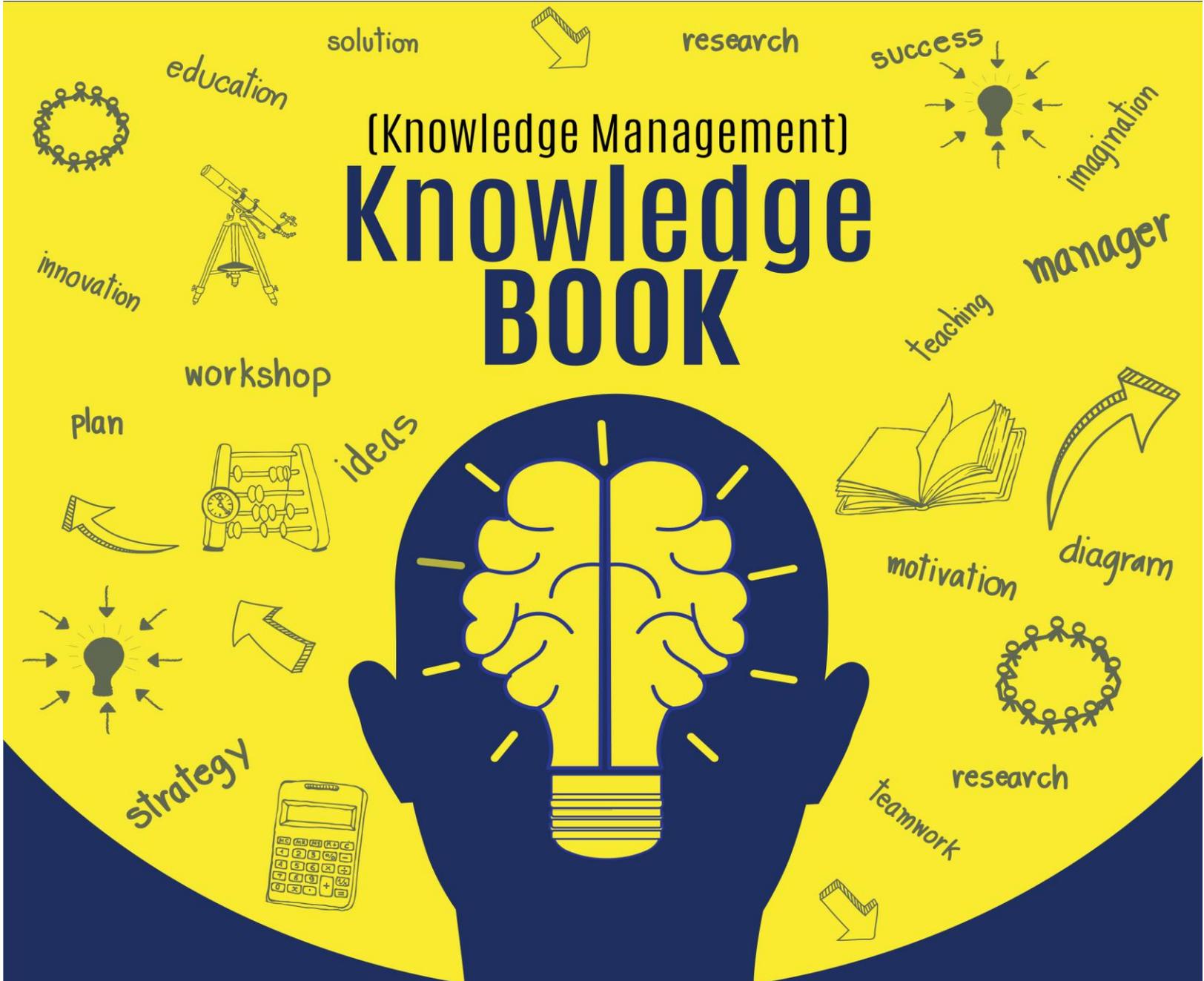


solution research success imagination manager teaching

(Knowledge Management)  
**Knowledge  
BOOK**



education innovation workshop plan ideas motivation diagram research teamwork

education

innovation

workshop

plan

ideas

motivation

diagram

research

teamwork

success

imagination

manager

teaching

research

education

innovation

workshop

plan

ideas

motivation

diagram

research

teamwork

success

imagination

manager

teaching

## Alkaline Fusion for Rare Earth Recovery

Penulis :

Khaironie Mohamed Takip, Dr Roshanorlyza Hazan, Wilfred Paulus, Norhazirah Azhar, Jacqueline Kones, Nur Aqilah Sapiee, Dr Julie Andrianny Murshidi

---

# RARE EARTHS PROCESSING

## In-House Study:

Pre-treatment, Cracking and Trisodium Phosphate Removal

---

KHAIRONIE MOHAMED TAKIP, DR. ROSHASNORLYZA HAZAN,  
WILFRED PAULUS, NORHAZIRAH AZHAR, JACQUELINE KONES, NUR  
AQILAH SAPIEE, DR. JULIE ANDRIANNY MURSHIDI

## TABLE OF CONTENTS

### CHAPTER 1: INTRODUCTION

<b>1.1</b>	<b>Introduction to Project.....</b>	<b>7</b>
<b>1.2</b>	<b>Monazite.....</b>	<b>7</b>
<b>1.2.1</b>	<b>Elemental content.....</b>	<b>8</b>
<b>1.3</b>	<b>Xenotime.....</b>	<b>9</b>
<b>1.3.1</b>	<b>Elemental content.....</b>	<b>10</b>
<b>1.4</b>	<b>Ilminite.....</b>	<b>11</b>
<b>1.4.1</b>	<b>Elemental content.....</b>	<b>13</b>
<b>1.5</b>	<b>Zircon.....</b>	<b>13</b>
<b>1.5.1</b>	<b>Elemental content.....</b>	<b>14</b>

