

**SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING
UNIVERSITI SAINS MALAYSIA**

**PHYSICAL AND CHEMICAL CHARACTERIZATION OF GRANITIC
ROCK FOR REE UTILIZATION**

By

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CHARACTERIZATION OF GRANITIC ROCK
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UNIVERSITI SAINS MALAYSIA

2022

DECLARATION

I hereby declare that I have conducted, compiled the research work and written the dissertation entitled “**Physical and Chemical Characterization of Granitic Rock For REE Utilization**”. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or University.

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ABSTRACT

Rare earth element (REE) is important mineral as petroleum nowadays. It is highly demand in varies field especially electronic and green technology. Granitoid and some intrusive volcanic rocks are widely spreading in Malaysia and thus, it could occupied the demand by industries and population needs as based on previous research, Malaysia's S-type granite is rich with yttrium. The objective of this research is to analyse the mineralogy of granitic deposits by its physical and chemical characteristics. The study was conducted at Kupang, Baling District, Kedah. This geological area is known as Kupang Granite and contacted with Kroh Formation. To achieve the objective, a proper flowchart was planned by divided it into three parts which are physical characteristic, petrology characteristic and geochemical characteristic. Several methods could be conducted to achieve the objective by using polarizing microscope, X-ray diffraction (XRD), X- ray fluorescence (XRF) and Inductive Coupled Plasma Mass Spectrometer (ICP-MS). The results obtained from the visual assessment and under polarizing microscope shown the mineral exists in granite such as quartz, feldspar and biotite. XRD analysis justified the mineral existed in the thin section as all the highest peak intensity show kaolinite, quartz, albite and orthoclase. XRF analysis showing that all the samples have higher weight percent of SiO₂. The major element results interpreted the type of rock by plotting on TAS diagram. ICP-MS was utilized to obtain the concentration of REE. From the observation, the REE content in all samples are quite lower that the required mineable recovery. This information can be utilized for further exploration of REE in granitic rock.

ABSTRAK

Unsur nadir bumi (REE) merupakan mineral penting seperti petroleum pada masa kini. Ia sangat mendapat permintaan yang tinggi dalam pelbagai bidang terutamanya teknologi elektronik dan hijau. Granitoid dan beberapa batuan gunung berapi yang meletus tersebar secara meluas di Malaysia, ia boleh memenuhi permintaan oleh industri dan keperluan penduduk kerana berdasarkan penyelidikan terdahulu, *S-type* granit Malaysia kaya dengan yttrium. Objektif kajian ini adalah untuk menganalisis mineralogi mendapan granit mengikut ciri fizikal dan kimianya. Kajian dijalankan di Kupang, Daerah Baling, Kedah. Kawasan geologi ini dikenali sebagai Kupang Granite dan mempunyai interaksi dengan Formasi Kroh. Untuk mencapai objektif, carta alir telah dirancang dengan membahagikannya kepada tiga bahagian iaitu ciri fizikal, ciri petrologi dan ciri geokimia. Beberapa kaedah boleh dijalankan untuk mencapai objektif dengan menggunakan mikroskop polarisasi, pembelauan sinar-X (XRD), pendarfluor sinar-X (XRF) dan Spektrometer Jisim Plasma Berganding Induktif (ICP-MS). Keputusan yang diperoleh daripada penilaian visual dan di bawah mikroskop polarisasi menunjukkan mineral wujud dalam granit seperti kuarza, feldspar dan biotit. Analisis XRD membuktikan mineral wujud dalam keratan tipis kerana semua keamatan puncak tertinggi menunjukkan kaolinit, kuarza, albit dan ortoklas. Analisis XRF menunjukkan bahawa semua sampel mempunyai peratusan berat SiO₂ yang tinggi. Keputusan elemen utama mentafsir jenis batuan dengan memplot pada rajah TAS. ICP-MS telah digunakan untuk mendapatkan kepekatan REE. Daripada pemerhatian, kandungan REE dalam semua sampel adalah agak rendah daripada nilai pemprosesan yang dibenarkan. Maklumat ini boleh digunakan untuk penerokaan lanjut REE dalam batuan granit.

CHAPTER 1

INTRODUCTION

1.1 Background Project

Malaysia is listed as one of the country that have abundance of rare earth element (REE) and readily to be extracted and produced. Table 1.1 shows the list of countries and their ore reserves of REE. Malaysia presently manufactures monazite as a byproduct of tin production and has previously generated xenotime. Production varies year to year, with only 233 tonnes extracted in 2008 (J. Weser, 2020). Many reports cite that Malaysia has reasonably substantial amounts of REEs.

Table 1.1 Estimated world REE reserves defined by USGS (Hendrick, 2010)

Country	Reserves (tonnes)
China	36 000 000
Commonwealth of Independent States	19 000 000
United States	13 000 000
Australia	5 400 000
India	3 100 000
Brazil	48 000
Malaysia	30 000
Other Countries	22 000 000
World Total	99 000 000

Over the last three decades, several technologies that use REE and the alloys in their components have been increasing gradually. These technologies including computer memory, DVDs, rechargeable batteries, autocatalytic converters, super magnets, mobile phones, LED lighting, superconductors, glass additives, fluorescent

materials, phosphate binding agents, solar panels and magnetic resonance imaging (MRI) agents (Balaram, V., 2019).

Figure 1.1 shows the summarization of REE consumption by volume in the industries. Table 1.2 represent the examples of REE being utilized in various places across the world.

