A Review of Characterization Techniques and Processing Methods for Lithium Extraction

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Abstract

The on-going drive and demand for green-energy revolution has reflected in attendant increase in the demand for strategic and critical metals and elements such as lithium, tantalum, cobalt, and rare earth elements. Specifically, the projected demands for portable electric devices, plug-in hybrid electric vehicles (PHEVs), hybrid electric vehicles (HEVs) and electric vehicles (EVs) have highlighted the probable increasing demand for lithium-ion batteries (LIBs). The use of lithium in the production of LIBs has earned it a strategic label in most technologically advanced countries. It has been demonstrated in the literature that lithium extraction from differing resources is based on the generic mineral processing and extractive metallurgical techniques and resource type. Over the years, significant number of flowsheets have been proposed through numerous metallurgical testing programmes, with the overall aim of extracting lithium from both primary and secondary resources. Process mineralogy plays a key role in defining the properties of ores, identifying key opportunities, and ascertaining potential challenges associated with the extraction of lithium. To this end, literature has underscored the importance of mineralogical tools such X-ray diffraction, Quantitative Evaluation of Minerals by Scanning Electron Microscopy, automated scanning electron microscopy, and electron probe microanalysis on the characterisation of ores and separation products, which is crucial in selecting unit operations and subsequent process optimisation campaigns. With Ghana and other developing countries continue to discover lithium deposits, this paper seeks to provide examples of processing opportunities and challenges associated with lithium recovery processes determined through process mineralogical studies, with the overall aim of stimulating ideas and bridging existing knowledge gap in developing countries. Overall, the learnings from this review could serve as a source of inspiration to explore different avenues for sustainable lithium recovery from ores and secondary resources.

Keywords: Critical minerals, green energy, Lithium, Lithium-ion batteries, Process Mineralogy, QEMSCAN

1 Introduction

Lithium is a soft, silvery-white to grey alkaline metal with a metallic lustre when fresh and tarnishes to dull silver grey and then black when in air. Lithium is the third element on the periodic table and has numerous physical and chemical properties. It is the lightest of the alkaline metal with an atomic weight of 6.939 and atomic radius of 1.33 Å. Its density is about half of the water density and can float on water even at the point of reaction (Christie and Brathwaite, 2008). The demand for lithium minerals has increased considerably in recent years due to the application of lithium compounds in lithium-ion battery technology, portable electronic gadgets and power storage systems (Feng *et al.*, 1995; Swain, 2017; Tran and Luong, 2015).

Lithium is a key component in green energy storage technologies and is rapidly becoming a metal of crucial importance to both developing and developed countries (Kavanagh *et al.*, 2018). Carbon-based energy system has negative impacts on environment, society and economy (Greim, *et al.*, 2020). It is now admitted that greenhouse gases do not just pollute but hold important responsibility in global warming with terrible consequences. In this age of increasing population growth and increasing energy demand, ongoing climate change and fossil fuel depletion call for alternative, sustainable solutions that deeply depend on renewable energy (RE) (Griem *et al.*, 2020). The recent increased demand for batteries and various projections showed continued increase in its demand to support the achievement of RE-based energy supply (Breyer, *et al.*, 2018; Hummel *et al.*, 2017; Berger *et al.*, 2017).

Lithium is used primarily in batteries, glass and ceramics, with other uses including rocket fuel and lasers. Lithium finds significant importance in metallurgy as a flux in welding or soldering to primarily reduce energy costs. Specifically, it reduces the melt viscosity and improves flow rates in continuous steel casting (Martin *et al.*, 2017). Lithium compounds have been extensively used in medicine for the treatment of bipolar disorder and to alleviate suicidal thoughts in patients (Oruch, 2014).

One of the important applications of lithium includes the manufacture of batteries. Lithium batteries have widely been used in portable electronic instruments and more importantly, lithium-ion batteries (LIBs) are used to power the next generation of electric vehicles (EVs) with the aim to be environmentally friendly (Dunn *et al.*, 2011). LIB production is the largest consumer of lithium resources today. Lithium batteries are classified as primary and secondary batteries. Primary lithium batteries contain a solid lithium metal with compounds like manganese dioxide and sulphur dioxide as the cathode and are not rechargeable (Lisbona and Snee, 2011; Talens Peiró *et al.*, 2013)

It is worth noting that the global lithium battery market is projected to grow substantially in coming years, from US $ 41.1 billion in 2021 to about US $116 billion by 2030 (Research and Markets, 2021). The electric vehicle market will propel the growth of the lithium market as the number of hybrid and electric vehicles powered by rechargeable lithium batteries picks up (Jetin, 2020; Yu, 2021). It has been forecasted that the top producers of lithium battery cells based on production capacity will be CATL, LG Chem, and Tesla. It is expected that Germany, China, Japan, and France will be leading electric vehicle producing countries (Frieske *et al.*, 2013).