### CHAPTER 2: METHOD

<b>2.1</b>	<b>Introduction to Method.....</b>	<b>17</b>
<b>2.2</b>	<b>RE Processing.....</b>	<b>17</b>
<b>2.2.1</b>	<b>Pre-Treatment.....</b>	<b>18</b>
<b>2.2.2</b>	<b>Cracking Process.....</b>	<b>21</b>
<b>2.2.3</b>	<b>TSP Removal.....</b>	<b>23</b>
<b>2.3</b>	<b>RE Characterization.....</b>	<b>25</b>
<b>2.3.1</b>	<b>X-ray Fluorescence (XRF).....</b>	<b>25</b>
<b>2.3.2</b>	<b>X-ray Diffraction (XRD).....</b>	<b>28</b>
<b>2.3.3</b>	<b>Field Emission Scanning Electron Microscope (FESEM). .....</b>	<b>30</b>

### CHAPTER 3: TRISODIUM PHOSPHATE

<b>3.1</b>	<b>Introduction to TSP.....</b>	<b>33</b>
<b>3.2</b>	<b>Application.....</b>	<b>35</b>

### CHAPTER 4: ACHIEVEMENT

<b>4.1</b>	<b>Introduction to Achievement.....</b>	<b>37</b>
<b>4.2</b>	<b>International linkage, Expertise and Training.....</b>	<b>37</b>
<b>4.3</b>	<b>Industrial Training Supervision.....</b>	<b>41</b>

<b>4.4</b>	<b>Innovation</b> .....	<b>43</b>
<b>4.5</b>	<b>Patent</b> .....	<b>44</b>
<b>4.6</b>	<b>Publication</b> .....	<b>45</b>

**AUTHOR'S BIOGRAPHY**

## LIST OF FIGURES

---

<b>Figure 1.</b> Monazite crystal from Brazil <sup>[1]</sup> .....	8
<b>Figure 2.</b> Malaysia monazite sand <sup>[2]</sup> .....	8
<b>Figure 3.</b> Xenotime crystal <sup>[4]</sup> .....	10
<b>Figure 4.</b> Xenotime Sand from Kinta Valley, Perak.....	10
<b>Figure 5.</b> Imenite ore from Bukit Beruntung, Selangor.....	12
<b>Figure 6.</b> Zircon ore from Dengkil, Selangor.....	14
<b>Figure 7.</b> Schematic of REEs Processing Step.....	17
<b>Figure 8.</b> Fritsch Pulverisette ball mill.....	20
<b>Figure 9.</b> Example of (a) sieve and (b) sieve shaker used in the laboratory.....	20
<b>Figure 10.</b> Monazite and NaOH mixture during cracking process.....	22
<b>Figure 11.</b> Hydrous REE oxide cake.....	22
<b>Figure 12.</b> TSP crystallization process.....	22
<b>Figure 13.</b> Mixture of xenotime and NaOH pellets before (left) and after (right) fusion cracking process. .....	23
<b>Figure 14.</b> Anhydrous REE oxide cake (left) and Trisodium Phosphate (TSP) solution (right).....	24
<b>Figure 15.</b> Monazite (left) and xenotime (right) processing flow.....	24
<b>Figure 16.</b> The Thermo ARL QUANT'X EDXRF (a), PANalytical ZETIUM WDXRF (b) and XRF sample preparation apparatus (c).....	25
<b>Figure 17.</b> XRF loose powder sample preparation method.....	27
<b>Figure 18.</b> XRF pellet sample preparation method.....	28
<b>Figure 19.</b> The PANalytical X'Pert PRO MPD PW 3040/60 diffractometer (a) and XRD sample preparation apparatus (b).....	29
<b>Figure 20.</b> The Carl Zeiss Gemini SEM 500 microscope (a) and FESEM sample preparation apparatus (b). .....	30
<b>Figure 21.</b> TSP powder from alkaline fusion cracking of xenotime.....	33
<b>Figure 22.</b> TSP from the studied process is called as TRINAF.....	34
<b>Figure 23.</b> Process flow of TSP generation.....	35
<b>Figure 24.</b> Common applications of TSP.....	36
<b>Figure 25.</b> Technical Cooperation Fellowship in Thailand.....	39
<b>Figure 26.</b> IAEA Expert Mission by Dr. Ahmed El Sayed Mady Hussein from Nuclear Material Authority of Cairo, Egypt.....	39
<b>Figure 27.</b> Third Research Coordination Meeting (RCM) for Coordinated Research Project (CRP).....	40
<b>Figure 28.</b> Last day presentation for Introductory Training Course on Radiation Technology and its Applications.....	40
<b>Figure 29.</b> Member state presentation during sharing experience session.....	40
<b>Figure 30.</b> IAEA Fellowship Training on Mineral Characteristic and Ore Processing.....	41
<b>Figure 31.</b> Mineral Processing Training in Ipoh, Perak.....	41
<b>Figure 32.</b> Briefing on XRF sample preparation to industrial students.....	42
<b>Figure 33.</b> Awards giving ceremony during the innovation competitions.....	44
<b>Figure 34.</b> Total number of publications.....	45

## LIST OF TABLES

---

<b>Table 1:</b> Elemental analysis of monazite from Kinta Valley, Perak <sup>[3]</sup> .....	9
<b>Table 2:</b> Elemental analysis of xenotime from Kinta Valley, Perak <sup>[5]</sup> .....	11
<b>Table 3:</b> The elemental analysis of ilmenite from Bukit Beruntung, Selangor. ....	13
<b>Table 4:</b> Elemental analysis of zircon from Dengkil, Selangor, Malaysia.....	15
<b>Table 5 :</b> Elemental analysis result of monazite from beneficiation process .....	19
<b>Table 6:</b> Elemental content of TSP from alkaline fusion cracking of xenotime <sup>[5]</sup> .....	34
<b>Table 7:</b> List of linkage, expertise and training. ....	38
<b>Table 8:</b> List of Industrial training students. ....	42
<b>Table 9:</b> Malaysia REE and its mineral innovation achievement. ....	43
<b>Table 10:</b> List of patents filed. ....	44

# CHAPTER 1: Introduction

---

## 1.1 Introduction to Project

This chapter gives an overview of the major and minor local rare earth (RE) minerals which are being studied in Malaysian Nuclear Agency (Nuklear Malaysia). Since the minerals discussed are mined locally, differences in the rare earth element (REE) content may arise because the same metal ore deposits also occur in several other parts of the world. The explanation of RE minerals in this chapter is primarily intended to show that the REE and by-product from these minerals appear to have opportunities for industrial use.