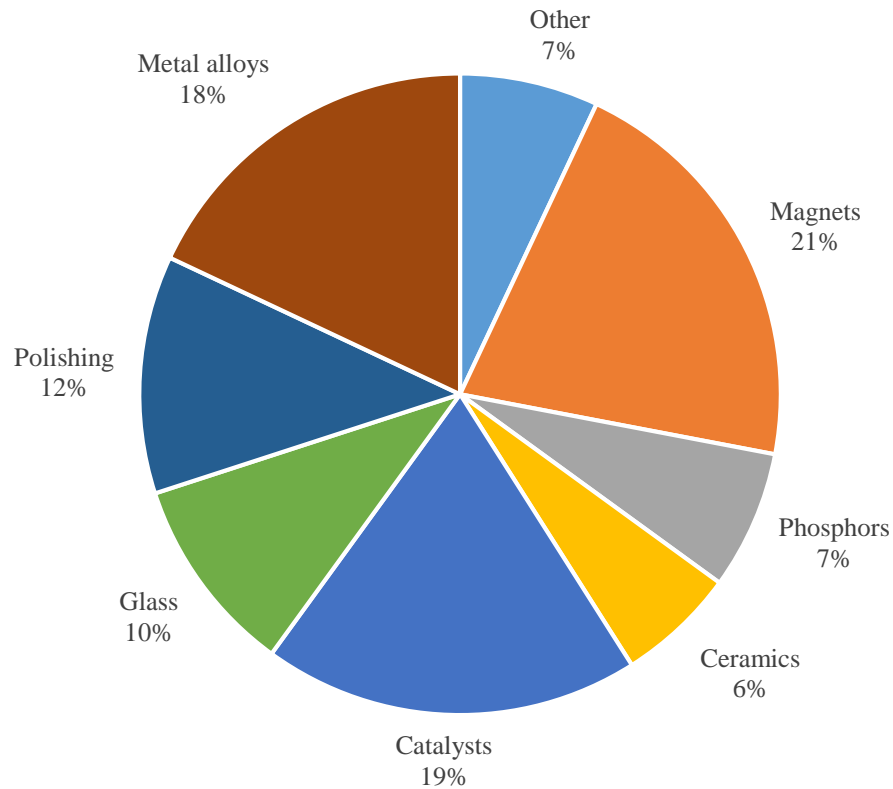


Figure 1.1 REE consumption by volume (Kingsnorth, 2009)

The global market's relationship between demand and availability of REE resources has been increasing gradually in recent years (Roskill, 2007). Prices of heavy rare earth elements (HREE: Gd-Lu) have risen considerably in comparison to light rare earth elements (LREE: La-Eu) (Burton, C.K., 1986). In Malaysia, the industries are started to explore those minerals to achieved the demands. The exploration including the study formation of granitic rock in Malaysia. Based on many researches, Malaysia's granitic rock has potential of the REE existence and able to meet the

demands as Malaysian S-type granites have a high yttrium concentration (Cobbing et al., 1992).

Table 1.2 Examples of REE Being utilized in different areas (Kingsnorth, 2009)

Area	Applications
Electronics	Television screens, computers, cell phones, silicon chips, monitor displays, long-life rechargeable batteries, camera lenses, light emitting diodes (LEDs), compact fluorescent lamps (CFLs), baggage scanners, marine propulsion systems
Manufacturing	High strength magnets, metal alloys, stress gauges, ceramic pigments, colorants in glassware, chemical oxidizing agent, polishing powders, plastics creation, as additives for strengthening other metals, automotive catalytic converters
Medical Science	Portable X-ray machines, X-ray tubes, magnetic resonance imagery (MRI) contrast agents, nuclear medicine imaging, cancer treatment applications, and for genetic screening tests, medical and dental lasers
Technology	Lasers, optical glass, fiber optics, masers, radar detection devices, nuclear fuel rods, mercury-vapor lamps, highly reflective glass, computer memory, nuclear batteries, high temperature superconductors
Renewable Energy	Hybrid automobiles, wind turbines, next generation rechargeable batteries, bio-fuel catalysts
Others	<p>The europium is being used as a way to identify legitimate bills for the Euro bill supply and to dissuade counterfeiting. An estimated 1 kg of REE can be found inside a typical hybrid automobile.</p> <p>Holmium has the highest magnetic strength of any element and is used to create extremely powerful magnets. This application can reduce the weight of many motors</p>

1.2 Importance Of Project

This project is focusing on physically and chemically characterized the granitic rock at Kupang, Kedah. The granitic formation in this area is called as Kupang Granite. Kupang Granite has potential on producing rare earth elements such as apatite, zircon, xenotime and allanite. Apatite is a source of phosphate in the manufacture of fertilizer and industrial uses. Zircon is widely known used for casting and refractory applications in the foundry industry. Therefore, the information from this research can be utilized for exploiting valuable rare earth element and minerals in the Kupang Granite's deposits.

1.3 Problem Statement

Characterization of ore is extremely important to identify the existence of REE components, content and condition it is in the weathered granitic rock. However, a visual assessment is not enough to support the findings. From the characterization, the corresponding extractive processes can be suggested for a proper extraction of the recovery.

1.4 Objectives

The objectives in this study are as follows:

- 1.4.1 To characterize the mineral based on the physical characteristics of weathered granitic rock sample.
- 1.4.2 To determine the petrology of granitic rock sample based on the mineral existed.
- 1.4.3 To stimulate geochemical of granitic rock sample based on LREE and HREE contents.

1.5 Thesis Outline

The physical and chemical characterization of Kupang Granite involving several assessments such as particle size distribution, elemental composition, identification of mineral phases, mineral identification and mineral liberation. Chapter 2 will cover the geological background of Kupang, Kedah which then indicates where the chemical composition of heavy minerals found. This section will demonstrate the previous studies regarding characterization of REE in granitic rock.

Chapter 3 comprises in details of proper procedure for each analysis performed in the characterization of Kupang Granite. This section classified into three which are physical, petrology and geochemistry characteristics. In physical characteristic, it is mainly focusing on the moisture content, loss on ignition and particle size distribution. Petrology study comprises the analysis of petrographic and mineral phase of the samples. Geochemistry study focusing on the analysis of major element and rare earth element using ICP-MS.

The results from overall assessment will be discussed in Chapter 4. In this chapter, the samples will be further classified into its granite type. This can be achieved by analyzing the major element oxides present in the samples. To support the the results, it can be related to identifying the minerals under polarizing microscope.

Lastly, the conclusion from the discussed results will be summarized in Chapter 5 in agreement with the objectives of this characterization studies. Moreover, the recommendation for future research is added in this chapter to improvise the experimental work for a better result.

