and its use after treatment for the different test scenarios as explained.

Table 3-3 Experimental Conditions for Microwave-Assisted Comminution at Bench Scale

Power (Watts)	Frequency (MHz)	Residence time (minutes)	Size fraction of sample (mm)	Mass of sample (grams)	Purpose for treatment
3200	2450	8	(-3.35 and +1.7)	50	Test was conducted to investigate how the sample reacts to microwave treatment
3200	2450	8	(-6.7 and +4.75)	200	Combined with 5 samples (200 g each) to make a total of 1000 g, samples were further crushed into particle size fractions below 2 mm for flotation tests
3200	2450	8	(-3.35 and +1.7)	200	Combined with 5 samples (200 g each) to make a total of 1000 g. This batch was used for time grind tests for particle size analysis.
3200	2450	8	(-6.7 and +4.75)	200	Combined with 5 samples (200 g each) to make a total of 1000 g. This batch was also used for time grind tests for particle size analysis.

Figure 3-2 illustrates the bench-scale microwave oven, as provided by University of Toronto at Sepro Minerals in Langley. This equipment, utilized in the experimental setup, played a pivotal role in conducting the bench scale microwave treatment of the samples. The image depicts the chamber where the sample is placed for treatment, showcasing its sufficient size to accommodate sample containers or crucibles. Observation windows or ports on the chamber walls allow researchers to monitor the sample during treatment without interrupting the process. Additionally, the picture highlights the control panel located on the exterior of the oven, containing various controls and settings for adjusting parameters such as power, frequency, and treatment duration. Indicators or displays on the panel offer real-time feedback on the status of the microwave treatment process, including temperature or power levels. These features enable precise control and monitoring of experimental conditions, ensuring the reliability and reproducibility of research findings.

Figure 3-2 Bench scale microwave oven

(Courtesy UofT at Sepro Minerals, Langley)

Figure 3-3 presents the systematic methodology applied in conducting microwave bench-scale experiments within this study. This flowsheet visually outlines the sequential steps involved in the experimental process, guiding the investigation from initial material preparation to subsequent data analysis. The methodology initiates with the preparation of feed material. Coarser samples are crushed to specific size ranges to ensure consistency across subsequent processing stages. For samples sized (3.35 and +1.7) mms, Ro-Tap screening is conducted for classification purposes. Subsequently, there is rifle splitting of these samples into two separate batches: one into 50-gram portions and another into 200-gram portions. Both batches undergo microwave heating tests using the experimental setup depicted in Figure 3-2, followed by grinding tests to assess the impact of microwave pretreatment on comminution processes for the 200-gram portions. However, the 50-gram portions are subjected to microwave heating tests only to investigate the immediate effects of microwave treatment without subsequent grinding.

Additionally, another batch of feed material, crushed to sizes (-6.7 and +4.75) mms, undergoes the same initial treatment. It is subjected to Ro-Tap screening, followed by riffle splitting into 200-gram portions before microwave heating tests and subsequent grinding tests, using the same experimental setup as depicted in Figure 3-2.

After microwave heating tests and grinding, the experimental process concludes with particle size analysis, providing detailed information on the particle size distribution of the processed material. This analysis offers valuable insights into the effectiveness of microwave pretreatment on particle size reduction and distribution, enhancing understanding of its effects on material properties.

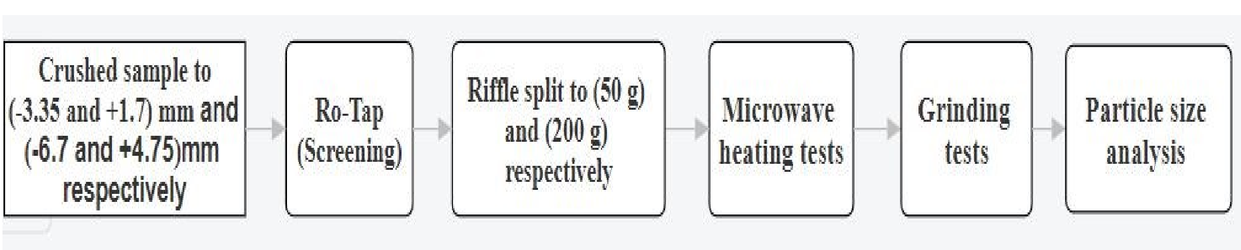


Figure 3-3 Flowsheet for microwave bench-scale work

3.3.2.1 Bench-scale Microwave Heating Tests for Individual Coarse Rocks

Coarse rocks of (-63.5 to +50.8) mm size range were individually treated at the bench-scale. These rocks were numbered from 1 to 50 for better identification. Figure 3-4 shows a picture of the numbered rocks on the tray before microwave treatment. After these rocks were treated, a Forward Looking Infrared (FLIR) A8300sc handheld camera was used to take pictures to identify the hotspots (temperature) of the rock after microwave treatment. The individual coarse rocks were treated at the bench-scale till breakage occurred or experiment was stopped due to multiple electric glow discharges. Most of the rocks could withstand 120 seconds of residence time with breakage experienced between 60s to 90s . A few had massive glow discharges and pop sounds between 5s and 15s from the start of the experiment. This experiment was conducted to assess the effects of microwave energy on the treated material. Parameters such as heating efficiency, temperature profiles, exhibited material discharges, and the sensory effects during the heating process were meticulously monitored and analysed during this phase.

Figure 3-4 A tray of individually numbered rocks before microwave treatment.

3.3.3 Pilot-Scale Microwave Heating Tests for Coarse Rocks

The pilot plant microwave system located at Sepro Minerals at Langley, BC was designed and constructed by Thermowave (Danvers, MA, USA). It is equipped with two 75 kW microwave generators with a total power of 150 kW (from both).

Each Wave Generator includes a water-cooled (19 L/min) magnetron tube which produces microwaves at a frequency of 915 MHz. A water circulator dissipates any reflected microwave power thereby preventing any damage to the magnetron. The system is controlled via an HMI control panel equipped with an Allen Bradley PLC. The applicator is 1 m long, with a microwave transparent polypropylene bridge, and a built-in arc sensor.

The conveyor belt is made of microwave transparent habasit silicone/polyester, capable of reaching speeds of up to 5 m/s. A Forward Looking Infrared (FLIR) A8300sc infrared (IR) camera, which is located 180 cm above the belt as seen in Figure 3-5 records a video of the sample trays and transfers the data via a Gigabit Ethernet port connected to FLIR's Research IR software.

Figure 3-5 provides a detailed overview of the pilot-scale microwave system, situated within the Bobicki lab at Sepro Minerals, Langley, BC. This image offers valuable insights into the experimental infrastructure utilized in our research endeavours. The pilot-scale setup, reminiscent of a pilot plant in its design and functionality, is meticulously arranged to facilitate efficient operation and data collection. From large-scale equipment to intricate instrumentation, every component plays a crucial role in advancing our understanding of microwave-assisted mineral processing techniques. This comprehensive setup serves as the cornerstone of our experimental approach, enabling us to conduct rigorous investigations and push the boundaries of scientific exploration in mineral processing.

Figure 3-6 illustrates the systematic methodology adopted for conducting microwave pilot-scale experiments in this study. The flowsheet provides a visual representation of the sequential steps involved in the experimental process, guiding the investigation from material preparation to data analysis. The methodology commenced with the preparation of the feed material, which was crushed to a specific size range (-63.5 to +50.8) mm. This initial step ensured uniformity and consistency in subsequent processing stages. Following material preparation, the experimental workflow progressed to microwave heating tests, a pivotal phase in the investigation. Microwave heating experiments were conducted at the pilot scale to assess the effects of microwave energy on the treated material. Parameters such as heating efficiency, temperature profiles, exhibited material discharges, and the sensory effects during the heating process were meticulously monitored and analysed during this phase. However, due to unforeseen constraints or limitations, the last two steps of the methodology, including grinding tests and QEMSCAN analysis, were not executed as initially planned. It's important to acknowledge these deviations from the intended methodology to maintain transparency and integrity in the research process.

Figure 3-7 shows pictures of the ore samples arranged on the tray and on the conveyor belt for treatment. The size fraction of crushed sample (-63.5 to +50.8) mm was used. The use of the pilot plant microwave system becomes imperative as high-power tests are more on the applied side as opposed to fundamental bench scale. After the general heating behaviours, and the potential for efficient downstream processes has been determined by the bench-scale, these can be projected onto pilot experiments to establish its further practicability on a large scale.

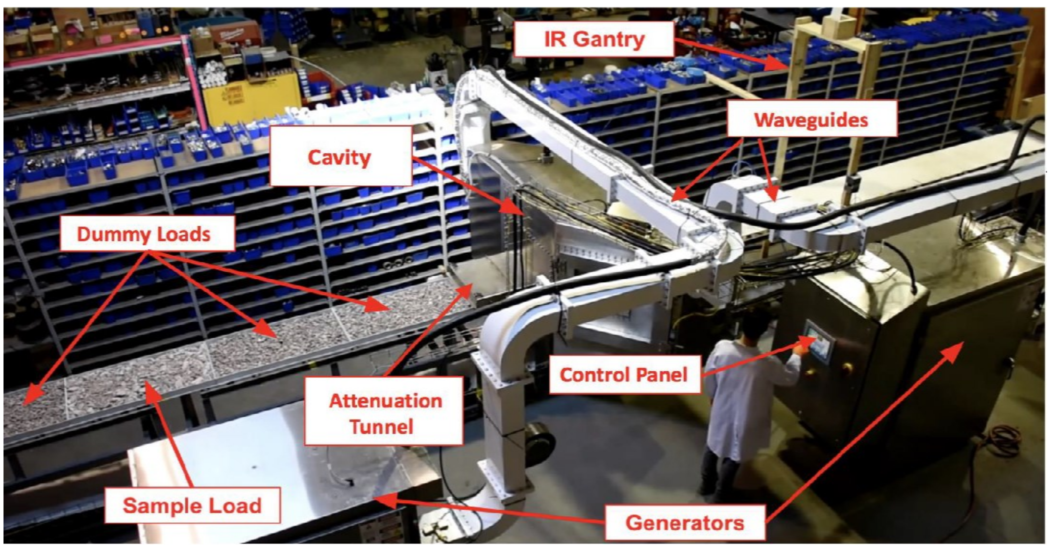


Figure 3-5 Overview of pilot-scale microwave system (Forster 2023)

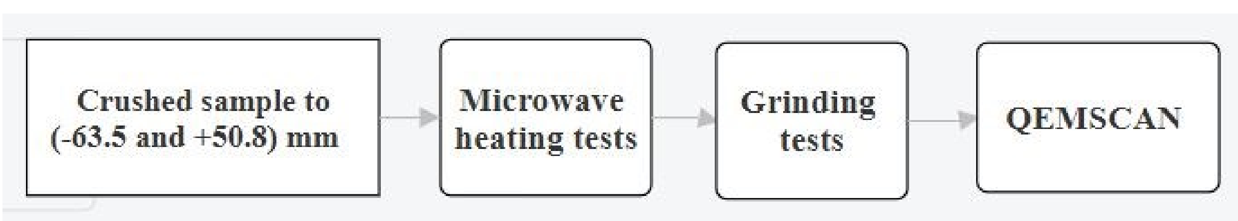
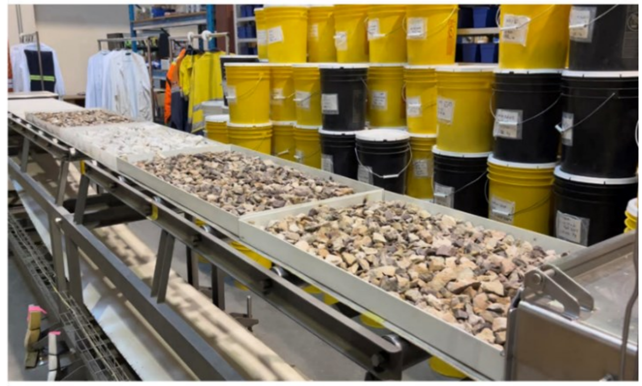


Figure 3-6 Flowsheet for microwave pilot-scale work.