At this present, the principal ore minerals for REE are monazite and xenotime, and the secondary are from ilmenite and zircon. The REE are a group of 17 chemically similar elements consisting of the lanthanides, Yttrium (Y) and Scandium (Sc). From the lanthanide series, Lanthanum (La), Cerium (Ce), Praseodymium (Pr), Neodymium (Nd) and Samarium (Sm) are classified as LREE meanwhile Europium (Eu), Gadolinium (Gd), Terbium (Tb), Dysprosium (Dy), Holmium (Ho), Erbium (Er), Thulium (Tm) and Ytterbium (Yb) are classified as HREE. Among these REE, there are four elements (Ce, La, Nd and Y) that planned to be recovered and extracted from the following minerals which are currently on comprehensive laboratory scale studies.

## 1.2 Monazite

In Malaysia, monazite and xenotime are minerals phosphates that are present as by-product of tin mining processing. Monazite chemical formula  $CePO_4$  was generalized by Breithaupt in 1829. Physically, it is a brown crystalline mineral, as displayed in Fig. 1, consisting of REE and associated with naturally occurred radioactive material (NORM). Malaysian monazite sand (Fig. 2) with resinous luster is produced from heavy mineral concentrates. It is then separated out by specific gravity, magnetic processes and several other beneficiation procedures prior to carrying out further RE recovery and extraction process.



**Figure 1.** Monazite crystal from Brazil <sup>[1]</sup>.



**Figure 2.** Malaysia monazite sand <sup>[2]</sup>.

### 1.2.1 Elemental content.

The REE contained in local monazite include La, Ce, Nd, Pr, Sm, Gd, Dy and Tm as the compositions are listed in Table 1. These are mostly the LREEs besides thorium (Th) and / or uranium (U), but the amounts in monazite are generally too low to be extracted as a valuable by-product.

**Table 1:** Elemental analysis of monazite from Kinta Valley, Perak [3].

Analyte	Wt.%
Ce	32.0
La	12.2
Nd	11.7
Y	4.31
Pr	3.47
Sm	2.59
Gd	1.91
Dy	1.03
Er	0.66
Tm	0.39

Monazite forms during the igneous rock crystallization and during the clastic sedimentary rock metamorphism. When these rocks weather, monazite is one of the more resistant minerals and becomes concentrated in the weathering debris. The soils and sediments found near a weathering outcrop can have a higher monazite concentration than the source rock. Because of that, all monazite mining is focused on placer deposits as they are easier to mine and the monazite is often present in higher concentration.

### 1.3 Xenotime

Xenotime is another valuable RE mineral deposits that similar to monazite. A brown to brownish yellow crystalline mineral with chemical formula  $YPO_4$  is to describe the major component of yttrium orthophosphate (Fig. 3). Alike to monazite, xenotime has been commercially extracted from alluvial deposits as a by-product or locally known as amang, from the exploration of cassiterite, rutile, zircon and ilmenite. It is discovered most locally in Kinta Valley of the Perak State in Peninsular Malaysia (Fig. 4).



**Figure 3.** Xenotime crystal <sup>[4]</sup>.



**Figure 4.** Xenotime Sand from Kinta Valley, Perak.

### 1.3.1 Elemental content

Comparing in term of elements content, phosphorus (P) and HREE particularly yttrium content is higher in xenotime than that in monazite, however, the content of NORM is much lower (Table 2).

**Table 2:** Elemental analysis of xenotime from Kinta Valley, Perak [5].

Composition	Wt. %
Y <sub>2</sub> O <sub>3</sub>	35.17
P <sub>2</sub> O <sub>5</sub>	28.83
ThO <sub>2</sub>	0.63
U <sub>3</sub> O <sub>8</sub>	0.57
Heavy Rare Earths ( Dy <sub>2</sub> O <sub>3</sub> , Gd <sub>2</sub> O <sub>3</sub> , Tb <sub>2</sub> O <sub>3</sub> , Er <sub>2</sub> O <sub>3</sub> )	10.18
Light Rare Earths (CeO <sub>2</sub> , La <sub>2</sub> O <sub>3</sub> , Nd <sub>2</sub> O <sub>3</sub> )	5.97
Others (SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> , HfO <sub>2</sub> , TiO <sub>2</sub> , BaO, ZrO <sub>2</sub> , PbO, SO <sub>3</sub> , As <sub>2</sub> O <sub>3</sub> )	3.69

In general, the decomposition of xenotime concentrate is more difficult than monazite. The methods adopted for decomposing its concentrate mostly are the ones with concentrated sulfuric acid, alkali solution with high pressure and high temperature, such as alkali fusion method which is currently applied in the on-going studies of REE extraction from xenotime at Nuklear Malaysia.

## 1.4 Ilmenite

The primary for titanium (Ti) ore mineral, ilmenite was named after the Ilmen Mountains in the south Urals [6]. Malaysia's tin mining industries produce numerous varieties of spin-off product such as xenotime, monazite, zircon and ilmenite. Fig. 5 shows ilmenite ore collected from Bukit Beruntung, Selangor.



**Figure 5.** Ilmenite ore from Bukit Beruntung, Selangor.

Ilmenite is one of the important mineral aids Malaysia's economy growths. Alternatively, ilmenite becomes a crucial mineral in the oxygen carrier process compare to the other minerals (olivine, hematite, biotite and cuprite). Marvelous mechanical resistance and great thermal behaviour played a crucial cause for this process. Rather than its desirable possession, ilmenite likewise one of the lowest price minerals compares to other minerals resources. Major ilmenite content is titanium dioxide ( $\text{TiO}_2$ ) which can be utilized in numerous industries, for examples paint industry and iron oxide ( $\text{Fe}_2\text{O}_3$  or  $\text{Fe}_3\text{O}_4$ ) for catalytic and magnetic industries<sup>[7]</sup>.