1.6 Location Of Study Area

The study areas were located at Kupang, Baling District, Kedah, where located within the western granite belt of Peninsular Malaysia. The distance of the research areas from Universiti Sains Malaysia is approximately 99.7 km. Around 1 hour and 24 minutes of journey if travelled by car.



Figure 1.2 The geological map of research area

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Chemical weathering of granitic rocks and subsequent chemical adsorption of REE on kaolinite and halloysite form ion-adsorption type deposits. These deposits are distinguished by one to five times the condensation of REE in weathered crusts relative to parent rocks and the convenience for which REE can be recovered from clayish ores using weak acids. The average REE content of ion-adsorption ores ranges from 300 to 2000 ppm and varies depending on the parent rocks (Yang, Y., 1981).

Bao and Zhao (2008) reported that the parent rocks of the weathered crusts are I-type and S-type granitic rocks, with some A-type granitic rocks and are primarily classified as ilmenite-series (Bao & Zhao, Z., 2008). According to Koljonen and Rosenberg (1974), the behaviour of REE in granitic rocks may be summarized as follows:

- a. During magmatic differentiation the content increases and in granites it is nearly three times that in basalts (Herrmann 1970).
- b. Light REE from La to Sm are enriched compared with the heavy ones from Gd to Lu.
- c. Europium differs from the other REE in having the possible valence +2 (ionic radius 1.09 Å) as well as +3.
- d. Ce may oxidize to Ce^{4+} .

The majority of these granitic rocks have biotite muscovite mineral assemblages with K-feldspar phenocrysts (Dianhao et al., 1989). Hoshino et al., (2009) discovered in their researches that the majority of the granitic rocks collected are calc

alkaline biotite ± hornblende granodiorite, biotite granite and biotite granite porphyry. They are made up of plagioclase, K-feldspar, quartz, biotite, and hornblende, along with significant quantities of other minerals. The most common opaque minerals are ilmenite, hematite, and pyrite, whereas magnetite series granitic rocks contain magnetite. Apatite, zircon, monazite, xenotime, allanite, titanite, and almandine are indications of REE minerals and REE-bearing minerals (Hoshino et al., 2016).

This section covers the stages of mineral characterization was done by other researchers in their study as well as the results reported. The chemical analysis of REE in geological, industrial, and environmental materials is critical for geochemical exploration investigations, mining, extraction, quality checks of raw materials and final products in industry, and environmental monitoring. Characterizing REEs is challenging and expensive because of their physical and chemical affinities.

Among instruments available currently, Inductive Coupled Plasma Mass Spectrometry (ICP-MS) is commonly used for REE identification. The starting point in investigations of all types of samples is an adequate characterization in terms of their size, habit, chemical composition, morphology, texture, association with other minerals and other physical attributes (Cook, NJ., 2000). The common application research instruments such as X-ray diffraction analysis (XRD), image analysis, quantitative electron probe microanalysis (EPMA), and scanning electron microscopy (SEM) complements traditional microscopic investigations and geochemical analysis.

2.2 Definition Of Rare Earth Element (REE)

The rare earth elements (REEs) group is officially known as "lanthanoids" or "lanthanides," and is comprised of 15 different elements having atomic numbers varying from 57 to 71 in the periodic table. Yttrium with atomic number 39 have the

similarity in its chemical and physical affinities with the lanthanoids, thus it is regarded as REE. The REEs are commonly found together in the Earth's crust as they share the similar ionic radii and a trivalent charge (+3). Bradley et al., (2014) stated that the REEs do exist in minerals as either minor or major constituents but do not exist individually like gold or copper. The average rare earth content of the three major minerals; namely, monazite, bastnasite and xenotime is given in Table 2.1:

Table 2.1 Average rare earth content of the major ore minerals (%)
(Sastri et al., 2003)

Element	Monazite	Bastnasite	Xenotime
La	23	32	
Ce	46	50	
Pr	5	4	
Nd	19	13	
Sm	3	0.5	1.2
Eu	0.01	0.1	0.01
Gd	1.7	0.15	3.6
Tb	0.16		1.0
Dy	0.5		7.5
Ho	0.09		2.0
Er	0.13	*	6.2
Tm	0.01		1.27
Yb	0.06		6.0
Lu	0.006		0.63
Y	2		60.0
*	Almost absent		

2.3 Type Of REE Deposit

Rare earth elements were born in a supernova explosion around ten billion years ago. However, the formation of REE is complex and not fully understood but can be thought of as a geologic process from alkaline igneous rocks that form from cooling of magmas derived by partial melting of rocks in the Earth's mantle. The resulting alkaline magmas unusually enriched in elements such as zirconium, niobium, strontium, barium, lithium and the rare earth elements (Long et al., 2012). REE are relatively abundant in the Earth's crust, however they are rarely concentrated into mineable ore deposit. Instead, they are found in the same ores and deposit which have proved to be particularly complex to separate from each other due to similar physical and chemical properties. Balaram (2018) stated that the potential REE deposits can be categorized into primary and secondary deposits. The term primary deposits refers to those created by magmatic, hydrothermal, and/or metamorphic processes. These deposits are typically associated with alkaline igneous rocks and carbonatites that have been deposited in extensional environments. Secondary deposits, which might include placers, laterites, and bauxites, are those generated by erosion and weathering. Within these two groupings, REE deposits can be further classified based on genetic relationships, mineralogy, and occurrence type (Balaram, V., 2019).

In the present study, the REE deposits can be subdivided into five categories as followed below (Balaram, V., 2019):

- a. **Alkaline igneous rocks: pegmatites and carbonatites** - It is generated by the partial melting of deep mantle rocks that rise and cool beneath the Earth's crust. Alkaline magmas are frequently rich in both REE and also Zr, Nb, Sr, Ba, and Li. As magma rises, it experiences chemical changes due to the complex interaction of factors such as temperature, pressure, and the

chemistry of the surrounding rocks. This intricate interaction results in the formation of a diverse variety of REE deposits. (Ray & Shukla, 2004).