Packed ore material on tray



Packed trays on conveyor for treatment

Figure 3-7 Trays with packed ore material for continuous pilot microwave treatment.

Table 3-4 shows the input data for the parameters used during the continuous pilot plant microwave treatment of the (-63.5 to +50.8) mm size fraction. There were five (5) treatment cycles in all. The first three (3) treatment cycles had 6 kWh/t electrical energy dose input while treatment cycles 4 and 5 had 10 kWh/t and 8 kWh/t electrical energy dose respectfully. These variations were necessary and were made to better observe and determine how the ore sample behaves or responds to microwave-treatment. Other parameters such as initial temperature, input microwave power, conveyor belt speed and mass of the load or sample per tray are also recorded in Table 3-4.

Table 3-4 Input data for continuous pilot plant test experiments

Data	Description		Treatment Number				
			1	2	3	4	5
Input Parameters	**Initial Temperature**	(°C)	20.8	20.7	22	24.8	25.7
	Input MW Power	kW	120	150	150	150	150
	Conveyor Belt Speed	m/s	0.53	0.66	0.66	0.40	0.50
	Load Mass	kg	13.90	13.90	13.90	13.90	13.90
	Load PSD	mm	-63.5 to +50.8	- 63.5 to +50.8	-63.5 to +50.8	-63.5 to +50.8	-63.5 to +50.8
	Elec. Energy Dose	kWh/t	6	6	6	10	8

3.4 Grinding Test Procedure for Particle Size Analysis

A jar mill was employed for timed grind tests to compare the Particle Size Distribution (PSD) of HVC ore samples. The comparison involved both coarser (-6.7 and +4.75) mm and finer (-3.35 and +1.7) mm size fractions. To maintain consistency, the same grinding time was utilized for each batch of reference material and microwave-treated samples. The chosen milling time aimed to achieve a product size of 80% passing 200 μm, aligning with the product size introduced from the mill to the flotation circuit at the parent company. A jar mill with the parameters outlined in Table 3-5 was employed for milling. The milling process was carried out at a feed charge of 1000 g and 50 % solids.

The grinding tests comprised a total of 16 batches: 8 batches of reference material and 8 batches of microwave-treated material for both the coarser and finer size fractions. For comparison, grinding times of 15 minutes and 20 minutes were used for both the reference and treated material across both size ranges. After grinding, each batch was size classified following drying in the oven. Size classification was conducted using a ROTAP® for 15 minutes. The mass in each size fraction was recorded, and corresponding size fractions from each batch were combined for subsequent analysis of the microwave-pretreated and reference samples.

Table 3-5 Parameters for test procedure for particle size analysis

Jar mill parameters	**Value**	**Parameters for grinding test procedure**	**Value**
Ore Charge volume (cm^3)	210	Feed charge	1000 g
Mill volume (cm^3)	8648.89	Solids concentration	50 %
Ore Volume Loading (%)	2.43	Grinding Time (per batch) for Reference Material	15 minutes, 20 minutes
Weight of steel balls (kg)	8.04	Grinding Time (per batch) for Microwave-Treated	15 minutes, 20 minutes

3.5 Grinding Test Procedure for Flotation Tests

After microwave treatment of the (-6.7 and +4.75) mm size range, the ore was crushed to -2 mm before introducing it into the rod mill, at a feed charge of 1000g and 65% solids. A P_{80} of 200 µm is what is used/introduced at the mother company's flotation circuit as this enabled the efficiency and effectiveness of the separation process. This procedure aims to determine whether microwave treated samples of same size fraction or coarser fractions (200 µm and 250 µm) may have significant enhanced liberation benefits.

The rod mill depicted in Figure 3-8 is a critical component of the batch flotation process utilized in this study. This apparatus is specifically designed for grinding ore samples to a fine particle size, a crucial step in the preparation of material for the subsequent froth flotation experiments. The rod mill operates by tumbling rods within its cylindrical chamber, effectively breaking down the ore into smaller particles through repeated impacts and abrasion.

During operation, ore samples are introduced into the mill along with grinding media, typically steel rods, which serve to further fracture the material as they cascade and tumble within the mill. The rotation of the mill imparts kinetic energy to the ore particles, resulting in size reduction through the combination of impact and attrition forces. The efficiency of the grinding operation directly impacts the effectiveness of the froth flotation process, as finer particles facilitate better liberation of valuable minerals from the ore matrix.

In this study, the rod mill was operated under controlled conditions, with parameters such as rotational speed, grinding time, and feed rate carefully monitored and adjusted as needed. The resulting ground material was then subjected to batch flotation tests to assess the response of the ore to flotation under varying conditions.

Figure 3-8 A picture of the rod mill used for grinding for batch flotation.

Table 3-6 provides an overview of the parameters utilized during the grinding of ore samples, including both the reference samples (labelled 1R and 2R) and the samples subjected to bench microwave treatment (labelled 1M and 2M). Each sample batch, consisting of 2000 g of material, was ground to achieve a target particle size distribution, as indicated by the P_{80} value (the particle size at which 80 % of the material passes through the sieve) of 200 µm and 250 µm respectively. The grinding process was conducted under a solid content of 65 %, ensuring optimal conditions for particle breakage and efficient energy utilization.

The grinding duration, denoted as "Grind time," was set to 9.9 minutes for both the reference and microwave-treated samples at a P_{80} of 200 µm and 7.8 minutes for a P_{80} of 250 µm. This standardized grinding time allowed for consistent processing conditions and facilitated direct comparison between the two sample groups.

Additionally, reagents were added to the samples before the grinding process to enhance the efficiency of particle breakage and flotation recovery. Specifically, each sample received 0.5 mL of 10 % lime solution, along with 2 drops each of pine oil and fuel oil. These reagents were carefully selected to promote mineral surface modification and improve the selectivity of the flotation process.

Table 3-6 Parameters used for grinding samples (Reference & Continuous Pilot Plant Microwave Treated; 1R (first batch of sample for Reference and 1M for first batch of sample for Microwave Treated).

Samples	**P_{80} (µm)**	**Weight (g)**	**% Solids**	**Grind time (mins)**	**Reagents Added Before Grinding**
1R & 1M	200	2000	65	9.9	0.5 ml of 10% lime, 2 drops each of pine oil and fuel oil
2R & 2M	250	2000	65	7.8	

3.6 Batch Flotation Tests

Flotation tests were conducted on both the reference and microwave treated HVC ore samples with P_{80} values of 200 and 250 µm at 65 % solids. Where HVC 1R and 2R represent the reference materials whereas 1M and 2M represent the microwave treated materials corresponding to the particle size of the samples as stated. Flotation tests for the microwave treated HVC ore samples and its corresponding reference samples were conducted. The parameters used for grinding the samples is as seen in Table 2 below.

Before grinding, 0.5 mL of 10 % lime for the modification of pH was introduced into the mill. Two (2) drops of pine oil (frother) and fuel oil (collector) were also added to the mill before grinding to ensure better particle-surface interactions and eventually aid methyl isobutyl carbinol MIBC (frother) which is to be added during the conditioning steps in the flotation process. The surface tension of the liquid-gas interface is reduced by the frother which aids in the production of froth that acts as a medium in the separation of the hydrophobic from the hydrophilic minerals (mineral of interest from gangue) (Sahu et al. 2022)

3.6.1 Parameters Of Apparatus Used for The Batch Flotation Process

A SEPOR Denver style D-12 laboratory flotation machine with a cell of 5 L volume capacity and an agitator with speed of 1200 rpm as can be seen in Figure 3-11 was used. During the flotation process, a Hanna HI2210-01 Benchtop pH meter with 0.01 pH resolution was used to measure and monitor the pH and oxidation-redox potential (mV). A pH of 9.5 to 10 was maintained by adding drops of 10 % lime. Potassium Amyl Xanthate (PAX) and Sodium Isopropyl Xanthate (SIPX) both of which are collectors are introduced into the feed mixture at stipulated times during the conditioning steps as can be seen in Figure 3-9. The corresponding conditions are provided in Table 3-7. Collectors enhance the hydrophobic properties of the mineral particles which is very significant to promote the adhesion of the mineral particles to the air bubbles present.

Table 3-7 below shows the conditions by which the flotation test was conducted.

Table 3-7 Experimental conditions for batch flotation

Parameters	Value	Unit
Product particle passing (P_{80})	200, 250	µm
Initial solid concentration (pulp density solids by weight)	65	%
Slurry volume	5	litres
Impeller speed	1200	rpm
pH	9.5 to 10	-
Lime (10 %) (10 g/t)	0.2	ml
PAX (0.5 %) (1 g/t)	0.4	ml
SIPX (0.5 %) (1 g/t)	0.4	ml
MIBC (12 g /t)	6	drops
Pine oil (3 g /t)	2	drops
Fuel oil (3 g/t)	2	drops

During the flotation tests, three (3) rougher concentrates, two (2) scavenger concentrate, two (2) scavenger tails and the head sample was cut at stipulated times as can be seen in Figure 3-9 Collected froth (samples) from the cuts during flotation were dried at a temperature of 80 °C and later pulverized for XRF analysis

Figure 3-10, a schematic diagram of the froth flotation batch process is presented. The diagram provides a visual representation of the batch flotation process. During the flotation process, various reagents are added to the slurry in the flotation cell at stipulated times as can be seen in Figure 3-9 to selectively modify the surface properties of the minerals present. These reagents facilitate the attachment of hydrophobic particles to air bubbles and promote their flotation to the surface of the cell. As the flotation process progresses, air is introduced into the cell, the air bubbles generated attach to the hydrophobic mineral particles, forming a froth layer at the surface of the slurry. The froth layer, enriched with hydrophobic mineral particles, is then skimmed off the top of the cell and collected in a container. This rougher concentrate contains the desired minerals of interest and is subsequently subjected to further processing steps, that is dewatering using a sample pressure filter after which the sample was dried. Meanwhile, the remaining slurry in the flotation cell, known as the tailings or scavenger concentrate, contains the gangue material and any non-recovered minerals. The scavenger concentrate is also collected from the flotation cell and pressure filtered and then dried at 85 °C for 6 hours. Both the dried rougher and scavenger concentrates samples are later pulverized and a portion taken out for XRF analysis, the remaining are sent for QEMSCAN analysis.

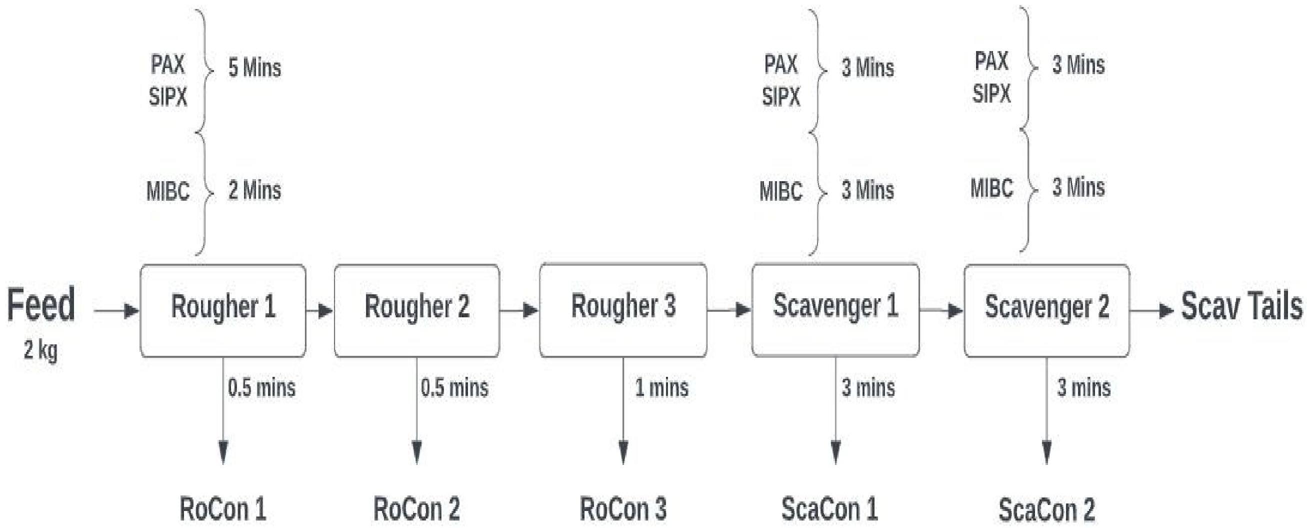


Figure 3-9 Flow chart of the froth flotation batch process.