2 Lithium Mineral Resources

2.1 Global Distribution of Resources

Typically, lithium occurs only in compounds as a result of its high reactivity. Figure 1 shows the distribution of lithium reserves worldwide in 2021, by country. The data presented by U.S. Geological U.S.G.S. (2022) indicate that Chile has the largest global lithium reserves by significant margin accounting for about 41% of the global lithium reserve, followed by Australia, with about 25%.

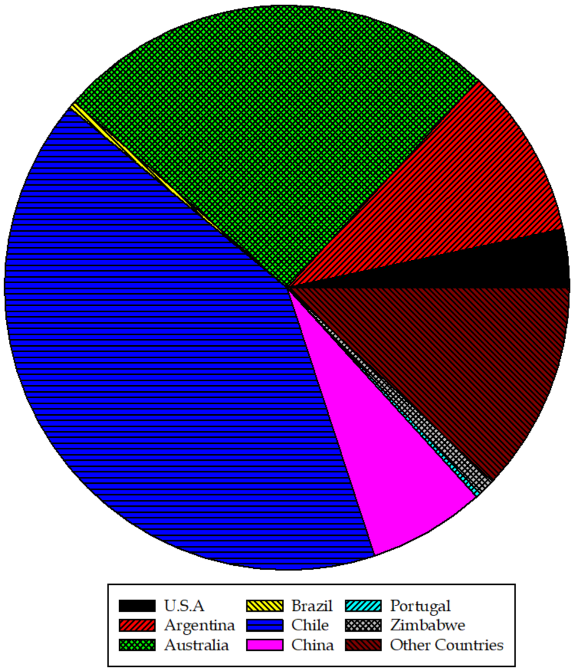

Fig. 1 Distribution of lithium reserves worldwide in 2021, by country.

2.2 Global Lithium Production

Fig. 2 shows lithium mine production from 2010 to 2021. The data suggests significant increase in lithium production from 28,100 metric tons in 2010 to 100,000 metric tons in 2021 (Garside, 2022). The increase in lithium production may be attributed to the increased battery demand for electric vehicles. It is projected that global lithium demand will increase to about 2 million metric tons by 2030 (Garside, 2022). In terms of production, Australia was reported as the highest, producing just a little over 50% of the global lithium products in 2021, with Chile being the second largest producer (Fig. 3).

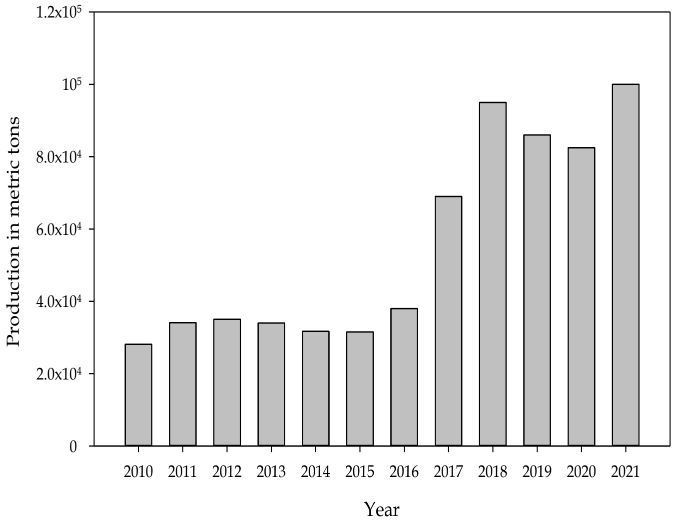

Fig. 2 Lithium mine production worldwide from 2010 to 2021 adapted from Garside (2022).