- b. **Residual deposits** - It is formed from deep weathering of igneous rocks, pegmatites, and iron-oxide copper-gold deposits. Carbonatite and peralkaline intrusive weathering may result in concentrated residual deposits of REE minerals.
- c. **Heavy mineral placers** - Most placer deposits with significant amounts of REE minerals are Tertiary or Quaternary deposits derived from source areas that include granitic rocks or high-grade metamorphic rocks (Balaram, 2019).
- d. **REE in coal** - Franus et al., (2015) discovered that coal fly ash, which is considered waste, is now seen to be a potential source of numerous elements, including REE. This is because the REE concentrations in many coals or coal ashes are comparable to or greater than those found in typical REE ores.
- e. **REE in the sediments of continental shelf and Ocean bottom** - Recent studies of marine phosphorite deposits reveal that there are significant quantities of REE contained within the phosphorites (Mazumdar et al., 1999). Minerals eroded off the land may be concentrated as placer deposits near the coast, and comparable placer deposits might be predicted further offshore, beneath the ocean. Massive quantities of phosphorite (phosphate rock or rock phosphate), a non-detrital sedimentary rock that is also a major source of REE, may also be found on continental shelves (Balaram, V., 2019).

REEs are found in a wide range of rock-forming minerals, however the most economically relevant sources, according to O'Driscoll (1991), are grouped into the following groups:

i. **Carbonates**

Bastnaesite, $(\text{RE})\text{FCO}_3$, is a magma-derived fluorocarbonate mineral containing 65-75 wt.% rare earth oxides (REO). The two major sources in the world for lanthanides are bastnaesite deposits at Mountain Pass, California (U.S.A) - devoted solely to REE production and Bayun-obo, Inner Mongolia (China) - mined primarily for iron ore and REE as by-product.

ii. **Phosphates**

Monazite, $(\text{RE})\text{PO}_4$ is a light RE phosphate containing 55-56 wt.% REO, associated with granites and beach sands in Australia, Brazil and India, while xenotime $(\text{Y,RE})\text{PO}_4$ is an yttrium rich phosphate containing 25-60 wt.% Y_2O_3 and other heavy REE. Both minerals are recovered as by-products of mining for titanium, zirconium and tin.

iii. **Ion-adsorption Clays**

Contain 0.05-0.2 wt.% REO adsorbed on the surface of alumino-silicate minerals (e.g. kaolinite, illite and smectite). Ion-adsorption clay deposits are formed as a result of in-situ weathering of rare-earth-rich host rocks (granite or igneous), resulting in the creation of alumino-silicate clays. For this lateritic process to occur, these very small mineral particles must be capable of adsorbing lanthanide ions. Southern China is the greatest example of this formation process.

The Table 2.2 below shows another summarization of REE studies by other researchers:

Table 2.2 The studies of REE existence

No	Title	Description	Author
1	REE mineralogy and resources. Handbook on the physics and chemistry of Rare Earths.	The REEs exist ion adsorption when the REE minerals decomposed and clay minerals absorbed the deionized REE due to surface weathering.	Hoshino, M., Sanematsu, K. and Watanabe, Y. (2016)
2	Rare earth element distributions and trends in natural waters with focus on groundwater	A middle REE (MREE) enrichment is observed for freshwater in contact with phosphate-rich minerals, while heavy REE (HREE) enrichment is found in carbonate-rich waters.	Noack, C.W., dzombak, D.A. and Karamalidis, A.K. (2014)

2.3.1 Ion-adsorption Clay

The term "clay" refers to a naturally occurring material composed mostly of fine-grained minerals (clay minerals), which is normally plastic at an adequate water contents and hardens when dried or burnt (Al-Ani & Sarapaa, 2008). Ion adsorption clay deposits are formed by in situ lateritic weathering of rare earth rich host rocks (granitic or igneous), which results in the formation of aluminosilicate clays over geological time. These extremely fine mineral particles can adsorb lanthanide ions that are released or dissolved during weathering (Papangelakis & Moldoveanu, 2014). The statement made by Hoshino et al., (2016) supported the fact that REE minerals are decomposed due to surface weathering. Those ionized REEs are absorbed on clay minerals such as halloysite and kaolinite (Hoshino et al., 2016).

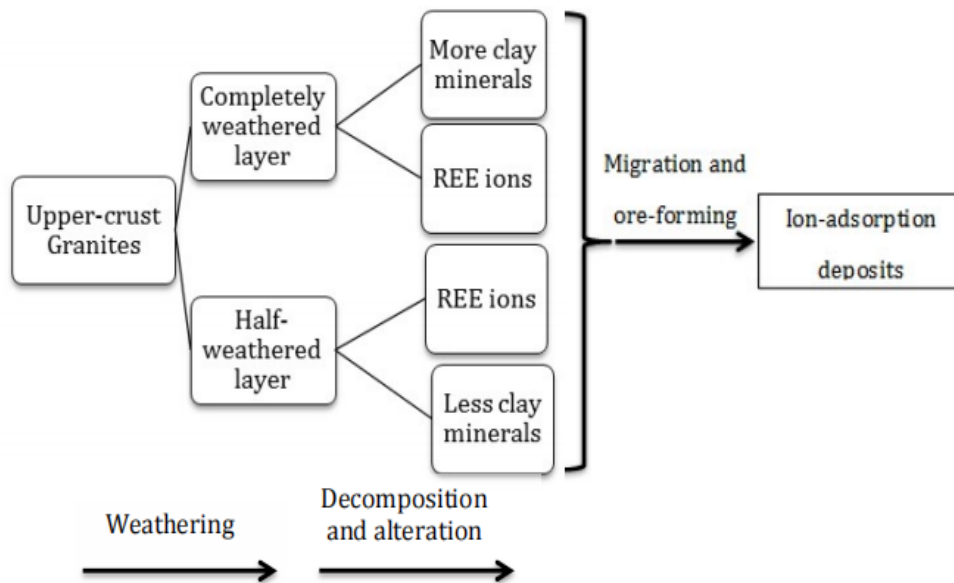


Figure 2.1 Metallogenic mechanism of formation for REE-rich adsorption-type ores (adapted from Peng and Hendrick) (Papangelakis & Moldoveanu, 2014)