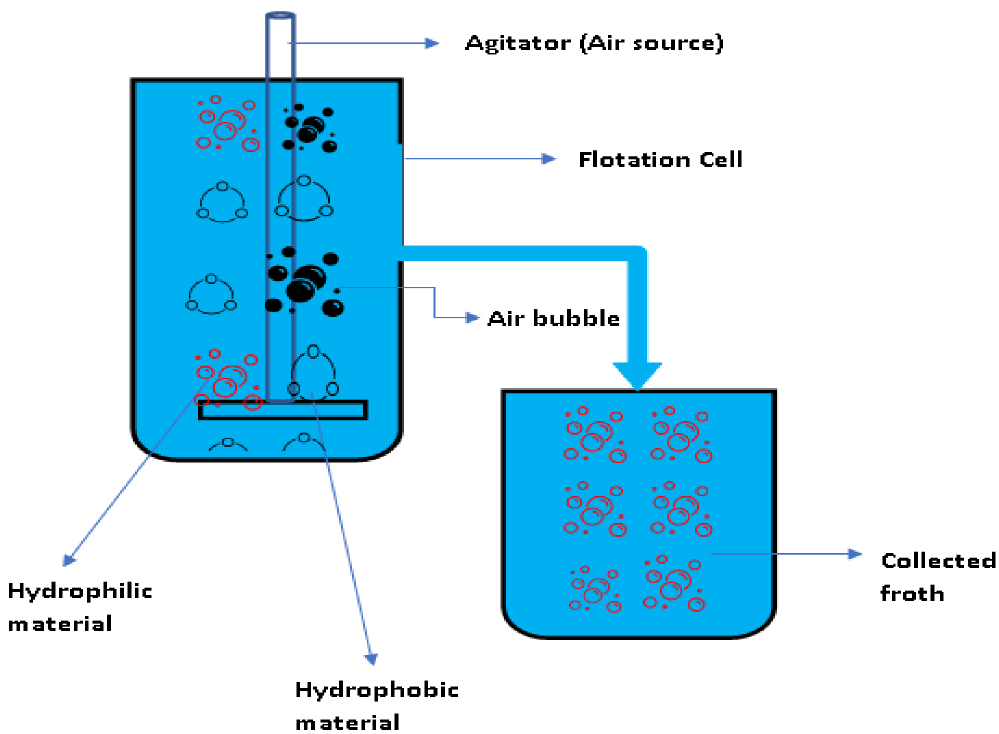


Figure 3-10 Schematic diagram of the froth flotation batch process

Batch Flotation Cell Setup

Figure 3-11 A picture showing the set up for the batch flotation.

3.7 X-ray Fluorescence (XRF)

X-ray diffraction analysis (XRF) analyses were carried out to determine the elemental composition of the dried and pulverized samples. The Epsilon 1 benchtop energy dispersive X-ray fluorescence spectrometer was used for the elemental analysis. This information was used in calculating the metallurgical assays (feed head grade, recovery) of the dried samples after the batch flotation tests. The finely pulverized sample is split, and a mass of 2 g per sample is used for the analysis. The results are used to calculate the cumulative grade for the necessary elements, the head assays and recovery.

3.8 QEMSCAN

Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN) was done on the respective ores (at various grind times). The QEMSCAN instrumentation uses computer grey-scale backscatter electron (BSE) imaging in combination with a custom-representation of X-ray spectral window database to identify phases of interest. This procedure makes it possible to know the mineralogy and liberation. The samples were evenly divided, mounted in epoxy, polished, carbon coated, and taken for imaging. The number, size, mineral composition, and associations of the particles were calculated during the analysis. The liberation degree of the target minerals are represented as an area percent. The mineralogical data were obtained from QEMSCAN analyses conducted by Expert Process Solutions (XPS). The samples were measured using a combination of either FEI QEMSCAN 650 (tungsten filament) or QEMSCAN 650 FEG (Field Emission Gun). The beam conditions were set to a beam current of 10 nA and accelerating voltage of 25 kV. The data acquisition was completed with iMeasure with a point spacing of 1µm to 2.5 µm (depending on sample particle size). The data was processed with iDiscover using a Species Identification Protocol (SIP) designed for base-metal sulphide ores.

The data from this work will allow one to calculate the degree of liberation as a function of particle size, mineralogy, particle size distribution etc. This is essential so that future concentration tests may be conducted at the optimum grind sizes. After microwave treatment of the ore, phase transformations are expected especially for the bench-scale which will most likely affect subsequent downstream processes. The goal of the QEMSCAN of the current work is to observe whether coarser particle size of microwave pre-treated ore may be used during flotation, without compromising liberation and recovery, which will then reduce energy consumption during grinding. The precision of the QEMSCAN results will be verified by comparing them with X-ray diffraction (XRF).

False colour maps are used to identify which mineral is present and in which form, either locked, free and or liberated.

Chapter Four

4 Results And Discussion

This section of results and discussions presents and explains the experimental results. The main objectives of this research were to evaluate the influence of microwave treatment on mineral liberation, investigate the influence of microwave treatment on particle size distributions, and determine the optimal operating parameters for the microwave-assisted comminution of copper porphyry ore. These objectives are directly related to the results presented in this section as they guide the analysis and interpretation of the data collected. By evaluating the degree of mineral liberation achieved, examining the effects on particle size distribution, and identifying optimal operating parameters, this section aims to provide insight into the efficacy and potential applications of microwave technology in ore comminution. Discussion of the findings, comparing them with traditional methods and considering the relevant literature, it is aimed to provide a broader understanding of the impact and significance of microwave-assisted comminution using copper porphyry ore as the case study.

4.1 Presentation of Experimental Results

4.1.1 Bench-scale Microwave Pre-Treatment of Ore

The microwave heating curves and corresponding heating rate curves for the HVC ore (-3.35 and +1.7) mm size fractions which were bench microwave treated at 50 g and 200 g charge.

Figure 4-1 shows the comparison of the effect of heating time on sample mass. It can be observed that, higher temperatures were recorded for the 200 g as opposed to the 50 g sample using the same heating time. This variation can be attributed to heat capacity being directly proportional to mass of a material.

Figure 4-2 shows the microwave heating rate curve, which shows the heating rate against the change in temperature through the course of the microwave treatment for 8 minutes for (-3.35 and +1.7) mm 50 g charge. The heating rate is an important parameter because it enables efficient utilization of microwave energy influencing energy efficiency, treatment effectiveness, and process control. Efficiently controlling the heating rate allows for desired outcomes during the treatment process. The maximum heating rate was about 66 °C/min. with a corresponding change in temperature of 33 °C. A maximum temperature of 268.1 °C recorded at 8 minutes, with a corresponding mass loss of 2.3 % was achieved after 8 minutes of microwave heating time.

The mass loss is highly likely to be bound water loss, in relation to a report by Pillai et al. (2018). The mass loss is less likely to be sulphur since there were no sulphur gas emissions or gas perceived during the experiment. However, to efficiently know the cause of the mass loss, a Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) analysis is recommended to know the exact reason behind the mass loss after microwave treatment of the HVC ore.

According to report by Xia and Pickles (1997), being amongst one of the earliest research about microwaves, suggested that Copper (II) Sulphide can be heated to a maximum of 600 °C. Again, according to a reports by Wong D (1975) and WR Tinga (1988), Copper (II) Sulphide falls under the active class being the characteristic by which a material can get heated based on a heating rate of 120 °C/min. Other classes mentioned for other metal oxides based on their heating rates were hyperactive, difficult-to-heat and inactive. Forster (2023) reported on minerals that could be defined as highly microwave-amenable phases (HMAPs). A database based on wt.% HMPAs and the ore dielectric properties was created to predict the bench-scale heating behaviour of any ore based on the wt.% HMAPs. From the database created by Forster (2023), the HVC ore used in this present research falls under class II where most ores heated to (250-300)°C with a maximum heating rate of 133 °C/min. From Table 4-1 a maximum heating rate of 78.75 °C/min with a maximum temperature of 314.2 °C was recorded for the (-3.35 and +1.7) mm size fraction at 200 g at 8 minutes.

With the copper porphyry ore, several minerals contribute to the microwave heating due to the variation in their dielectric properties causing microwave-induced fractures. Variation in the dielectric properties of sulphide minerals (chalcopyrite, pyrite) and the oxide minerals (magnetite and haematite) enables efficient heating and fracture development (Giyani 2023)

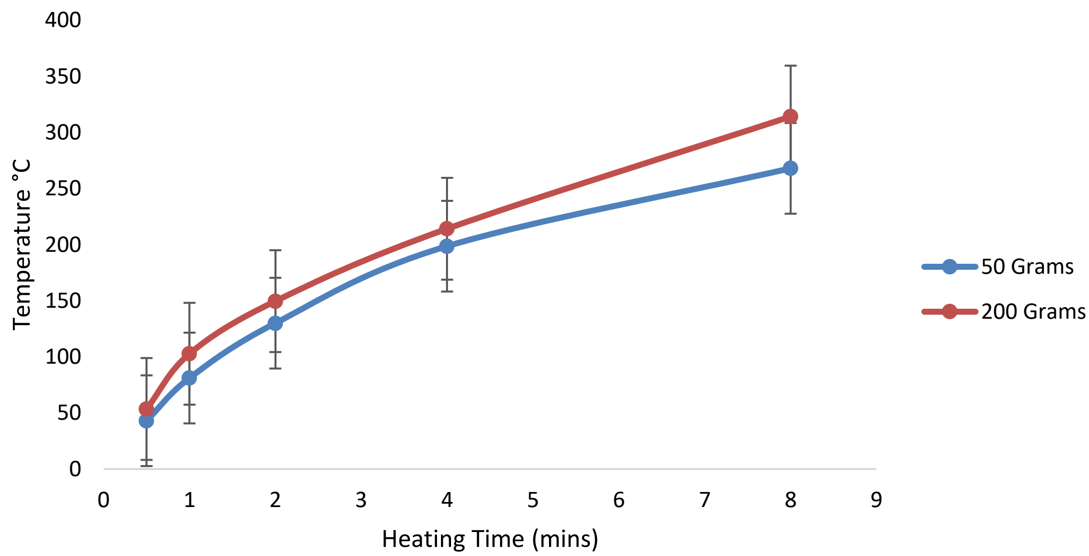


Figure 4-1 Comparison of Microwave heating behaviour curve for (-3.35 and +1.7) mm 50 g and 200 g charge