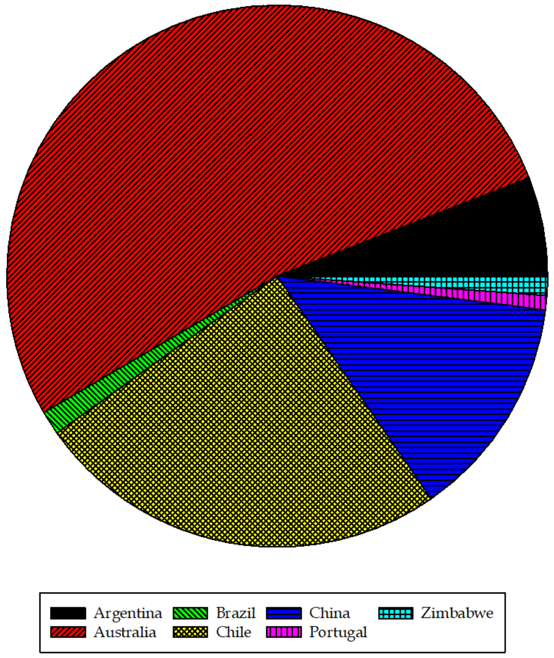

Fig. 3 Distribution of lithium production worldwide in 2021, by country.

2.3 Lithium Industry in Africa

Zimbabwe which holds about 1% of the global lithium resources, holds the highest proportion of lithium reserves in Africa (King, 2020; U.S.G.S., 2022). According to King (2020), Zimbabwe holds 28% of the active lithium projects in Africa. Fig 4 shows the distribution of lithium projects in Africa. In 2020, 18 operational mines spread across the continent, and were concentrated in only 8 countries, including Zimbabwe, Democratic Republic of Congo (DRC), Mali, Namibia, Ghana, Tanzania, Madagascar, and Mozambique (King, 2020). The reader is referred to King (2020) and Goodenough et al. (2021), where details of the lithium mining projects have been provided.

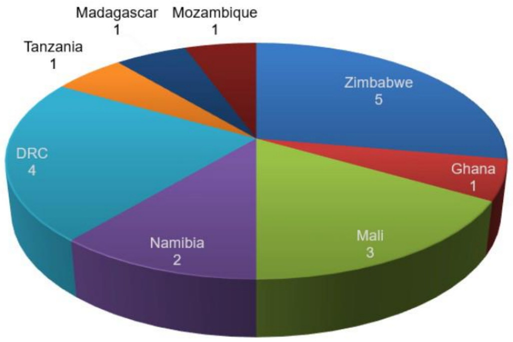

Fig.4 Distribution of Lithium Projects in Africa adapted from King (2020).

2.4 Lithium Industry in Ghana

Ghana has been tagged as a potential major lithium production hub in Africa and is set to become the first West African lithium producer. In 2021, it was reported in the mainstream media that, Iron Ridge Resources (Australia) entered into a conditional binding agreement with Piedmont Lithium to fund and expedite the Ewoyaa Lithium Project to full scale production. The Ewoyaa Lithium Project is estimated to contain 14.5 Mt Li_2O at a grade of 1.31% in the inferred and indicated category, including 4.5 Mt at 1.39% Li_2O in the indicated category (IronRidge Resources, 2021). The project is highly probable for tin, tantalum, niobium, caesium and gold, which occur as accessory minerals (Goodenough *et al.*, 2021; King, 2020; Resources, 2021). The project, once in operation is expected to benefit from proximity to infrastructure (including highway, power infrastructure, and a harbor, located in Takoradi). Specifically, spodumene concentrate from the project will be transported by road to Takoradi Port for export (Goodenough *et al.*, 2021).

2.5 Lithium Mineralization

Naturally, lithium does not occur in elemental form due to its reactivity. Lithium resources are mainly grouped into three categories, including brine (> 60% reserve), pegmatites (23-30% reserve) and sediment-hosted deposits (< 3% reserve) (Dessemond *et al.*, 2019). There are about 145 known lithium-bearing minerals, however, not all these minerals can be grouped as economically viable minerals (Karrech *et al*., 2020). The lithium pegmatite ores contain lithium minerals such as spodumene, petalite, lepidolite and amblygonite. There are other lithium minerals such as zinnwaldite, triphylite and eucryptite and jadarite (Tadesse *et al*., 2019). Lithium does accumulate to economic levels in some specific clays such as hectorite (Kavanagh *et al.*, 2018; Tabelin *et al.*, 2021).