Table 2.3 Summary of important clay minerals (NPTEL Civil: Geoenvironmental Engineering)

Mineral	Origin	Bond	Shape	Remark
Kaolinite	Orthoclase Feldspar (Granitic rocks)	Strong hydrogen bond	Flaky and platy	Approximately 100 layers in a regular structure $d = 7.2 \text{ \AA}$
Halloysite	Feldspar Tropical soil	Less strong bond	Tabular or rod like structure	At 600C it loses water and alter soil properties
Illite	Degradation of mica under marine condition Feldspar	K^+ provides bond between adjacent layers	Thin and small flaky material	Bond is weaker than kaolinite $d = 10 \text{ \AA}$ High stability
Vermiculite	Weathering of biotite and chlorite	Presence of H_2O and Mg^{+2} predominantly Mg^{+2}	Platy or flaky particle	Shrinkage and swelling less than montmorillonite

The formation of this clay deposit is due to physical, chemical and biological weathering of REE-rich granitic and volcanic rocks in subtropical zones under warm, humid, slightly acidic conditions. Bao and Zhao (2008) stated that small sizes of ion-adsorption deposits are due to the constraint that ore bodies can only develop near the surface. Thicknesses of the weathering profiles on parent rocks generally range from 6 to 10 m on average, although they can reach up to 30 m (Bao & Zhao, 2008):

- a. An upper humic layer of quartz, organic matter and soil: 0–2 m thick, with very low/nil REE content;
- b. A strongly weathered layer enriched in REE: 5–10 m thick with kaolinite, halloysite, quartz and mica;
- c. A semi- weathered layer: 3–5m thick with kaolinite and sericite;
- d. A weakly weathered bottom layer with the same mineral compositions as the host rock

2.3.2 Xenotime

Xenotime, a phosphate ore, is a valuable REE mineral deposit that have similarity to monazite. According to Zhang et al. (2015) the properties of xenotime are:

- i. The yttrium level is high, but the light rare earths content is low.
- ii. Thorium content is smaller in monazite than xenotime.
- iii. The chemical composition of tungsten-containing xenotime is quite complex. Tungsten has a larger percentage than rare earths, as well as more impurities like iron and silicon. However, niobium, tantalum, beryllium, and niobium concentration is lower.
- iv. The screening procedure is more difficult. Xenotime is harder to disintegrate than monazite in general.

2.3.3 Monazite

Monazite has ABO_4 stoichiometry similar to zircon and thorite, but it has monoclinic crystal structure and consists of chains of alternating BO_4 tetrahedra and AO_9 polyhedral sites. Monazite is a common accessory mineral in peraluminous granites, syenitic and granitic pegmatites, quartz veins and carbonatites but lesser in charnockites, migmatites and paragneisses (Rapp & Watson, 1986). In peraluminous granites, monazite constitutes a major host of LREE, excluding Eu, Th and U, with minor amount of Y and HREE (Hinton & Paterson, 1994). The monazite stability in silicate melts depends on SiO_2 , CaO and P_2O_5 , including oxygen fugacity, peraluminous content and content ratios of lanthanides and actinides (Cuney & Friedrich, 1987).

2.4 Malaysia's REE of Granitic Rock

In 1980s, Malaysia's xenotime-bearing alluvial placer deposits were the world's greatest source of Yttrium (Y). According to JMG (2019), the output of monazite and xenotime in Malaysia increased from 25 tonnes in 2009 to 1,654 tonnes in 2018, mainly from alluvial tailings in Ipoh, Perak. USGS (2019) estimated that Malaysia has 30,000 tonnes of rare earth oxides (REO) in 2018. For many decades, xenotime, monazite, zircon, ilmenite, and struverite were generated as byproducts of tin mining in Malaysia. Ti materials were further processed to generate TiO_2 , whilst efforts to extract REE from monazite and other heavy minerals were limited by the presence of Th/U in the minerals (Yaraghi, 2020). Malaysian monazite is often well-rounded discrete or as some areas with colour ranging from transparent to deep canary yellow through cream coloured with resinous lustre caused by the continuous

oxidation of REE. The Jerai monazite, on the other hand, has unusually elongated crystals flattened rolled grains with a rich green colour.

Malaysian S-type granites have a high yttrium concentration (Cobbing et al., 1992). This indicates that Malaysia has a sufficient supply of RE-bearing host rocks. Tropical weathering in Malaysia, on the other hand, is another satisfaction factor for the occurrence of ion adsorption type REE deposits. Figure 2.2 below shows the granite provinces in south East Asia. Shafiee et al., (2020) mentioned in their research, in the bulk of granite samples from the Gua Musang area, the abundance of light REE compared to total REE is up to 90%, with the maximum concentration being 574.39 ppm and the lowest being 79.79 ppm. This discovery also links REE enrichment to their genesis and mineral composition as major variables (Shafiee et al., 2020).

The presence of REE minerals are related to its rock formation. Below is the list of summaries from an article written by Hassan and Hamzah (1999) related to the studies of granitic rock in Peninsular Malaysia:

- a. Malayan granites divided the Peninsular into three tectonic provinces:
 - i. West Coast Province - Main Range granite, with smaller batholiths, namely Taiping, Kledang, Bukit Mertajam, Penang and Langkawi.
 - ii. Central Province - Lies in between the West Coast and East Coast, containing a narrow line of plutons extending from Stong Igneous Complex that contains the Noring granite near to the Thailand border through, Senting, Gua Musang and its neighbourhood, Damar, Bukit Kajang and Gunung Benom.
 - iii. East Coast Province - Includes pluton such as the Boundary Range, Maras-Jerong, Jemaluang, Muntahak etc.