### 1.4.1 Elemental content

**Table 3:** The elemental analysis of ilmenite from Bukit Beruntung, Selangor.

Analyte	Wt. %
Ti	36.29
Fe	29.41
Si	20.79
Al	5.87
Mn	2.93
P	1.08
Sn	0.78
Zr	1.04
K	0.50
S	0.35
Ce	0.26
I	0.21
Hf	0.16
Nb	0.12
Y	0.11

## 1.5 Zircon

Most of zircon minerals in Malaysia are found as the by-product of among alluvial tin mining in Hulu Kinta, Perak and Dengkil, Selangor<sup>[8]</sup>.

Zircon is the zirconium main mineral. It is a silicate mineral product ( $ZrSiO_4$ )<sup>[9]</sup>. In particular Malaysian zircon are gained from among and its related minerals contain high level of impurities. The properties of these mineral vary from individual source region to another because of the

variances in their impurities content, shape and particle dimension. Generally, zircon has been categorized keen on the finest and typical grades based on the ferum (Fe) and titanium (Ti) impurities in the mineral. The finest quality grade zircon has lesser impurity content compared to the normal grade zircon <sup>[10]</sup>. Fig. 6 shows the zircon minerals with some inclusion inside.

Naturally occurring radioactive materials (NORM) such as Th, U and radium (Ra) are discovered in numerous minerals and waste matter produced by the minerals downstream process. Some minerals found to have these NORM from the spin-off of the tin mining activity are monazite, xenotime, zircon, ilmenite and rutile.



**Figure 6.** Zircon ore from Dengkil, Selangor.

### 1.5.1 Elemental content

Lot of Fe in zircon may cause to brown stains and it is not preferable particularly for white ceramic piece. The existence of high content Fe in zircon may be because to the clay minerals presence such as goetite, gibbsite and kaolinite on the zircon surface and the impurity of other iron-bearing minerals such as ilmenite. Ilmenite frequently coexistence with the zircon mineral naturally.

All zircons bear several radioactive contaminants because of the existence of Th, U and Ra and their respective decay products in the zircon crystalline structure including potassium-40. Uranium and thorium replace  $Zr^{4+}$  in the mineral via an internal process called zirconium isomorphous replacement. Table 4 shows the elemental analysis of zircon mineral collected from Dengkil, Selangor, Malaysia using XRF analysis techniques.

**Table 4:** Elemental analysis of zircon from Dengkil, Selangor, Malaysia.

Analyte	Wt. %
P	35.36
Zr	31.14
Si	17.63
Ba	5.52
Ce	3.59
Nd	2.78
Hf	1.78
Sn	0.85
Th	0.48
Y	0.38
Ca	0.27
Cs	0.16

## REFERENCES

- [1] The Arkenstone. (n.d). *Monazite crystal: An exceptionally large monazite crystal, approximately two inches across, collected in Brazil* [Specimen and photo]. Retrieved on 26 May 2021. <https://www.irocks.com/>
- [2] Hobart, M. K., Monazite: A rare earth phosphate mineral mined from placer deposits for its rare earth and thorium content. Geology.com. Retrieved on 26 May 2021. <https://geology.com/minerals/monazite.shtml>.
- [3] Khaironie, M.T., Jacqueline, K., Shahida, S. (2020, November). *Selective Recovery of Rare Earth Elements from Malaysian Monazite Concentrate* [Poster Presentation]. Seminar R&D Nuklear Malaysia 2020. Kajang, Selangor, Malaysia

- [4] Minerals.net. (n.d). *The xenotime mineral series* [Specimen and photo]. Retrieved on 26 May 2021. <https://www.minerals.net/mineral/xenotime.aspx>.
- [5] Khaironie M.T., Jacqueline K., Roshasnorlyza H., Norhazirah A., Nur A.S. and Wilfred P. (2019, May-23). *Decomposition of xenotime concentrate using alkaline fusion method* [Paper Presentation]. ALTA Conferences 2019. Perth, WA, Australia.
- [6] Gázquez, M. J., Bolívar, J. P., Garcia-Tenorio, R. and Vaca, F. (2014). A Review of the Production Cycle of Titanium Dioxide Pigment. *Materials Sciences and Applications* 5: 441-458.
- [7] Hazan, R., Muhamad Yusop, M. A, Paulus, W. and Mohamed Takip, K. (2020). Obtaining TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> from Ilmenite via Alkaline Fusion Method. *Materials Science Forum* 1010: 385-390.
- [8] Minerals and Geoscience Department Malaysia, Ministry of Natural Resources and Environment Malaysia (2014, December 15). *Malaysian Mineral Yearbook 2013. Commodity Review: Titanium and Commodity Review: Zircon*.
- [9] Finch, R. J and Hanchar, J. M. (2003). Structure and Chemistry of Zircon and Zircon-Group Minerals. *Reviews in Mineralogy and Geochemistry* 53: 1 - 25.
- [10] Meor Sulaiman, M. Y. (2002). *Study in determining optimum conditions for reducing NORM in Malaysian zircon mineral* [Paper Presentation]. International Nuclear Conference 2002: Global Trends and Perspectives. International Atomic Energy Agency, Vienna, Austria

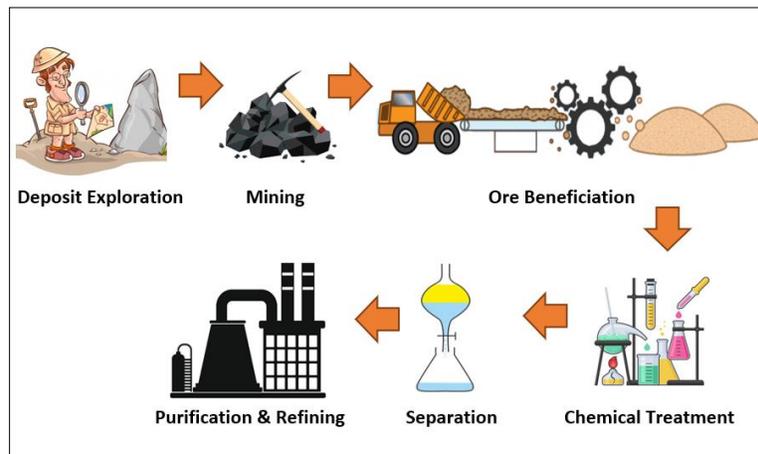
# CHAPTER 2: Method

## 2.1 Introduction to Method

This chapter discusses how exactly the study of Malaysia REE and its mineral were conducted. The samples were prepared and were characterized using several processes and analysis techniques. As there are various studies still ongoing, the explanation of mineral at this stage will only be referring to several studies that related to monazite and xenotime. In the same way, there are only three earlier approaches in RE processing namely pre-treatment, cracking and trisodium phosphate (TSP) removal were discussed. Subsequent processes will be updated occasionally. Three main characterization techniques namely X-ray Fluorescence (XRF), X-ray diffraction (XRD) and Field Emission Scanning Electron Microscope (FESEM) were used in order to study their elemental, phase and microstructural properties.