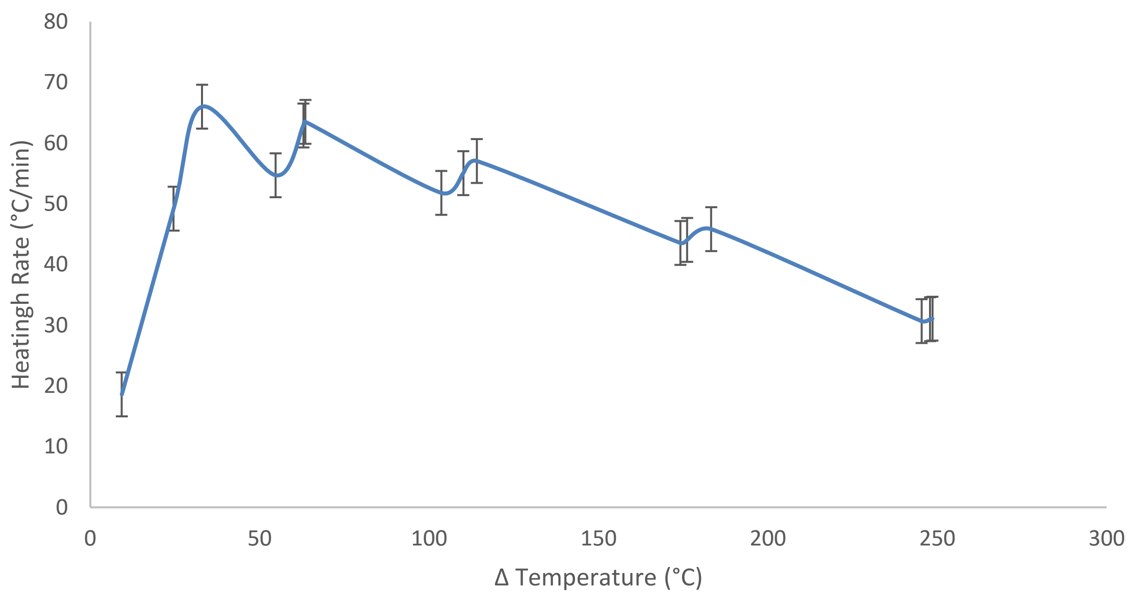


Figure 4-2 Microwave heating rate vs change in temperature for (-3.35 and +1.7) mm 50 g charge

The microwave heating curves and corresponding heating rate curves for the HVC ore (-3.35 and +1.7) mm size fractions which was bench microwave treated at 200 g charge is as shown in Figure 4-3.

Figure 4-3 shows the microwave heating rate curve, which shows the heating rate against the change in temperature through the course of the microwave treatment for 8 minutes. The maximum heating rate was about 78.75 °C/min with a corresponding change in temperature of 157.5 °C. A maximum temperature of 314.2 °C, with a corresponding mass loss of 5.82 % was achieved after 8 minutes of microwave heating time.

Again, the mass loss is highly likely to be bound water loss as opposed to sulphur since there were no sulphur gas emissions or gas perceived during the experiment.

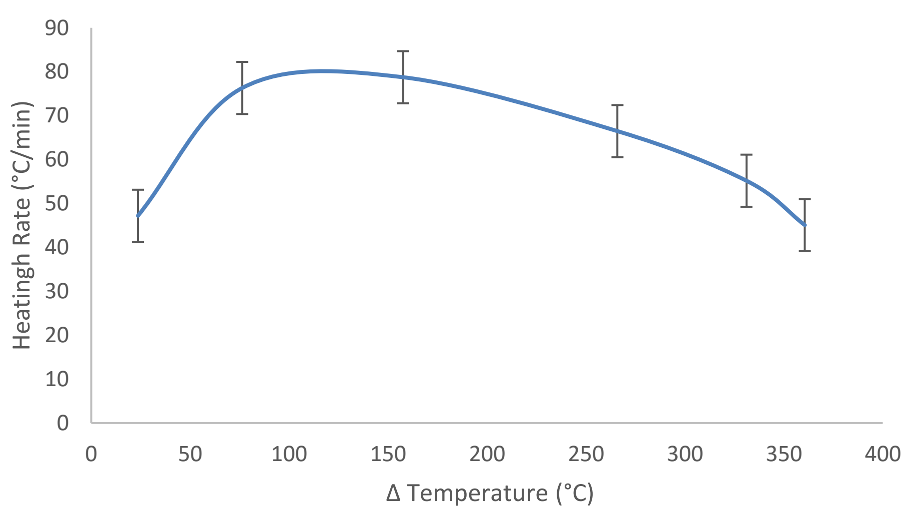


Figure 4-3 Microwave heating rate vs change in temperature for (-3.35 and +1.7) mm 200 g charge

4.1.1.1 Comparing the Results from the Varying Mass used to Treat (-3.35 And +1.7) mm Size Fraction at the Bench Scale

The comparison from Table 4-1 clearly depicts that, both the maximum heating rate with its corresponding change in temperature alongside their respective maximum temperatures and mass loss increased with sample mass. This highlights the impact the mass of the sample has on the heating behaviour during microwave treatment.

Table 4-1 Comparison of the results from the varying mass used to treat (-3.35 and +1.7) mm size fraction

Bench microwave treatment for (-3.35 and +1.7) mm size fraction at 8 minutes residence time					
50 g of sample			**200 g of sample**		
Max. heating rate °C/min	**Maximum temp. °C**	**Mass loss (%)**	**Max. heating rate °C/min**	**Maximum temp. °C**	**Mass loss (%)**
66	268.1	2.3	78.75	314.2	5.82

The microwave heating curves and corresponding heating rate curves for the HVC ore (-6.7 and +4.75) mm 200 g charge size fractions which was bench microwave treated at 200 g charge is as shown in Figure 4-4 and Figure 4-5 respectively. Figure 4-4 shows the effect of heating time on the 200 g sample's temperature. Figure 4-5 shows the microwave heating rate curve, which shows the heating rate against the change in temperature through the course of the microwave treatment for 8 minutes. The maximum heating rate was about 89.3 °C/min. at a corresponding change in temperature of 89.3 °C for the maximum heating rate. A maximum temperature of 393.3 °C, with a corresponding mass loss of 2.61 % was achieved after 8 minutes of microwave heating time.

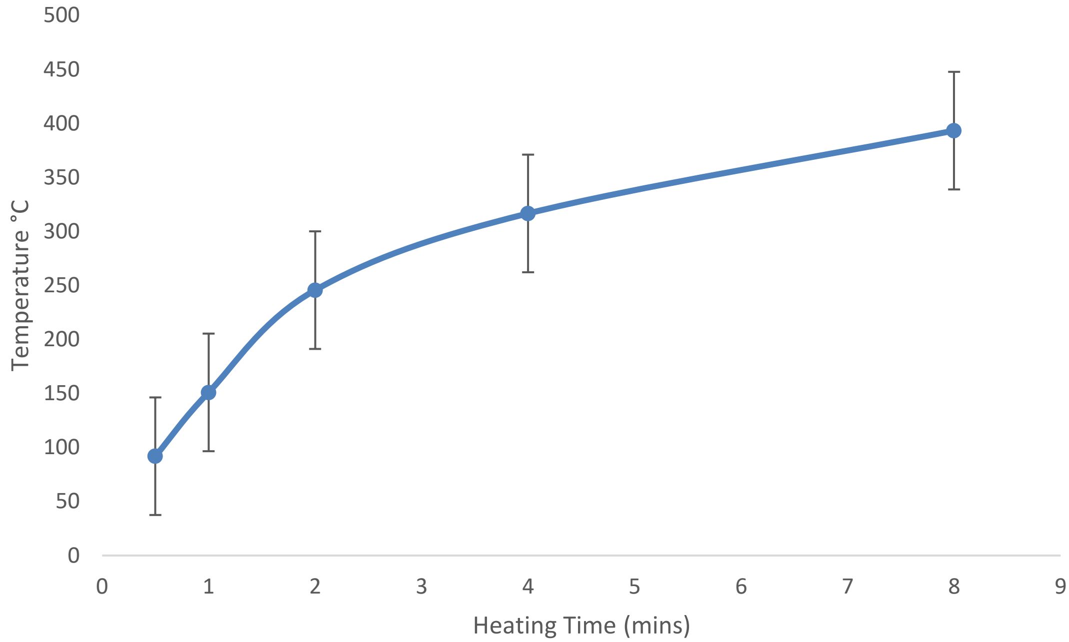


Figure 4-4 Microwave Heating Behaviour Curve for (-6.7 and +4.75) mm 200 g charge

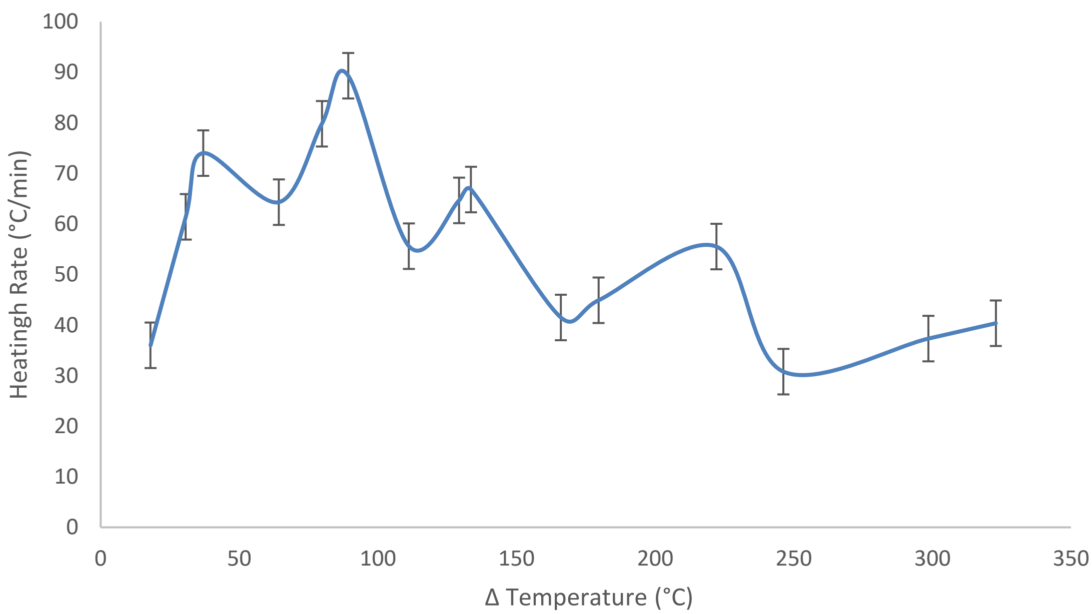


Figure 4-5 Microwave Heating Rate vs Temperature for (-6.7 and +4.75) mm 200 g charge

4.1.2 Arcing And Discharges During Pre-Treatment Methods

While conducting microwave heating at the bench-scale for the individual coarser rocks (-63.5 to +50.8) mm, noticeable discharging effects manifested, taking the form of glows, sparks, and audible popping sounds were observed as illustrated in Figure 4-6 The observed fracture or breakage of the copper porphyry ore during the treatment can be attributed to these discharges and the associated heating effects. These persistent arcing and discharges can be attributed to sulphide veins in the ore being very conductive. Highly conductive regions, also have regions which have very high electric field strength, also there is ineffective microwave penetration as such microwaves are reflected at the surface which may lead to arcing and the production of these discharges (Genn and Morrison 2014; Emerson 2022). During treatment at the pilot scale arcs were only observed for the highest energy dose which was 10 kWh/t.

Figure 4-7 and Figure 4-8 show the transformations of rocks 27 and 36 respectively. Some other rocks equally exhibited these changes. In Figure 4-7 large fractures were observed with a highest temperature of 392 °C recorded by the FLIR handheld camera. In Figure 4-8 large fractures were observed with a molten part of the rock with a highest temperature of 393 °C recorded. This observation supports the reports by Goldbaum et al. (2020) where there was melting of sulphide phases and a change in the structure of rocks due to a significant decrease in the permittivity of pyrrhotite.