Spodumene is of primary economic importance as it is the most abundant lithium-bearing mineral. It is a stable aluminum silicate ($LiAlSi_2O_6$) lithium mineral and has a theoretical lithium content of 3.73% (Dessemond *et al.*, 2019; Meshram *et al.*, 2014; Salakjani *et al*., 2021). It is currently being explored and processed at an industrial scale due to its high Li content and the occurrence of extensive deposits and commercially feasible to process (Garrett, 2004). Table 1 is a summary of selected major lithium-bearing minerals and their respective theoretical lithium content as reported in the literature.

Table 1. Selected major lithium-bearing minerals (Dessemond *et al.*, 2019).

Mineral	**Formula**	**Li content (%)**
Spodumene	$LiAlSi_2O_6$	3.73
Petalite	$LiAlSi_4O_{10}$	2.27
Eucryptite	$LiAlSiO_4$	5.51
Bikitaite	$LiAlSi_2O_6.H_2O$	3.40
Lepidolite	$KLiAlSi_3O_{10}(OH,F)_2$	3.84
Zinnwaldite	$KLiFeAl_2Si_3O_{10}(F,OH)_2$	1.59
Amblygonite	$(Li,Na)AlPO_4(OH,F)$	4.73
Lithiophylite	$LiMnPO_4$	4.43
Jadarite	$LiNaAlSiB_2O_7(OH)$	2.85
Zabuyelite	Li_2CO_3	18.79

3 Characterization Tools

In the assessment of resources for metallurgical beneficiation and subsequent extraction processes, the chemical and mineralogical compositions ought to be ascertained. This plays a crucial role in selecting various units of operations and understanding the response/behaviour of the material during processing and beneficiation. Typically, the chemical composition of the material could be determined using Inductively Coupled Plasma Spectroscopy (ICP) (Ammann, 2007; Ghosh *et al.*, 2013), Ion Selective Electrodes (Arnold and Meyerhoff, 1984; Dimeski *et al.*, 2010; Freiser, 2012), and X-ray fluorescence (XRF) (Bertin, 2012; Murphy *et al.*, 2013; Rousseau, 1984). On the other hand, X-ray diffraction (XRD) (Bunaciu *et al.*, 2015; Chauhan and Chauhan, 2014; Epp, 2016; Khan *et al*., 2020) and Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN) (Grammatikopoulos *et al.*, 2021; Simandl *et al.*, 2017; Wilde *et al*., 2021) have been instrumental in determining the detailed mineralogical composition of different materials including liberation and association relationships between minerals.

4 Lithium Beneficiation

4.1 Dense Media Separation

Dense media separation (DMS) or heavy media separation is a preconcentration technique usually employed for coarse gangue rejection prior to grinding, but can also be used to produce lithium concentrates from high grade ores (Tadesse *et al.*, 2019). This method utilizes the differences in specific gravity between the mineral of interest and the gangue minerals. DMS is commonly used in the separation of lithium minerals from the major gangue silicates based on its relatively high specific gravity between 3.1–3.2 (Oliazadeh *et al*., 2018). It is worth noting that lithium mineral such as spodumene is slightly heavier as compared to silicate gangue minerals present in the hard rocks including pegmatites-quartz (2.65), feldspars (2.6), and micas (2.8–3.0) (Gibson *et al*., 2021; Oliazadeh *et al.*, 2018; Tadesse *et al.*, 2019). Lithium minerals concentration by DMS is typically conducted on −9.5 mm +850 µm ore fraction since DMS is most effective at relatively coarse size fraction (Marion *et al*., 2017; Oliazadeh *et al*., 2018).

4.2 Magnetic separation

Magnetic separation utilizes the differences in the magnetic susceptibility behaviour of the mineral of interest relative to the gangue mineral(s) when exposed to an applied magnetic field. This separation process typically employed to remove iron bearing gangue minerals in association with the lithium values (Tadesse *et al*., 2019). Typically, zinnwaldite has a very high magnetic susceptibility due to its high iron content, which makes it amenable to magnetic beneficiation. For example, in Czech Republic, zinnwaldite is separated from tin-tungsten mining tailings (Botula *et al.*, 2013). Elsewhere, magnetic separation has been used to clean zinnwaldite flotation concentrate (Siame and Pascoe, 2011).