## 2.2 RE Processing.

In general, there are six main steps in REEs processing activities consisting of deposit exploration, mining, ore beneficiation, chemical treatment, separation, refining, and purification <sup>[1]</sup> as shown in Fig. 7.



**Figure 7.** Schematic of REEs Processing Step.

The entire process begins with the search and discovery of potential mineral deposits for mining. Exploration activities usually involves samples collection for extensive study with geochemical analysis. The following stage is mining which has four main methods: underground, open surface (pit), placer, and in-situ. The opted method is depending on targeted type of resource for extraction, its location and profit capacity. The third step is ore beneficiation in which physical and chemical separation methods are used to remove gangue mineral to produce high grade RE mineral concentrate. Gravity, flotation, and magnetic separations are the common types of physical separation while froth flotation, leaching, and electrowinning are among types of chemical separation used to remove some properties that cannot rely on physical separation method. The last three steps in REEs processing involves hydrometallurgical methods for extracting REEs. The fourth step requires chemical treatment which is known as cracking or decompose process in modifying REEs structures. Acid baking and alkaline cracking are the most common treatments, however the alkaline has been used more due to its ability in removing sulphates as well as phosphates and simplifying thorium refining step <sup>[2]</sup>. The fifth step requires separation techniques for example solvent extraction and ion exchange in which RE mineral concentrate is converted into RE compound as an intermediate product for the subsequent process. Finally, the purification and refining process is carried out in the last step for the production of either individual REEs or other compounds.

### **2.2.1 Pre-Treatment.**

Raw material or mineral supplied for our study have been undergone earlier beneficiation process by mineral suppliers. The undesired impurities have been removed along the process to enhance the concentration of REE. One of the elemental analysis results provided by the supplier is shown in Table 5 <sup>[3]</sup>. The generated REE concentrate will go through a physical pre-treatment in the laboratory prior to further chemical treatment or cracking process. The treatment will include both milling and sieving processes. It is beneficial for increasing total contact of surface

area as smaller solid particle of REE concentrate will result in greater surface area to react with chemical reagent <sup>[4]</sup>. This reaction will make a homogenous matrix during cracking process <sup>[5]</sup>.

**Table 5 :** Elemental analysis result of monazite from beneficiation process

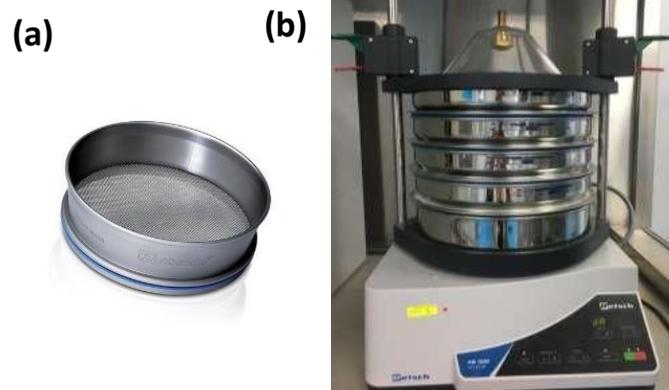
<b>Element</b>	<b>Before beneficiation (%)</b>	<b>After beneficiation (%)</b>
<b>Ce</b>	30.69	47.41
<b>P</b>	21.77	6.40
<b>La</b>	13.53	35.28
<b>Nd</b>	12.09	8.51
<b>Si</b>	6.70	0.76
<b>Th</b>	4.62	0.34
<b>Al</b>	3.82	0.75
<b>Zr</b>	2.38	0.08
<b>Y</b>	2.37	0.27
<b>Ca</b>	0.65	0.06
<b>S</b>	0.43	0.02
<b>Sn</b>	0.18	0.03
<b>Yb</b>	0.17	-
<b>U</b>	0.17	-
<b>W</b>	0.12	0.02
<b>Ta</b>	0.11	0.04
<b>Hf</b>	0.10	-
<b>Zn</b>	0.06	0.01
<b>Se</b>	0.04	0.01

A planetary ball mill (Fritsch Pulverisette) as shown in Fig. 8, is used to mill the concentrate to reduce its particle sizes. This planetary ball mill is able to rotate both vertically and horizontally rotation in a simultaneous way.



**Figure 8.** Fritsch Pulverisette ball mill.

Sieving is the following process after milling. The milled concentrate is dry sieved on a sieve machine as shown in Fig. 9, through several stages of sieving with different mesh sizes.



**Figure 9.** Example of (a) sieve and (b) sieve shaker used in the laboratory.

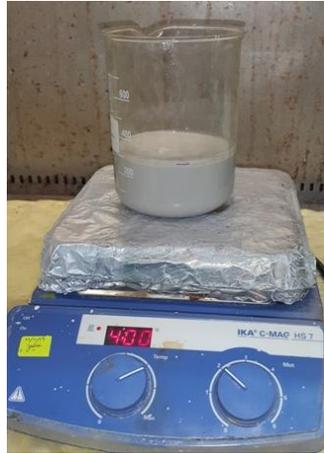
The sieve mesh sizes are arranged in descending order ( $< 20 \mu\text{m}$ ,  $20 - 45 \mu\text{m}$ ,  $45 - 90 \mu\text{m}$ ,  $90 - 180 \mu\text{m}$ ) according to the desired particle size. The optimum particle size of REE concentrate for our study is  $45 \mu\text{m}$ . Upon sieving, the collected concentrate is ready for cracking process.