Microwave oven during treatment | Bright glow discharge which lasted 20s | Sharp sparks with pop sounds for at 10 s

Figure 4-6 Pictures showing the arcing and discharges experienced during bench scale microwave treatment.

Rock 27 before treatment | Molten Rock 27 after treatment | FLIR image of Rock 27 after treatment

Figure 4-7 Showing the transformation of rock 27 before and after bench-scale microwave treatment.

Rock 36 before treatment | Molten Rock 36 after treatment | FLIR image of Rock 36 after treatment

Figure 4-8 Showing the transformation of rock 36 before and after bench-scale microwave treatment.

4.1.3 Microwave Pre-Treatment of Ore (Continuous Pilot-scale)

All five (5) treatment cycles were fully completed. During the treatments, arcing (3 arcs) was observed for treatment cycle 4 which was the cycle with the highest electrical energy dose (10 kWh/t) applied. The heating was not uniform as each treatment cycle with same conditions had different hotspots detected from the tray. Completion of all the treatment cycles of the ore without the system stopping because of arcs supports the work by Forster (2023) which reported that the most suitable ores for pilot-scale treatment should not contain many reflective phases and as such copper porphyry ore can be processed before arcing occurs.

4.2 The Effects of Microwave-Treatment on Particle Size Distribution

In Table 4-2 P_{80} for 200 µm is recorded for both the microwave treated and reference for the (-3.35 and +1.7) mm size range samples. After 10, 15 and 20 minutes of grinding time, the P50s and P_{80s} for the reference material was lower than that of the MWT material. A report by Javad Koleini et al. (2012) suggested that, higher P_{50} and P_{80} values depicts coarser mineral particle size, implying the mineral particles are closer in size with the rock or ore material being grinded which in turn slows down the rate of breakdown during grinding. Generally, as reported by (Amankwah et al. 2005; Kingman et al. 2004; Adewuyi et al. 2020) and others, after microwave pretreatment, the grindability of the ore is improved. However, a report by (Olmsted 2021; Forster 2023) saw liberation benefits without an improvement in ore grindability as can be seen in Figure 4-9. It should however be noted that, the percentage solids for the jar mill tests was 50%. P_{80} for 200 µm for MWT is achieved at a time grind of 23.80 minutes, while the P_{80} for 200 µm for reference is achieved at a time grind of 23.28 minutes, slightly lower.

Table 4-2 Comparison Between Particle Size Analysis Data of HVC ore sample (-3.35 and +1.7) mm reference & bench MWT Samples

Samples	Grinding Time (Minutes)	% Passing Size	Microns (µm)
MWT	0	P_{80}	2913.9
	10		1008.2
	15		416.0
	20		293.2
Ref	0		2893.0
	10		640.8
	15		393.3
	20		276.5

Figure 4-9 shows particle size distribution for P_{80} for 200 µm for both the microwave treated and reference for the (-3.35 and +1.7) mm size range samples. It was observed that there was a significant difference between the PSD for the MWT and the reference samples. The MWT samples had poorer PSD compared to the reference samples. This can be attributed to the fact that the finer particle size (-3.35 and +1.7) mm relative to the coarser particle size (-6.7 and +4.75) mm has a larger surface area and as such have poorer heat dissipation and absorption taking into account the penetration depth as well. Once the penetration depth is greater than the particle sizes of the material, microwave irradiation is inefficient. Thus, the benefits of microwave irradiation is then impeded (Horikoshi et al. 2018)

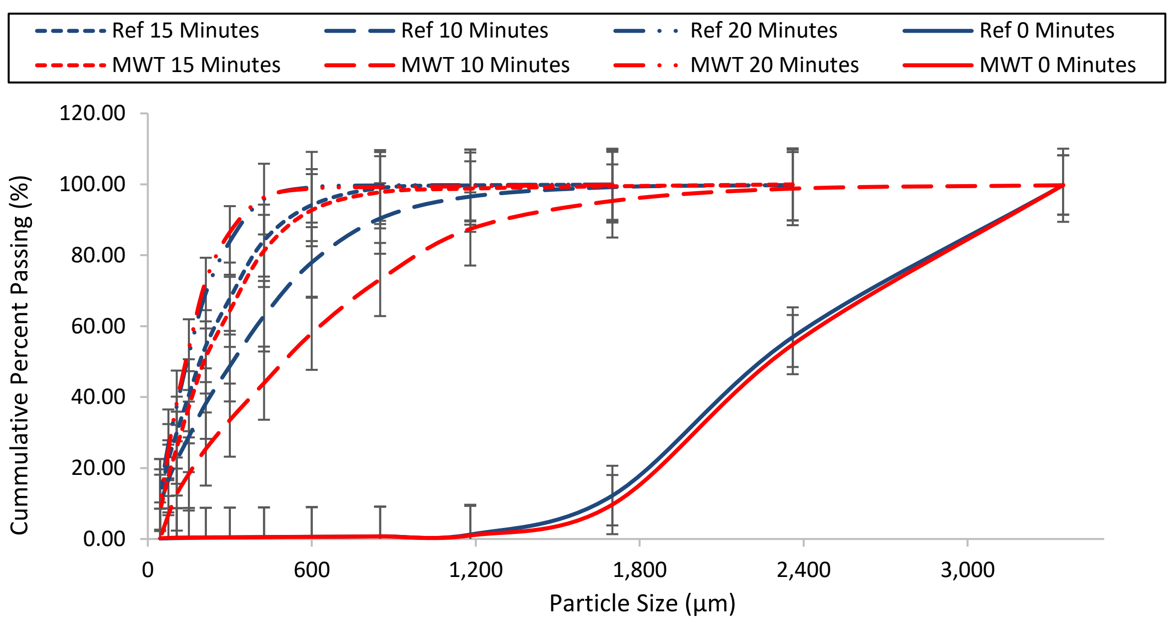


Figure 4-9 A graph showing the PSD comparisons between the MWT and reference material for the (-3.35 and +1.7) mm using a ball jar mill.

Table 4-3 P_{80} for 200 µm is recorded for both the microwave treated and reference for the (-6.7 and +4.75) mm size range samples. After 15, 20 and 25 minutes of grinding time, the P_{50s} and P_{80s} for the microwave treated material was lower than that of the reference material. Comparing the effects of microwave treatment on the (-3.35 and +1.7) mm (-6.7 and +4.75) mm size range samples, it can be observed that the larger particles responded better to microwave treatment than smaller ones. The larger particles responding better to microwave treatment than smaller ones aligns with recent studies in the field reported by Olmsted (2021). P_{80} for 200 µm for MWT is achieved at a time grind of 26.80 minutes while the P_{80} for 200 µm for reference is achieved at a time grind of 26.77 minutes. Though it can be observed that the time taken to achieve P_{80} between both MWT and reference is quite negligible.

Table 4-3 Comparison Between Particle Size Analysis Data of HVC ore sample (-6.7 and +4.75) mm reference & bench MWT Samples

Samples	Grinding Time (Minutes)	% Passing Size	Microns (µm)
MWT	0		6257.3
	15		459.5
	20		295.7
	25		225.7
		P_{80}	
Ref	0		6269.1
	15		581.2
	20		328.4
	25		233.6

Figure 4-10 shows particle size distribution for P_{80} for 200 µm for both the microwave treated and reference material for the coarser particle size fraction (-6.7 and +4.75) mm, it was observed that, the P_{80s} for the MWT material was lower than that of the reference material but with increased mill residence time and lower size particles, the reference material tends to have lower P_{80s} just as earlier observed. Microwave treated samples had better grinding benefits at coarser sizes (-6.7 and +4.75) mm have a smaller surface area relative to finer particle sizes and as such have better heat dissipation and absorption taking into account the penetration depth as well. Once the penetration depth matches the particle sizes of the material, microwave irradiation is better. Thus, the benefits of microwave irradiation is then realized (Horikoshi et al. 2018).

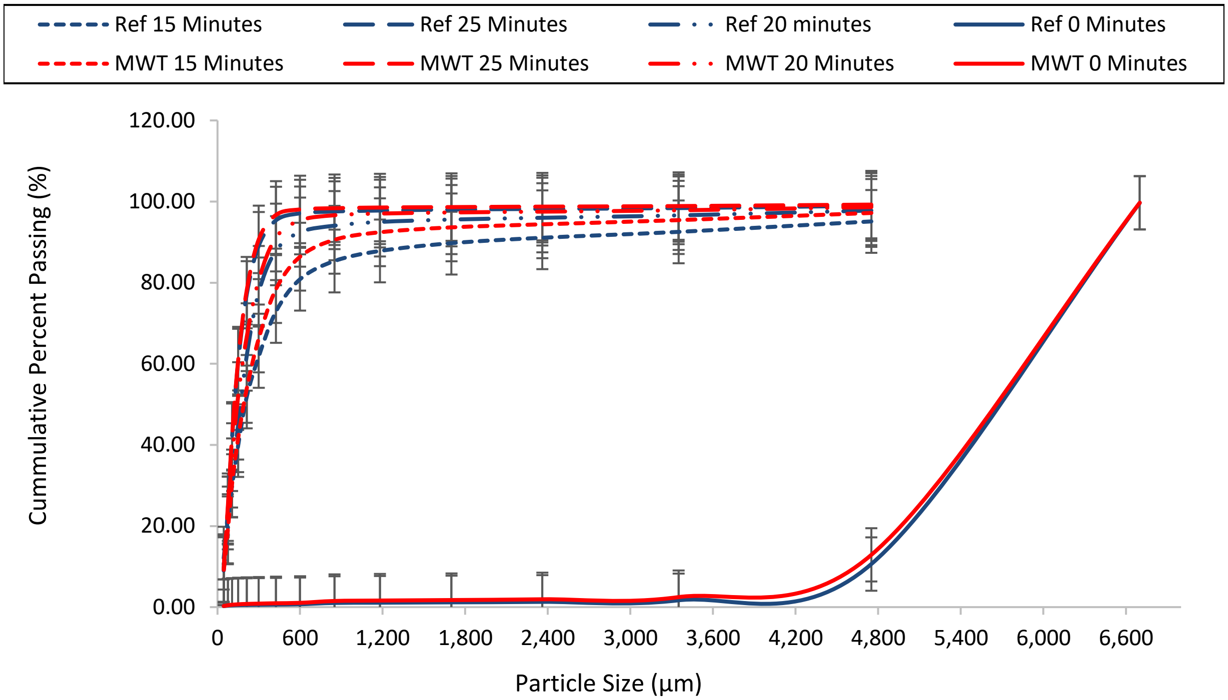


Figure 4-10 A graph showing the PSD comparisons between the MWT and Reference material for the coarser size fraction (-6.7 and +4.75) mm using a ball jar mill.

By comparing both the MWT and reference material for both the (-3.35 and +1.7) mm and (-6.7 and +4.75) mm size fractions as can be seen from the graph in Figure 4-11 it gives a clear and better understanding of the effects of microwave treatment at varying size fractions with the same mill residence time. It can be observed that the larger particle sizes had better particle size distribution and analysis relative to the smaller particle size range. This suggests that MWT seems to have better effects when there are larger particles relative to the smaller particle size range. This may be as a result of the larger particles having dimensions that are closer to the penetration depth of the microwave radiation. Due to this, microwave energy can penetrate into larger particles leading to efficient and better outcomes during microwave heating (Horikoshi et al. 2018).