4.3 Froth Flotation

Froth flotation exploits the differences in the hydrophobicity of mineral particles to separate them. It is the most widely used method for the beneficiation of lithium bearing minerals (Tadesse *et al*., 2019; Tian *et al*., 2018). The efficiency of flotation recovery of lithium minerals can be affected by the surface chemistry of minerals, collector type and concentration used, pulp pH, pretreatment methods, and the presence of slimes. However, this method produces "richer" lithium concentrate as compared with other preconcentration methods (Tadesse *et al*., 2019). Typically, anionic collectors including oleic acid, sodium oleate, sulphonated and phosphorated fatty acids are important surfactants in the flotation of lithium minerals (Tadesse *et al*., 2019).

4.4 Hydrometallurgical Processes

Lithium preconcentrates are subjected to different extraction processes using direct leaching methods. For example, hydrochloric acid (HCl) has been proposed to leach zinnwaldite and β-spodumene (Martin *et al*., 2017). Margarido *et al*. (2014) suggested that higher reagent concentrations are necessary to achieve better recoveries using HCl, which renders the process prohibitively expensive in terms of energy and chemical costs. Elsewhere, lepidolite concentrate has also been leached directly with sulphuric acid (H_2SO_4) at a temperature of 138ºC for 10 h (Liu *et al.*, 2019). Hydrofluoric acid was effective in producing 90% lithium recovery at 75ºC (Rosales *et al.*, 2014).

5 Lithium Extraction

5.1 Conventional ore

Spodumene is the most abundant lithium mineral that has been commercially mined and processed to produce lithium compounds around the world among the lithium-bearing ore. Spodumene accounts for approximately 90% of global lithium carbonate equivalent production (Dessemond *et al.*, 2019; Tran and Luong, 2015).

Spodumene processing starts in a similar approach to many minerals; the ore is crushed and upgraded by physical separation methods such as dense media separation (DMS), ore sorting, magnetic separation, and flotation. These are employed to rejects associated gangue minerals such as feldspar, micas, and quartz before further extraction processes (Sousa *et al.*, 2019; Tadesse *et al*., 2019; Tran and Luong, 2015; Xu *et al*., 2016).

Subsequently, spodumene in the α-phase is difficult to treat hence has to be transform to β-spodumene by roasting at about 1040ºC to 1100ºC to enhance leaching under moderate chemical extraction conditions (Dessemond *et al*., 2020; Tran and Luong, 2015). The β-spodumene concentrate (calcined) is further roasted with acidic, alkaline or chlorinated chemicals (Meshram *et al.*, 2014).

In the acid process, the β-spodumene is ground and mix with concentrated sulfuric acid (H_2SO_4) and roasted at a temperature of 250°C. This process produces an insoluble ore residue and soluble lithium sulfate Li_2SO_4 (Meshram *et al.*, 2014). On the other hand, the β-spodumene concentrate is ground and calcined with limestone or soda ash at 825-1050ºC to convert lithium silicates into a soluble form (Meshram *et al.*, 2014). The soluble lithium is then water leached and then reacted with carbon dioxide to convert lithium to aqueous lithium bicarbonate. Pregnant leach solution is obtained and then subjected to evaporation at around 90ºC to crystallize the lithium as Li_2CO_3 if soda ash is used for neutralization or as lithium hydroxide monohydrate if limestone is used for neutralization (Meshram *et al*., 2014).

Chlorination roasting of spodumene takes place at temperatures above 1000°C in the presence of chlorine gas (Barbosa *et al.*, 2014; Yan *et al.*, 2012). This process produced a soluble lithium chloride (LiCl) which can be water leached and subjected to evaporation or other purification processes. Roasting β-spodumene with Cl_2 gas at 1100°C for 2.5 h could results in almost complete extraction of lithium as $LiCl_2$ (Barbosa *et al.*, 2014).