### 2.2.2 Cracking Process

Cracking is a chemical treatment in which the structure of RE concentrate is modified either in alkaline or acid treatment to facilitate REE dissolution in weak acid throughout the next process that is leaching<sup>[6]</sup>. Most studies worldwide employed inorganic acids such as sulfuric acid ( $\text{H}_2\text{SO}_4$ ), hydrochloric acid (HCl) and nitric acid ( $\text{HNO}_3$ ) in acid treatment, while sodium hydroxide (NaOH) and sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) are commonly used in alkaline treatment<sup>[7]</sup>.

Alkaline treatment is the common route for RE cracking process due to its ability in recovery valuable crystalline trisodium phosphate ( $\text{Na}_3\text{PO}_4$ ) or widely known as TSP<sup>[8, 9]</sup>. In our previous and current studies<sup>[10 - 11]</sup>, two different techniques from alkaline treatment route namely alkaline cracking and alkaline fusion cracking were used for decomposing monazite and xenotime concentrates respectively. Alkaline cracking differs from alkaline fusion in the usage of NaOH solution in a low temperature, whereas alkaline fusion requires solid NaOH in a much higher temperature to decompose RE concentrate<sup>[11]</sup>. However, the main drawbacks of alkaline cracking were requiring high-grade REE concentration (>90%) and fine grinded monazite (325 mesh) as its raw material<sup>[14]</sup>.

In alkaline cracking process, the finely grinded monazite concentrate was digested in sodium hydroxide (NaOH) solution. The mixture, as shown in Fig. 10, was continuously stirred until the formation of homogeneous slurry.

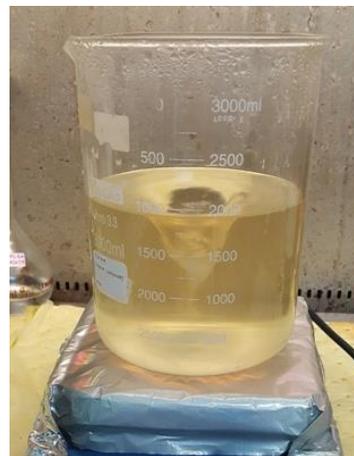


**Figure 10.** Monazite and NaOH mixture during cracking process.

The generated slurry was diluted with hot distilled water and filtered to collect the hydrous REE oxide cake (Fig. 11). The filtrate will undergo crystallizing process as shown in Fig. 12 to form crystalline TSP. The obtained oxide cake was washed thoroughly until pH constant and dried overnight before proceed to the acid leaching <sup>[12]</sup>.



**Figure 11.** Hydrous REE oxide cake.



**Figure 12.** TSP crystallization process.

In contrast to monazite, it is not crucial to grind fine xenotime concentrate to decompose in alkaline fusion cracking process <sup>[10]</sup>. Xenotime concentrate and NaOH pellets was well mixed in an alumina crucible before placing the solid mixture into a furnace to undergo the fusion process <sup>[10, 11, 13]</sup>.



**Figure 13.** Mixture of xenotime and NaOH pellets before (left) and after (right) fusion cracking process.

The fusion product as shown in Fig. 13, was put to cool down at room temperature before adding distilled water to soak overnight. The unstiffen product was then diluted with distilled water and heated on a hot plate for several hours until homogeneous slurry formed.

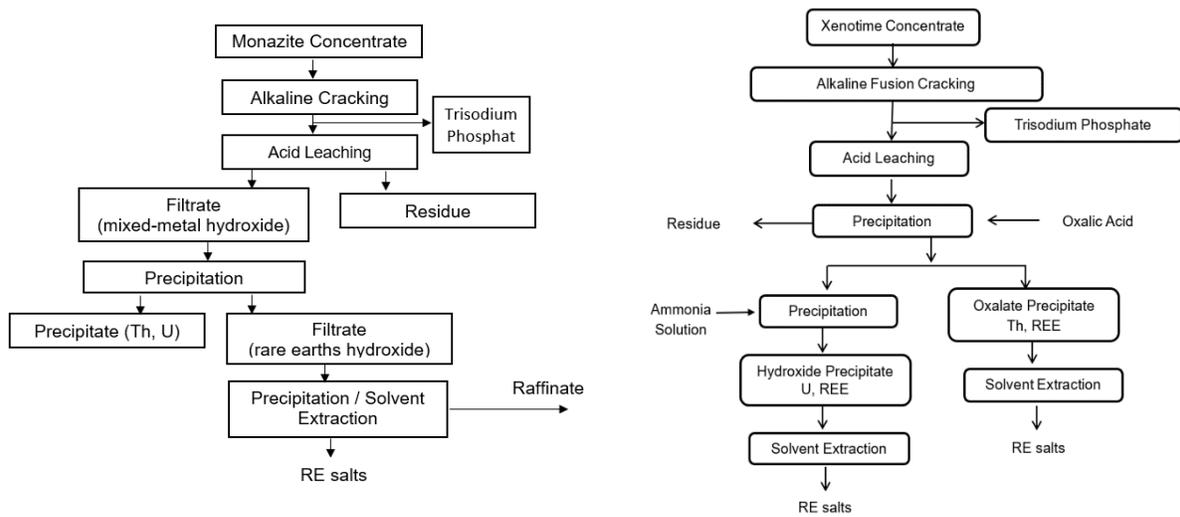
### 2.2.3 TSP Removal

The generated slurry was vacuum filtered to separate hydrous REE oxide cake from TSP filtrate. It is a necessity to remove TSP at this stage in order to avoid formation of RE complexes that could obstruct REE recovery in the following processes. The hydrous REE oxide cake as shown in Fig. 14, was dried overnight before carrying out further chemical treatment. TSP solution as displayed in Fig. 14 was collected and stored for further study.



**Figure 14.** Anhydrous REE oxide cake (left) and Trisodium Phosphate (TSP) solution (right).

Basic monazite and xenotime processing flowsheets for these studies are shown in following Fig. 15.