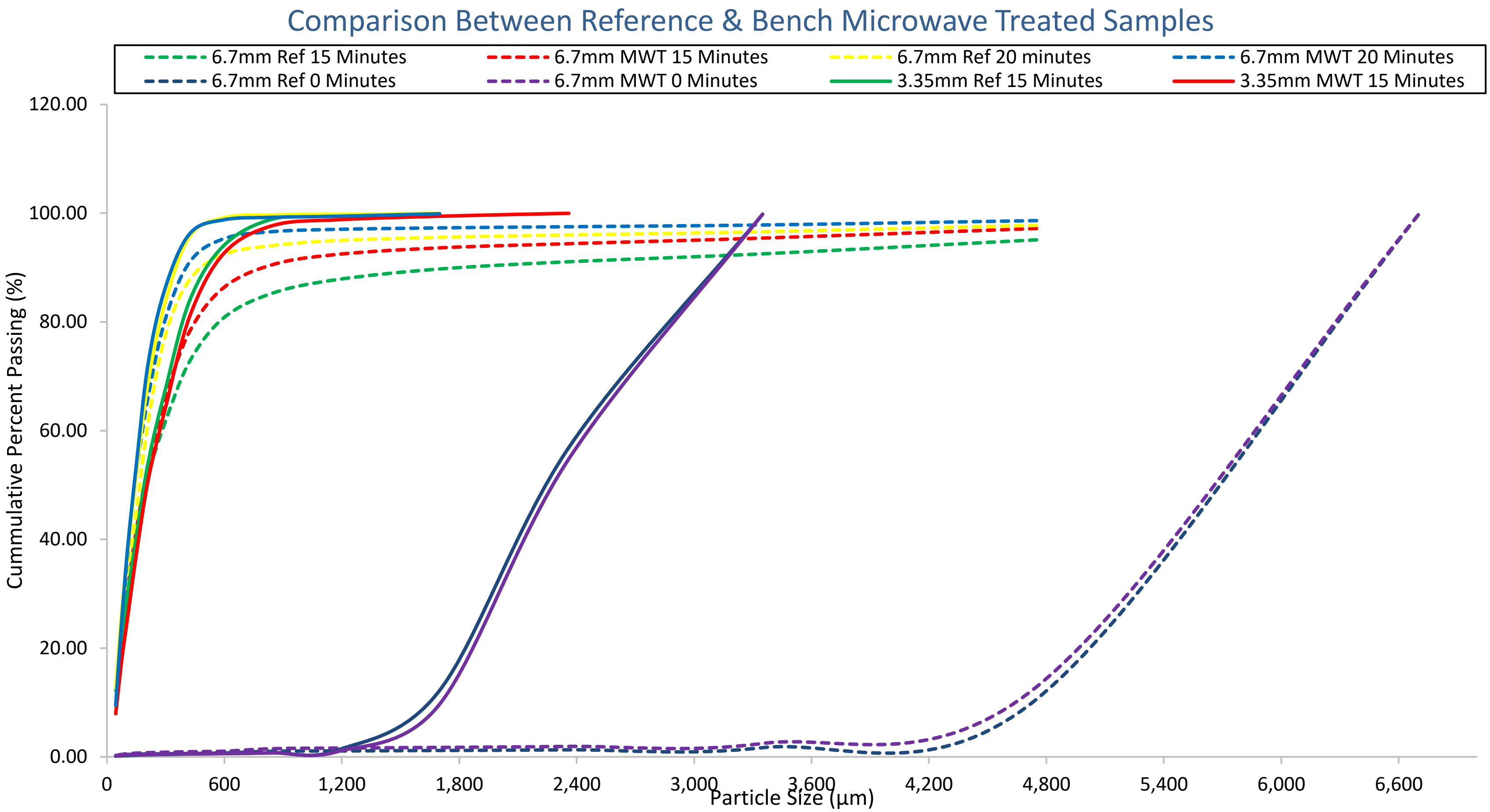


Figure 4-11 A graph showing the comparison between reference & bench microwave treated samples for both finer and coarser size fractions.

4.3 Evaluating the Degree of Mineral Liberation Achieved

Mineral liberation is mostly ensured by comminution. With conventional methods of comminution, there is the possibility of overgrinding as most minerals of interests are linked together with other elements or minerals in the ore body. This situation of overgrinding results in the production of slime and inefficient recovery in the same vein expends excess energy thus not economically viable (Amankwah et al. 2005). Mineral liberation can be enhanced by microwave pretreatment as microfractures are introduced which will make breakage with lower impact or stress a possibility (Scott et al. 2008). After microwave treatment the ore was crushed to -2 mm before introducing it into the rod mill, at a feed charge of 1000g and 65% solids. A P_{80} of 200 µm is what is used/introduced at the mother company's flotation circuit. This research aims to determine whether microwave treated samples of same size fraction or coarser fractions (200 µm and 250 µm) may have significant enhanced liberation benefits. QEMSCAN analysis was done after the batch flotation tests. Table 4-4 shows the results produced after the analysis.

In Table 4-4 there is distribution of particles into grade classes ranging from the barren class (0-30%) and fully liberated (100% value). As can be seen in the Table 4-4, there was better liberation observed for the microwave treated material between the 95 % to 100 % liberation mass range. MWT had better liberation at 250 µm relative to its reference material. This again supports the report by (Olmsted 2021; Forster 2023) and (Horikoshi et al. 2018) where larger particle sizes had better microwave treatment outcomes relative to their smaller counterparts and reference samples.

Cumulatively, from 0 % to 50 % liberation, the untreated material had 78.03 % locked up valuable minerals or gangue material as opposed to 31.89 % for the microwave treated material which was relatively lower than the former.

Again, even though the average particle size for the microwave treated material was coarser than that of the untreated, cumulatively from $\leq$ 80% to 100% liberation saw a 68.10 % liberated material as opposed to 21.97 % liberated material for the untreated material.

Table 4-4 Table showing the extent of liberation of copper sulphide minerals for treated and reference material after batch froth flotation.

	Average Particle Size (µm)	117	94
	Liberation (mass %)	**Microwave Treated**	**Reference Material**
Copper Sulphide	≤ 30%	31.68	77.88
	≤ 50%	0.21	0.15
	≤ 80%	1.13	7.67
	≤ 95%	0.15	1.81
	< 100%	56.28	8.43
	100%	10.54	4.06
	Total	100.00	100.00

Figure 4-12 and Figure 4-13 are false colour maps showing the extent of liberation illustrating the distribution of mineral phases and their liberation for both the microwave treated and the reference samples respectively. It can be observed that copper sulphides were significantly liberated in the microwave treated samples relative to the reference samples where most of the copper sulphides were locked in the gangue material. The improved liberation observed by the microwave treated samples supports the work by Forster (2023).

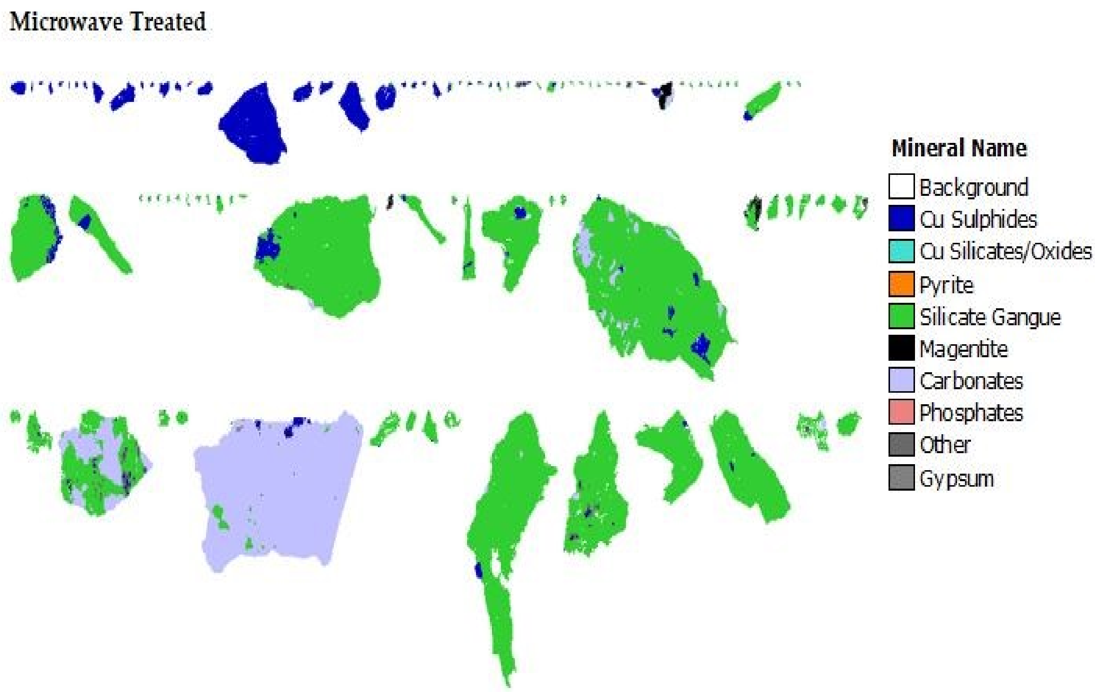


Figure 4-12 Graph illustrating the distribution of mineral phases and their liberation for microwave treated material.

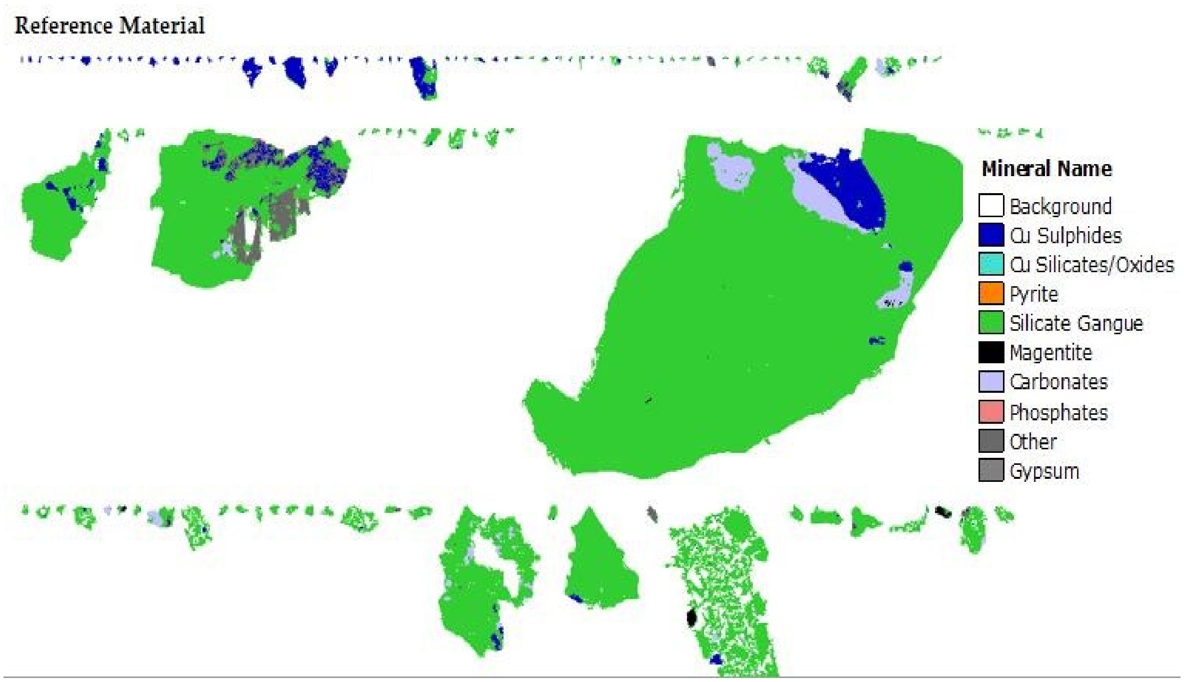


Figure 4-13 Graph illustrating the distribution of mineral phases and their liberation for untreated material.