- b. Main Range belt are commonly coarse to very coarse grained and contain large phenocrysts up to 5 cm long. It is strictly confined to the granite field, exclusively of S-type and of the Ilmenite-series.
- c. Hutchison (1977) believed that the Main Range is underlain by a well established continental sialic basement based on rock geochemistry and strontium isotope ratios.
- d. According to Liew and Page (1985), the West Coast Province is composed of S-type granites that were recirculated, at least in part, from mid-Proterozoic crystalline basement sources. Langkawi granite has an intermediate microcline and significant contact metamorphic aureole. The Eastern Province granites have an equigranular to weakly porphyritic structure, showing that they were emplaced epizonally (Liew & Page, 1985).
- e. Hutchison (1989) stated that since the Triassic period, the Eastern Province has been stable. Biotite granite and biotite-hornblende granite with euhedral hornblende are the major lithologies. East Coast granite has various affinities in terms of granite series. The northeast subprovince from Kuantan to Kelantan, including the Boundary range, is of mixed I and S kinds, whereas eastern Johor and Singapore are I-type.

Figure 2.2 below shows the granite province of SE Asia. In Kelantan, Patah MFA and other authors stated in their research (2021) that Kelantan is consider as a unique territory to have all three belts:

- i. Western Belt - Main Range Granite
- ii. Central Belt - Jeli Granite, Kemahang Granite, Noring Granite, Kenerong Leucogranite, Berangkat Tonalite abd Senting Granite
- iii. Eastern Belt - Boundary Range Granite

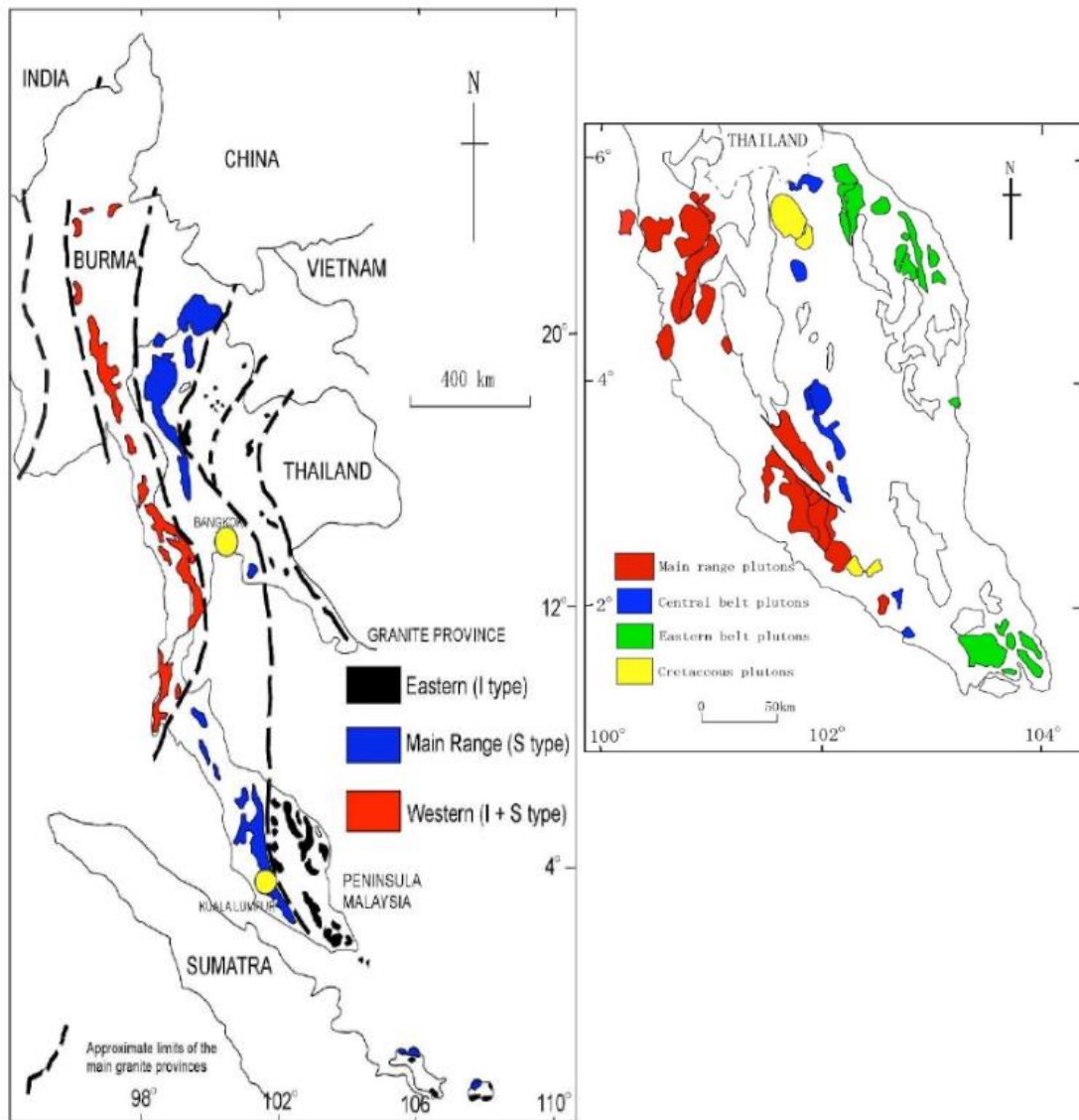


Figure 2.2 Granite province of SE Asia (Cobbing et al., 1992)

2.4.1 Geological Characteristic of Kroh Formation

Kroh Formation is a part of Peninsular Malaysia's Western Belt formations. The Kroh Formation in the Western Belt ranges from the Upper Ordovician to the Lower Devonian. It is made up of black, extremely thin- to thin-bedded mudstone, siliceous carbonaceous shale, calcareous rocks, and chert with arenaceous lenses (Burton, C.K., 1986). Figure 2.3 shows the stratigraphic column of Kroh Formation.