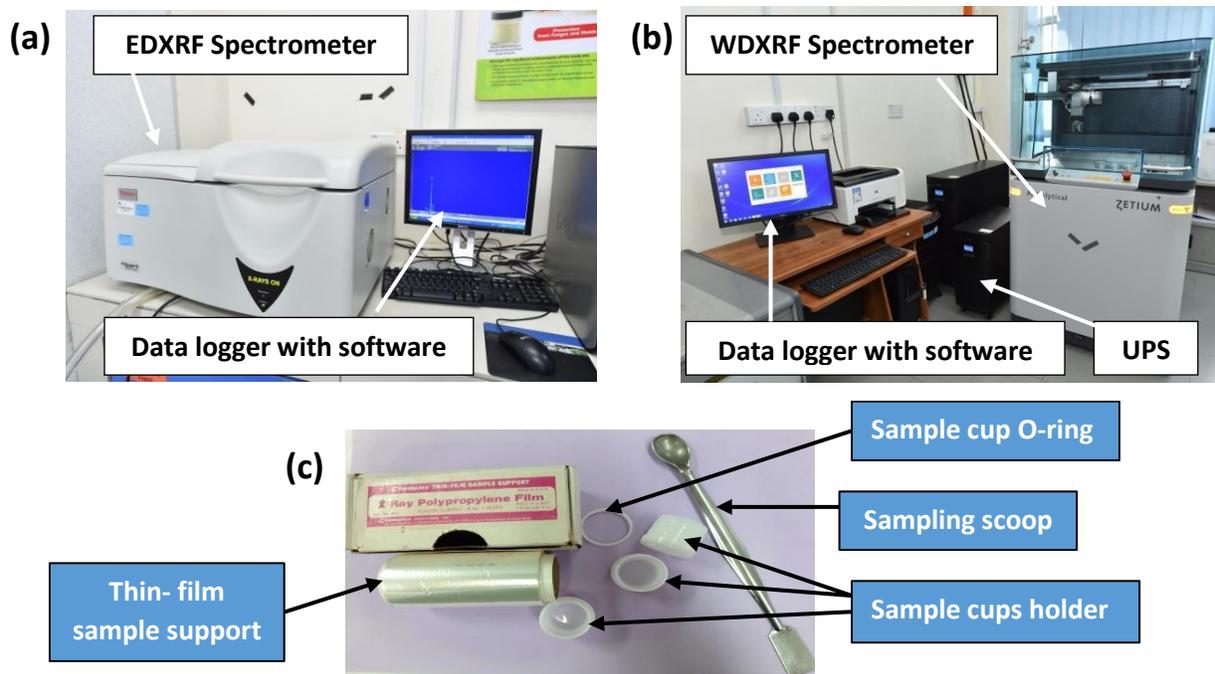
**Figure 15.** Monazite (left) and xenotime (right) processing flow.

## 2.3 RE Characterization

### 2.3.1 X-ray Fluorescence (XRF)

XRF is a widely used technique for elemental analysis due to its rapid analysis ability. It is more convenient technique with simple or no sample preparation at all. It can produce better results with easier development of correct calibration curve using few standards which can be applied to most of its analysis.

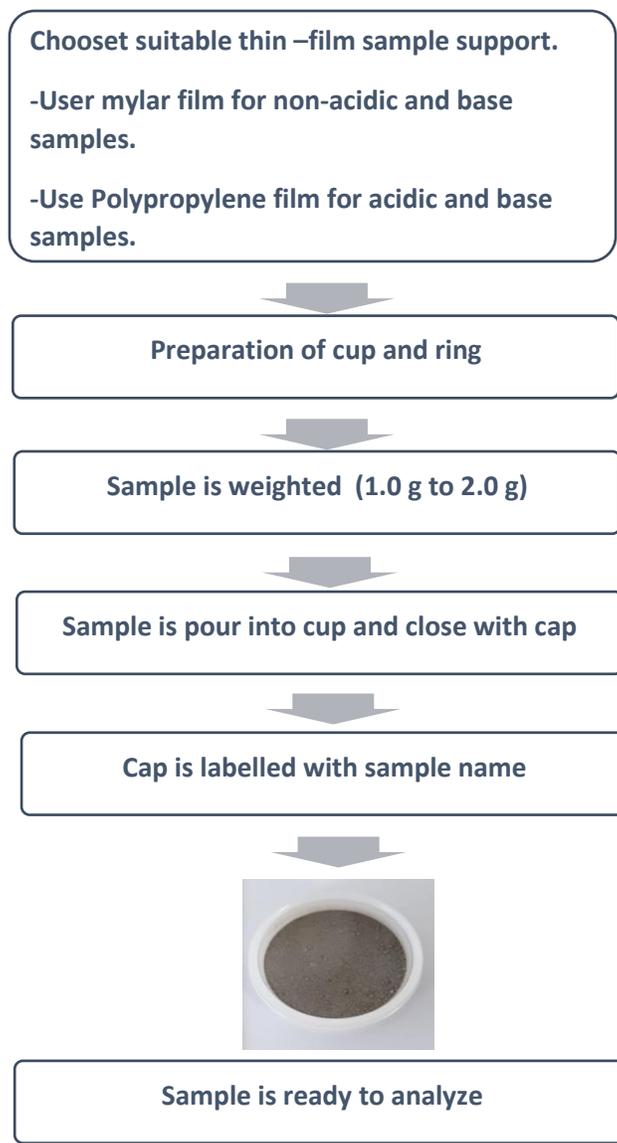
There are two types of XRF used for REE and its mineral studies at Nuklear Malaysia. They are energy-dispersive x-ray fluorescence (EDXRF) and wavelength-dispersive x-ray fluorescence (WDXRF) as shown in Fig. 16. Both XRFs were employed to identify wide range of elements from Sodium (Na) to Uranium (U) and also measure their concentration in the samples. The basic components of all type of XRF spectrometers are a radiation source (X-ray tube), a sample and a detection system (detector).