4.3.1 Exploring the Influence of Microwave Treatment after Batch Flotation

The efficacy of various separation processes can be influenced by a myriad of factors. In the case of flotation, considerations extend beyond the process itself to encompass elements impacting particle surfaces. These encompass phenomena like particle oxidation, surface coatings induced by fine gangue particles, or complications arising in the operational aspects of the equipment. The rate of flotation demonstrates a strong correlation with particle size, as noted by Trahar (1981)and Martin et al. (1991). Ore liberation, a consequential outcome of size, manifests as finer particles being more liberated, whereas coarser particles exhibit reduced liberation. Consequently, flotation performance is contingent not only on size and liberation but is a multifaceted interplay of several factors demanding consideration. These factors include, but are not limited to, the presence of excessive ore slurry flotation reagents, aeration and mixing conditions, pH levels of the slurry, flotation conditions, and properties of the ore itself. Figure 4-14 and Figure 4-15 are graphs showing the recovery of the ore for 200 µm and 250 µm respectively. The head grade of the copper porphyry ore used is 0.15 g/t (gram per tonne).

Figure 4-14 shows there was a sharp recovery after 1 min, for MWT Mo which later dropped at 2 mins with a steady rise in recovery till the float reaches a plateau after five minutes. In this case five concentrates were collected at half minute, a minute, and three minutes intervals up to a total of eight minutes. Molybdenite (Mo) for the microwave treated material had better recovery with respect to time relative to the untreated material for Molybdenite. The microwave treatment process is likely to have facilitated the breakage of molybdenite-pyrite or molybdenite-quartz associations, promoting the exposure of molybdenite particles for flotation recovery. Molybdenite and copper sulphide minerals have different crystal structures and as such may have responded differently per the present parameters used for the microwave treatment of the ore and subsequently the flotation process.

At 5 minutes, approximately 51 % recovery had been achieved while the untreated material had approximately 42 % recovery.

Copper (Cu) for both the MWT and reference material had a steady rise in recovery till the float reaches a plateau after five minutes. At 2 minutes through to 8 minutes, there was a steady rise and plateau of Cu recovery for both treated and untreated sample. There was no significant difference in recovery realized.

Figure 4-15 shows recovery was rapid, and the float reaches a plateau after just five minutes. In this case five concentrates were collected at half minute, a minute, and three minutes intervals up to a total of eight minutes. Molybdenite (Mo) for the microwave treated material had better recovery with respect to time relative to the untreated material for Molybdenite. At 5 minutes, approximately 60 % recovery had been achieved while the untreated material had approximately 30 % recovery. Copper (Cu) for the microwave treated material had poor recovery for the first two minutes relative to the untreated material for copper. After 1 min , approximately 57 % recovery had been achieved for the untreated while the microwave treated material had approximately 48 % recovery. At 2 minutes through to 8 minutes, there was a steady rise and plateau of Cu recovery for both treated and untreated sample. There was no significant difference in recovery realized.

Comparing the recovery for both 250 µm and 200 µm respectively, Molybdenite (Mo) had better recovery for both relative to Cu. However, the 250 µm showed better recovery of Mo than 200 µm.

Table 4-5 shows the legend used for the Recovery-Time and Recovery-Grade graphs.

Table 4-5 Legend used for Recovery-Time and Recovery-Grade Graphs

Abbreviation	Depiction
Cum Rec Cu-HVC-1M	Cumulative recovery of copper for MWT sample for 200 µm
Cum Rec Cu-HVC-1R	Cumulative recovery of copper for reference sample for 200 µm
Cum Rec Cu-HVC-2M	Cumulative recovery of copper for MWT sample for 250 µm
Cum Rec Cu-HVC-2R	Cumulative recovery of copper for reference sample for 250 µm
Cum Rec Mo-HVC-1M	Cumulative recovery of molybdenite for MWT sample for 200 µm
Cum Rec Mo-HVC-1R	Cumulative recovery of molybdenite for reference sample for 200 µm
Cum Rec Mo-HVC-2M	Cumulative recovery of molybdenite for MWT sample for 250 µm
Cum Rec Mo-HVC-2R	Cumulative recovery of molybdenite for reference sample for 250 µm

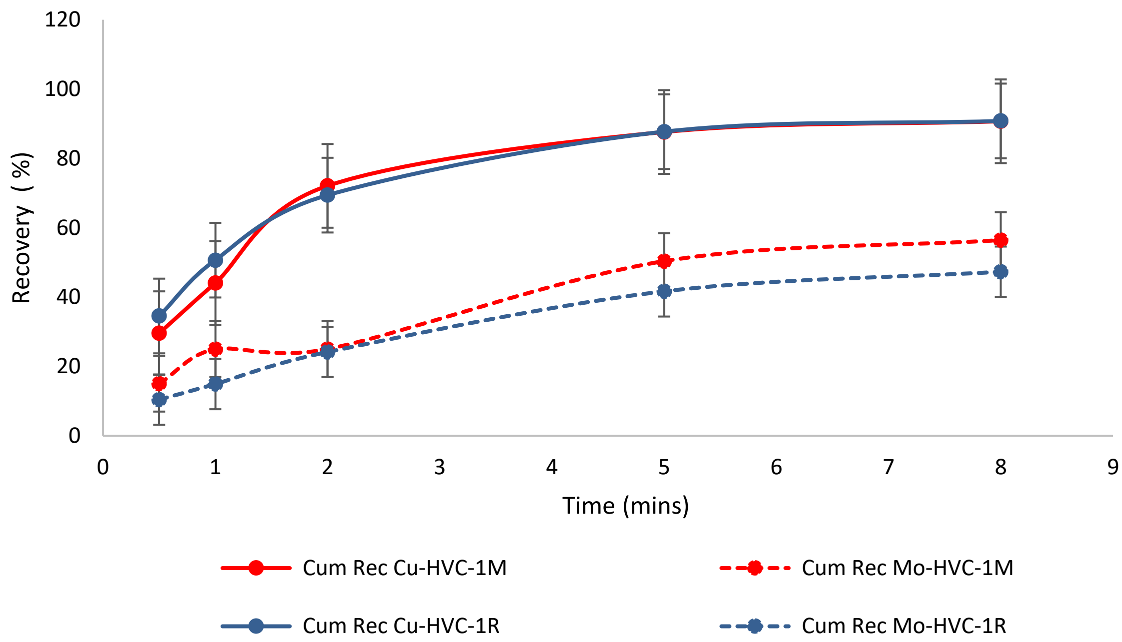


Figure 4-14 Recovery vs time curves comparison between HVC-1R and HVC-1M (200 µm)

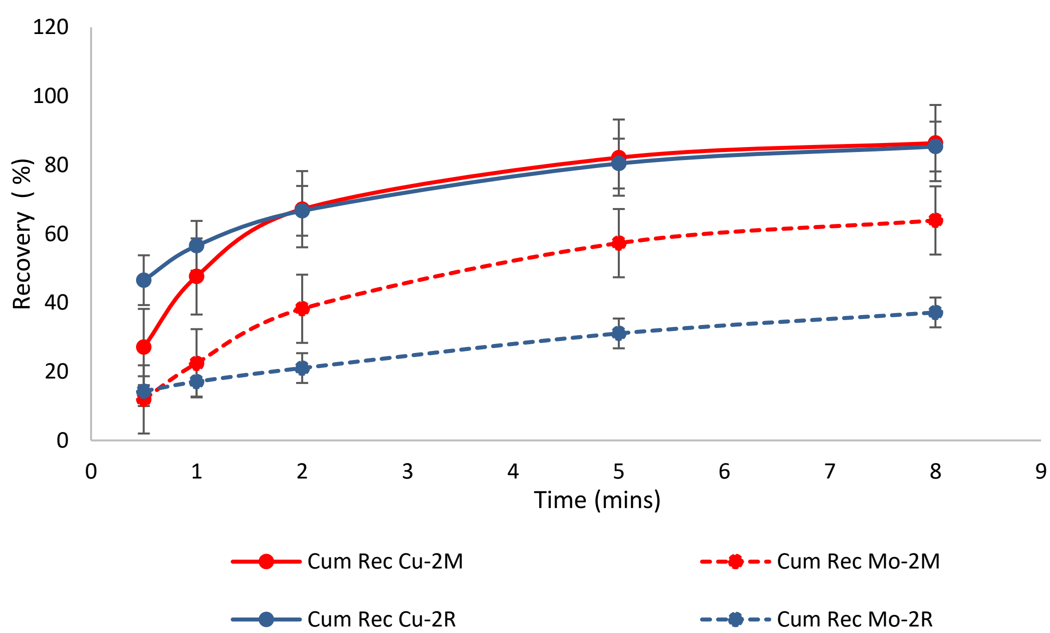


Figure 4-15 Recovery vs time curves comparison between HVC-2R and HVC-2M (250 µm)

Flotation performance is typically shown through grade-recovery curves, where an upward and rightward shift signifies enhanced performance. The method hinges on the premise that both concentrate grade and mass pull are determined by the cell's contents and operating conditions (Neethling and Cilliers 2012).

However, from Figure 4-16 Cu for the microwave treated material had poor flotation performance relative to the untreated material for copper at 200 µm. At 10 % grade the untreated material had 70 % recovery while the treated material had approximately 50 % recovery. Also from Figure 4-18 for 250 µm at 10 % grade the untreated material had 67 % recovery while the treated material had approximately 48 % recovery This outcome contradict reports from (Batchelor et al. 2016b; Sahyoun et al. 2005; Gholami et al. 2021) as they reported a significant improvement in the recovery of copper after microwave treatment. However, in this present study, such benefits were not observed. Carrying out other tests with same particle size conditions while optimizing the amount of reagents added during flotation may help in this situation. This is because the by-product, molybdenite had significant recovery as opposed to copper.

From Figure 4-17 molybdenite (Mo) for the microwave treated material had better flotation performance relative to the untreated or reference material for molybdenite at 200 µm. At 0.1 % grade the treated material had 60 % recovery while the untreated material had approximately 10 % recovery. From Figure 4-19 molybdenite (Mo) for the microwave treated material had better flotation performance relative to the untreated or reference material for molybdenite at 250 µm. At 0.1 % grade the treated material had 60 % recovery while the untreated material had approximately 15 % recovery.