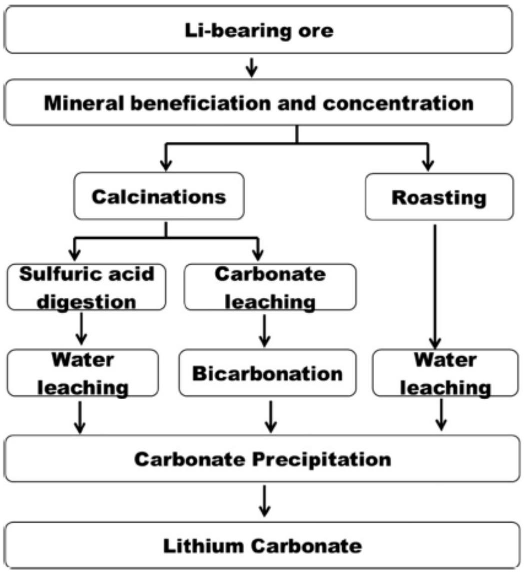

Fig. 5 Typical flowsheet for processing lithium ore (Swain, 2017).

5.2 Recycled Batteries

Spent lithium-ion batteries (LIBs) contain a cathode, an anode, and an organic electrolyte which consist of dissociated salts such as $LiPF_6$ and a separator. The cathode of LIB is usually aluminum foil coated with materials such as oxide formed by a lithium metal oxide that can produce higher potentials such as lithium cobalt oxide ($LiCoO_2$). On the other hand, the anode is a copper foil coated with graphitic carbon, which can hold Li in its layers. Prior to processing of spent LIBs, they must be discharged by contacting it with salt solutions such as NaCl to prevent short circuiting and self-ignition that will cause explosion (Wang *et al.*, 2018; Winter and Brodd, 2004).

Traditionally, hydrometallurgical and pyrometallurgical processes have been employed in the extraction of lithium from LIBs. This may be preceded by comminution processes to produce material of suitable size fraction followed by physical preconcentration methods to reject significant amount of gangue materials (Meshram *et al.*, 2014). In the leaching process, organic and inorganic acids and alkaline solutions are employed in the presence of H_2O_2 to recover lithium and cobalt. Subsequently, solvent extraction or selective precipitation processes are carried out to separate and recover both metals (Shuva and Kurny, 2013). Recently, biological treatments have been employed to recover lithium and other metals from spent LIBs (Purnomo *et al.*, 2018). Fig. 6 shows different processes employed in recycling LIBs.

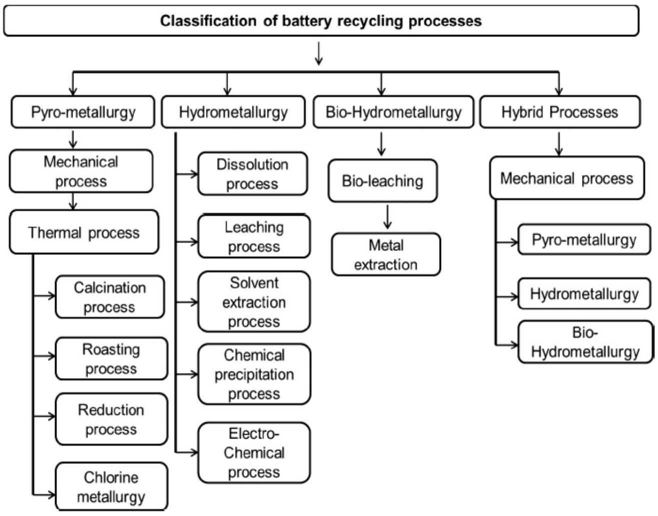

Fig. 6 Classification of processes for recycling LIBs (Swain, 2017).

6 Conclusions

The global distribution and production of lithium have been reviewed. Specifically, the distribution of lithium resources in Africa have been identified with majority of the projects located in Zimbabwe. Ghana is well-placed to become the first West African lithium-producing country.

Spodumene is the most abundant lithium-bearing mineral and has been commercially processed to produce lithium compounds. In lithium beneficiation, upstream methods including DMS, magnetic separation, and flotation are important to pre-concentrate lithium from deleterious gangue minerals, prior to hydrometallurgical and pyrometallurgical processes. The choice of the given preconcentration method depends on chemical and mineralogical composition of the ore. To this effect, characterization tools including Inductively Coupled Plasma Spectroscopy (ICP), Ion Selective Electrodes, X-ray fluorescence (XRF) are employed to identify the chemical composition of the ore. The data from these tools are instrumental in determining the mineralogical composition of the ore via X-ray diffraction (XRD) and Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN) analyses.

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