PERIOD/EPOCH	FORMATION/UNIT	STRATIGRAPHIC COLUMN	DESCRIPTION
QUATERNARY	HOLOCENE	Alluvium (Qa)	Surficial deposits - unconsolidated gravel, sand, silt and clay of fluvial and colluvial origin.
	PLEISTOCENE	Nenering beds (Qnn)	Semi-consolidated deposits consist of gravelly, sandy and silty beds, channelling is common.
TERTIARY			Unconformity
CRETACEOUS	Berapit formation (Kbr)		Conglomerate, grey to reddish, very poorly sorted, subangular to rounded clast of up to 40cm in diameter.
JURASSIC			Unconformity
TRIASSIC			
PERMIAN	Gerik formation (Pgk)		Mainly tuffs of rhyolitic and rhyodacitic composition, occasionally metamorphosed; limestone and calcareous shale lenses occur sporadically; interbeds of tuff, limestone and calcareous shale also occur.
CARBONIFEROUS	Kubang Pasu (Ckp)		Well-bedded arenaceous rocks with subordinate ribbon chert. The rocks had been invariably metamorphosed especially the one closer to the granite body. Ribbon chert
DEVONIAN			d Argillaceous facies: Mainly shale and phyllite; commonly carbonaceous, with subordinate chert.
SILURIAN	Kroh formation (SKr)		e Calc-silicate facies: Grey calc-silicate hornfels.
			b Calcareous facies: Grey to dark grey impure limestone.
			a Arenaceous facies: Mainly metasandstone, occur sporadically within the argillaceous facies.

(It is difficult to establish the order of the rock succession owing to folding, faulting, and the lenticular shape of the rock units as well as a repetitive character of its components)

Figure 2.3 Schematic stratigraphic column of the Betong-Pengkalan Hulu Transect area, Malaysia showing the Kroh Formation (Burton, C.K., 1986)

Krof formation comprised as follows (Burton, C.K., 1986):

- i. Argillaceous facies (41.5%)
Frequently metamorphosed to quartz-biotite hornfels, quartz-chlorite-sericite hornfels, phyllite and green schists
- ii. Siliceous facies (18.5%)
- iii. Calc-silicate facies (32.0%)
The mineral components are quartz, calcite, garnet, diopside, wollastonite, plagioclase and clinzosite in varying proportions
- iv. Limestone facies (7.0%)
- v. Arenaceous facies (1.0%)

2.5 Characterisation of Granitic Rock for REE Utilization

The Table 2.4 shows the characterization studies that had been done in previous field investigation to determine the granitic rock.

Table 2.4 List of characterisation studies of granitic rock.

No.	Title	Characterisation studies	Author, (Year)
1	REE Abundance and REE Minerals in Granitic Rocks in the Nanling Range, Jiangxi Province, Southern China, and Generation of the REE-rich Weathered Crust Deposits	BSE for texture of REE minerals of granite samples	Ishihara, S., Hua, R., Hoshino, M. and Murakami, H., (2008)
2	1. Mineralogical and Chemical Characterization of Zr-REE-Nb Ores from Khalzan Buregtei (Mongolia)—Approaches to More Efficient Extraction of Rare Metals from Alkaline Granitoids	i. Optical microscopic for mineralogical studies ii. SEM for element composition iii. EPMA for element mapping and quantitative chemical analyses of ore-forming minerals	Gronen, L.H., Sindern, S., Katzmarzyk, J.L., Bormann, U., Hellmann, A., Wotruba, H. and Meyer, F.M., (2019)
3	Comparison of characteristic and geochemical behaviors of REEs in two weathered granitic profiles generated from metamictized bedrocks in Western Peninsular Malaysia	i. Polarizing microscope for mineralogical studies ii. SEM for mineral morphology and grain size evaluation iii. XRD for phase composition iv. ICP-MS for trace element concentrations	Yaraghi, A., Ariffin, K.S. and Baharun, N., 2020

2.5.1 Particle Size Distribution

Mineral particle size is an important characteristic in any process involving mineral liberation and separation. The product grade and mineral recovery in most mineral processing facilities need an adequate mineral liberation and optimum size distribution. The most frequent particle size analysis methods include sieving, sedimentation, microscopy, digital image processing, and laser diffraction (Kursun, I., 2009). According to Bernhardt (2000), sieving is done by passing a known weight of sample material through finer sieves at the same time and measuring the amount collected on each sieve to calculate the percentage weight of each size fraction.

Suhaina et al., (2016) utilised Malvern Mastersizer E to analyse particle size distribution which applies a laser diffraction method. The entire sample is transmitted by the laser beam, and diffraction from all of the particles is obtained. Cumulative size distribution and volume distribution graphs were generated directly. The d90, d50, and d10 numbers reflect the 10th, 50th, and 90th percentiles of cumulative passing, respectively, and were determined using Malvern Mastersizer's software. The span value (ψ) meanwhile, for a sample distribution, is defined as equation (1) below (Ismail et al., 2016):

$$\psi = \frac{d_{90} - d_{10}}{2d_{50}} \quad (1)$$

However, d10, d50 and d60 were normally used for sieving method. Therefore, a graph of percentage passing against sieve size is plotted after sieving and obtain the d10, d50 and d60 manually from the graph [0]. The uniformity coefficient (C_u), defined as the ratio of D60 to D10, reflects the variation in particle sizes in soil as shown in equation (2) (Kozhakhmetova, 1995).