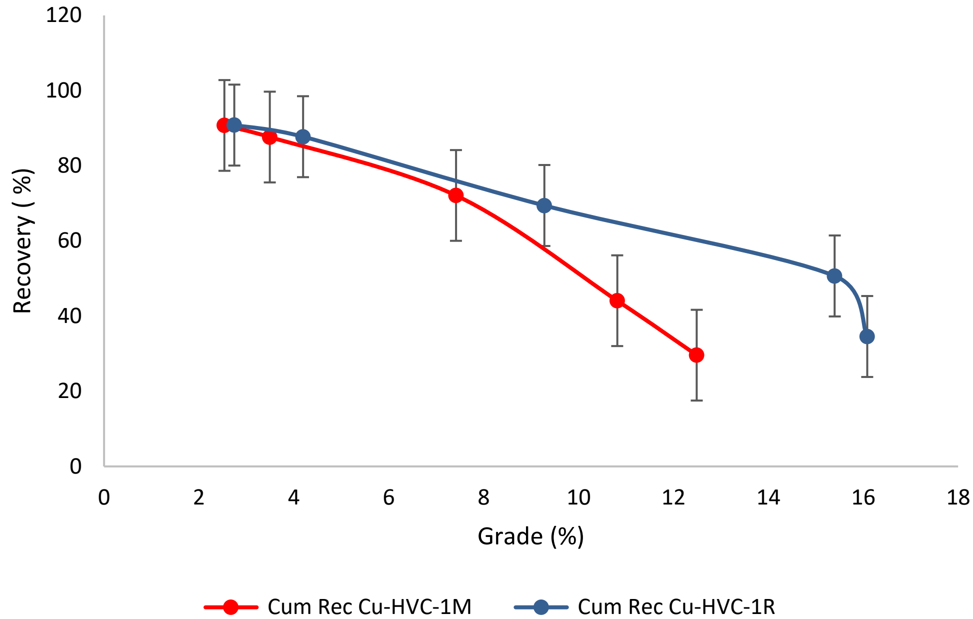


Figure 4-16 Recovery vs grade graph for Cu comparison between HVC-1R and HVC-1M (200 µm)

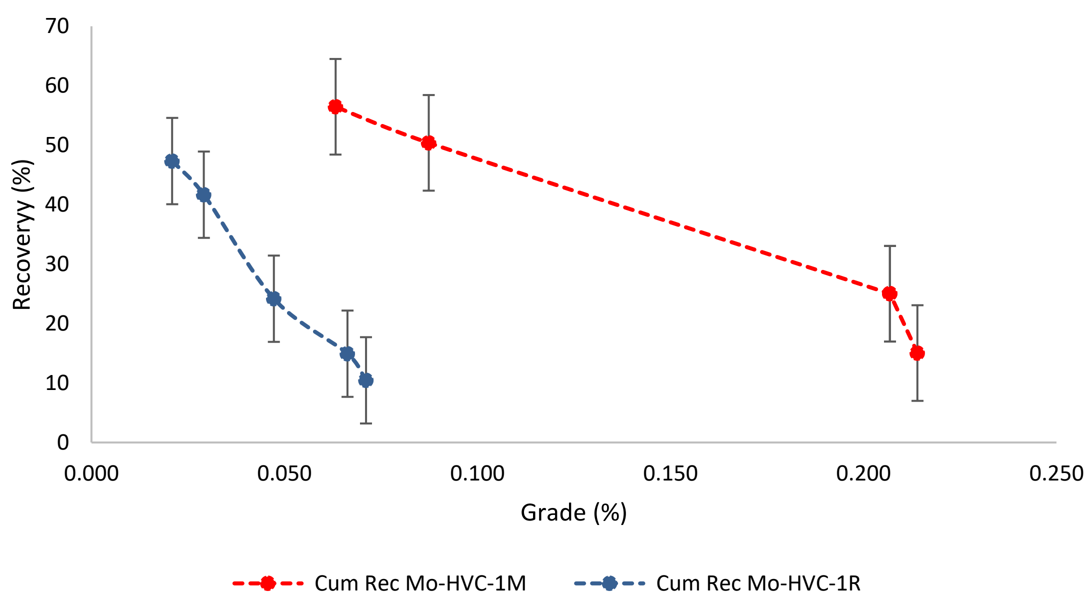


Figure 4-17 Recovery vs grade graph for Mo comparison between HVC-1R and HVC-1M (200 µm)

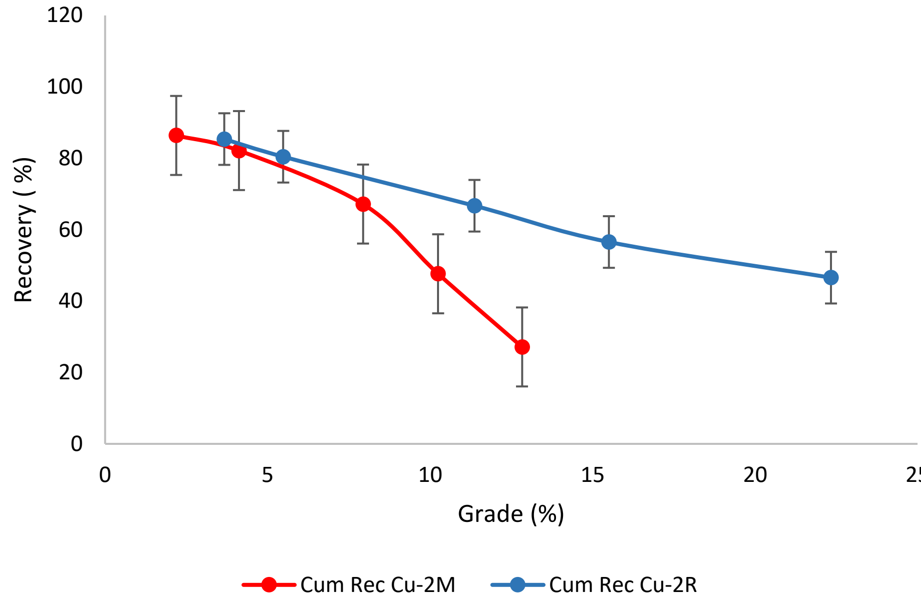


Figure 4-18 Recovery vs grade graph for Cu comparison between HVC-2R and HVC-2M (250 µm)

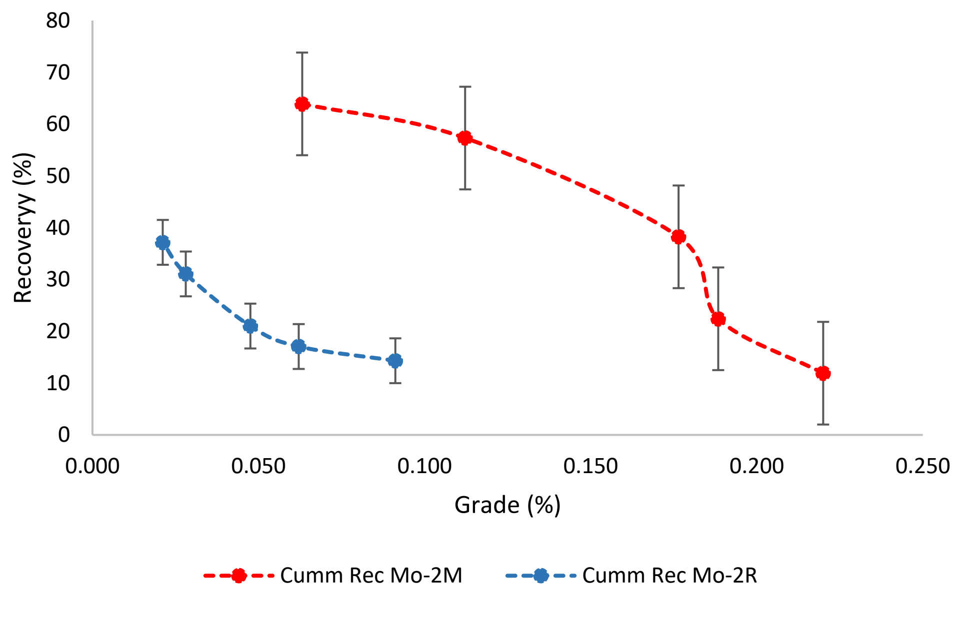


Figure 4-19 Recovery vs grade graph for (Mo) comparison between HVC-2R and HVC-2M (250 µm)

Chapter Five

5 Conclusions And Recommendations

This study has investigated the intricate relationship between microwave assisted comminution and the characterisation of energy efficiencies using microwave treated and untreated copper porphyry ore. The series of experiments and analysis in this project were aimed at quantitatively assessing the particle size distribution, mineral liberation improvements, and recovery after flotation, achieved through microwave treatment of HVC ore. In this conclusion section, there will be a summary of the main findings of the study, a discussion of the impacts made and suggestions for future research in this area.

5.1 Overview of Findings

There has been the explanation of interaction between mineral liberation and particle size by demonstrating the recovery behaviours of chalcopyrite-bearing particles of varied sizes with similar liberation characteristics. It was observed that larger particle sizes responded better or had better effects of microwave treatment relative to the smaller particle sizes. This observation is in agreement with the report by Javad Koleini et al.0 (2012) and Batchelor et al. (2016b). This was observed from the QEMSCAN analysis done on the 250 µm particle size range after milling. Even though the microwave treated material had coarser size particles, it still had better liberation than the finer untreated material. Also, coarser size range of the MWT samples had better PSD relative to the reference material.

It is however important to note that the effectiveness of microwave pre-treatment is dependent on factors such as the type of the ore, the mineral constituents and processing parameters employed.

Although there would have been an expectation of better recovery for copper in flotation, this was not the case in the experiments in this work. The relationship between recovery, grade, and residence flotation time showed favourable outcomes for microwave-treated material in terms of molybdenite, but not for copper.

5.1.1 Challenges or Limitations

During microwave treatment of the material, arcing incidents were particularly prevalent, notably during the individual microwave treatment of the rocks at the bench-scale and the microwave treatment of the coarser rocks at continuous pilot stage. The heat produced by these discharges has the potential to create localized hot spots and, in certain instances, cause the melting of metal during the microwave irradiation process. Ensuring the effectiveness of the microwave treatment process necessitates a broad understanding of quite a number of factors. These include determining the percentage of introduced power that efficiently absorbed by the rocks, permeability and permittivity, penetration depth and energy dosage. Additionally, understanding the ore's morphology and type is crucial, given their major influence on dielectric properties and, consequently, the ore's response to treatment. This is particularly relevant since most ores encompass diverse metals alongside the desired mineral of interest.

As these advancements are observed, it is imperative to address the challenges encountered during the microwave treatment process. The prevalence of arcing incidents introduces a critical consideration for the scalability and practical implementation of microwave-assisted techniques. The findings resonate with the cautionary notes from (Lin et al. 2023), emphasizing the need for a better understanding of various factors influencing the process, including penetration depth, permittivity, and ore morphology.

In conclusion, this study acts as a continuation of the work from others, with downstream benefits realized after bench-scale microwave treatment of copper porphyry ore. The prospect of microwave treatment of ores is very promising, researchers are urged to dive deeper into the diverse world of microwave-assisted comminution. As the complexities are untangled, collective efforts drive the industry toward innovative and sustainable practices, ensuring responsible extraction and use of earth's resources for posterity. This study has not only uncovered the intricate connection between microwave-assisted comminution and characterization of energy efficiency in copper porphyry ore material but has also highlighted the complex relationship between mineral liberation and particle size. These insights are crucial for improving strategies in mineral processing, aiming for better recovery and economic gains.

5.2 Recommendations for future work

It is suggested to further investigate and vary chemical parameters to assess if enhanced recovery can be achieved with coarser particle-sized materials, given the observed improvement in liberation after milling. In certain cases, the addition of extra collector might be considered to boost the recovery of coarsely liberated particles; however, this approach could lead to the retrieval of undesired particles, resulting in non-selective recovery and diminished grades. Different reagents scheme and or different conditioning time can be employed for the microwave treated samples. Looking at the relationship between recovery, grade, and residence flotation time, interesting dynamics have been discovered, particularly in molybdenite recovery. Although this study mainly focused on flotation kinetics, it suggests a need to explore chemical parameters further, as Brest et al. (2021) have also suggested. Their work emphasizes the potential impact of adjusting chemical variables on recovery rates, especially for larger particles. This aligns with the discovery of better downstream benefits, offering a promising path for future research. Optimizing the flotation process is critical for effectively separating molybdenite from chalcopyrite and maximizing the recovery of both.

Better liberation and recovery benefits were realized after the bench-scale microwave treatment. Completed and successful cycles of continuous-pilot microwave treatments were also achieved to analyse the microwave heating behaviour of the ore at the pilot-scale. It is recommended that grinding and flotation tests should be conducted after treatment at the pilot-scale to determine the downstream benefits. This is highly recommended as work from Forster (2023) also reported mineral liberation benefits after the pilot-scale treatment of the copper porphyry. As such conducting downstream flotation tests on these ores to ascertain the potential benefits in metal and mineral recovery is recommended.

Horikoshi et al. (2018) found that the optimal sample size can be determined by having a particle size that matches with the penetration depth. For future research, it is recommended to also know both the real and the imaginary permittivity, and the penetration depth which can be analysed to determine the amount microwave energy to be absorbed by the ore and the heating rate of the ore respectively. This can assist in optimizing the microwave heating process and enhance the heating efficiency, at an attempt to reduce of arcing and discharges.

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