$$C_u = \frac{D_{60}}{D_{10}} \quad (2)$$

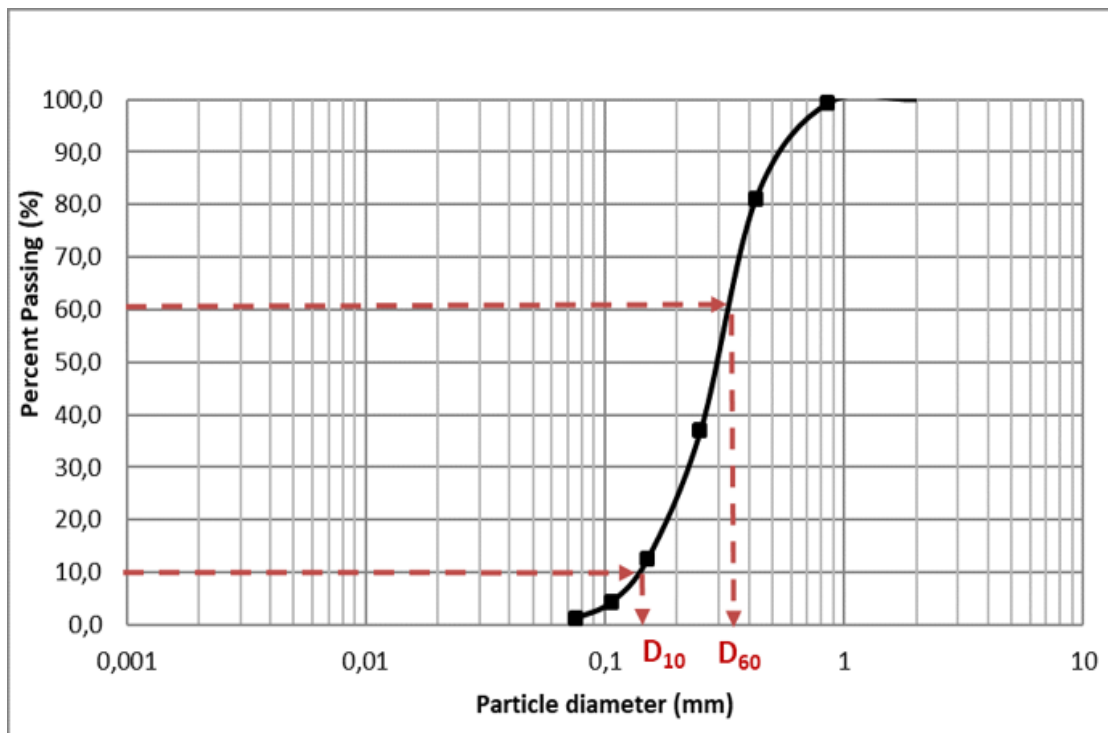


Figure 2.4 The expected graph of grain size distribution (Geoengineer, 2021)

2.5.2 Mineral Identification using Polarizing Microscope

The granitic rock comprised minerals such as quartz, feldspar, plagioclase, biotite, mica and others. In order to determine the type of host, intrusion, altered and mineralised rocks, petrography analysis will be carried out by cutting the rock sample into slice and attached it on the glass thin section before observe it under polarized microscope. According to Fruland (1983), thin sections are produced by attaching tiny slabs of a rock sample on a glass slide (1 inch by 2 inches) and then grinding to a thickness of 0.03mm or 30 microns. Most minerals become more or less transparent at this thickness and may then be investigated under a microscope using transmitted light. These minerals can be identified under crossed polarised light (XPL) and plane polarised light (PPL). Figure 2.5 and 2.6 show the photomicrograph of granite rock.

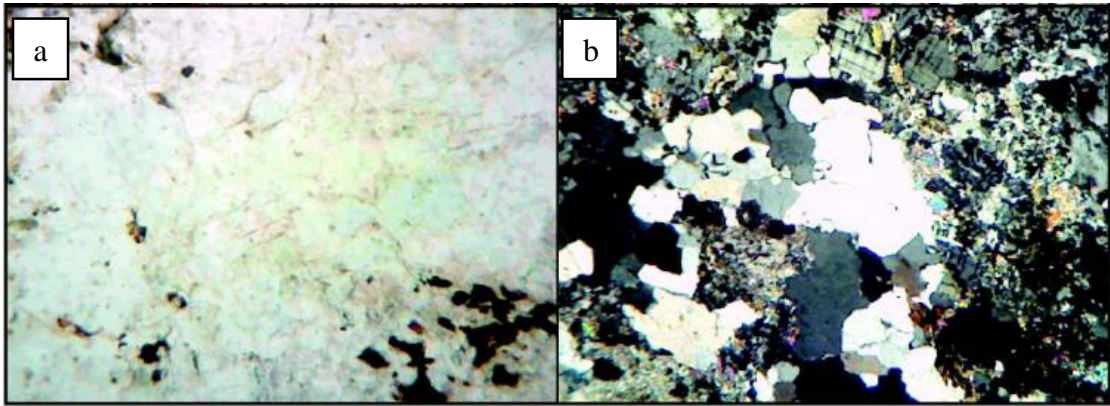


Figure 2.5 Photomicrograph of granite under (a) PPL, (b) XPL

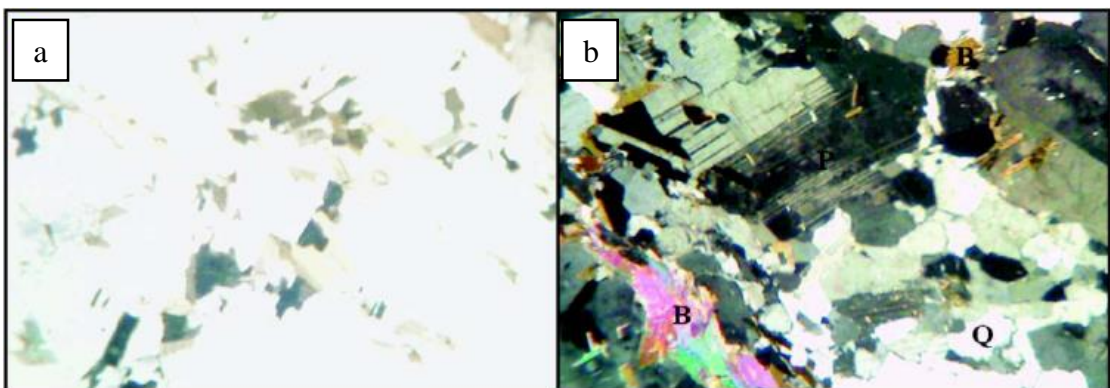


Figure 2.6 Photomicrograph of granodiorite under (a) PPL, (b) XPL

2.5.3 Phase Identification using X-ray Diffraction (XRD)

XRD is an instrumental technique that is used to identify minerals, as well as other crystalline materials. XRD is used to complement other mineralogical techniques in various geologic investigations, such as optical light microscopy, electron microprobe microscopy, and scanning electron microscopy. For ordinary mineral identification, XRD offers the researcher with a quick and reliable instrument. This analysis applies the using an x-ray in the nanometre range to illustrate the sample in which reflections off the different layers will yield bright spots at specific angle. The pattern of spot from x-ray diffraction measurements allows one to Index Diffraction (ID) specific substances means the pattern is unique to each crystal.