**Figure 16.** The Thermo ARL QUANT'X EDXRF (a), PANalytical ZETIUM WDXRF (b) and XRF sample preparation apparatus (c).

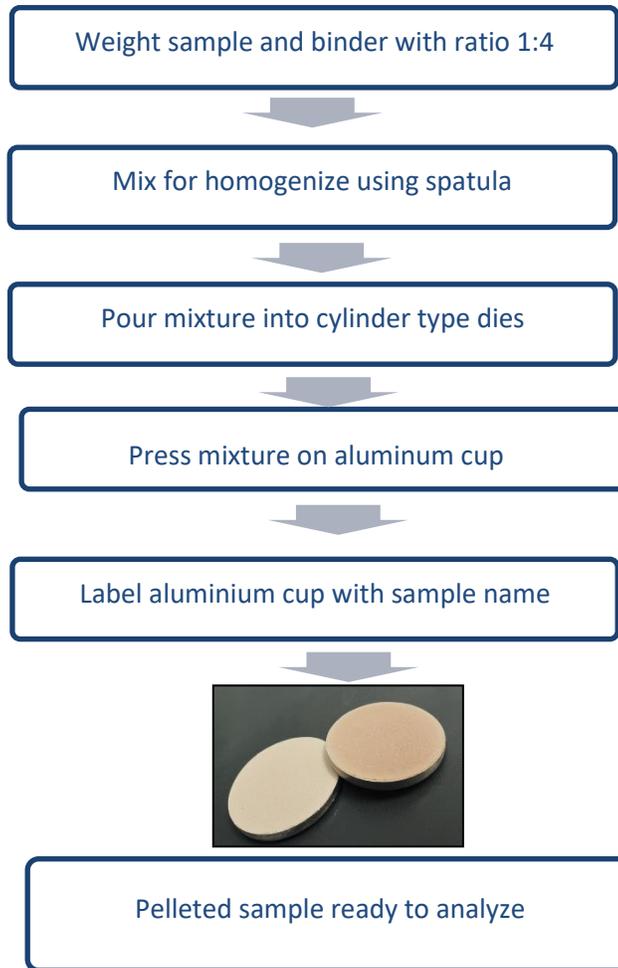
EDXRF is based on the principle that individual atoms, when excited by an external energy source, emit X-ray photons of a characteristic energy. The radiation intensity of each element signal (energy), which is proportional to the concentration of the element in the sample, is recalculated internally from a stored set of calibration curves and can be shown directly in concentration units. In WDXRF spectrometers, the X-ray tube acting as a source irradiates a sample directly, and the fluorescence coming from the sample is measured with a wavelength dispersive detection system. The characteristic radiation coming from each individual element can be identified using analyzing crystals which separate the X-rays based on their wavelength, or conversely their energies. Such an analysis can either be done by measuring the X-ray intensity at different wavelengths one after the other (sequential) or in fixed positions, measuring the X-ray intensities at different wavelengths all at the same time (simultaneous). In typical measurement condition, the EDXRF with an air cooled X-ray tube with Rhodium (Rh) target was used under normal atmospheric air. The exited X-rays from bombarded samples were detected by a Si(Li) detector with a resolution of 155 eV for 5.9 keV X-rays.

The measurement were performed under the following conditions: X-ray generator voltage of 5 - 40 kV and current of 0.84 - 1.90 mA, measuring time 30 s and dead time at 50%. All of XRF spectra were quantitatively analyzed with the use of the Thermo's Win Trace software version 4. The calibration curve of each element was developed by using soil standards (NIST SRM) to perform the quantitative analysis. In these studies, WDXRF was used when REE elements could not be identified using EDXRF.



**Figure 17.** XRF loose powder sample preparation method.

Good sample preparation is important in order to have high-quality elemental analysis using XRF. Whichever samples form are being assessed, finding the right approach to sample preparation for XRF is the first, and one of the most important steps in achieving accurate and reproducible results. Samples preparation method for both EDXRF and WDXRF used in these studies were shown in Fig. 17 and Fig. 18. All samples were analyzed as bulk sample.



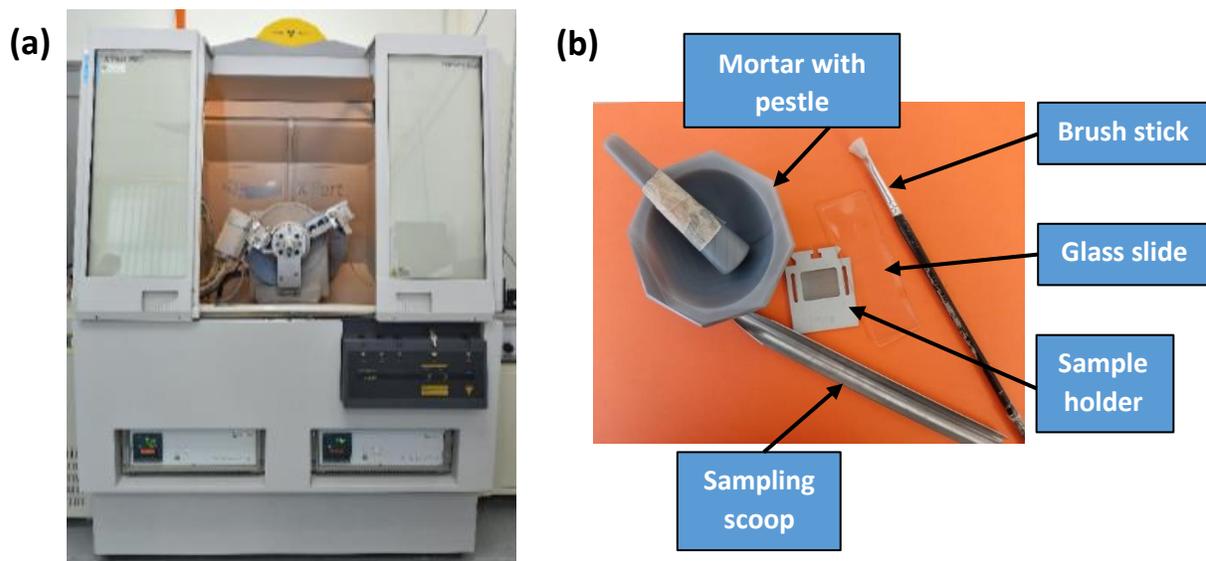
**Figure 18.** XRF pellet sample preparation method.

### 2.3.2 X-ray Diffraction (XRD).

XRD is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode of an X-ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of these x-rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ( $n\lambda=2d \sin \theta$ ). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample.

The characteristic x-ray diffraction pattern generated in a typical XRD analysis provides a unique “fingerprint” of the crystals exist in the sample. When properly interpreted, by comparison with standard reference patterns and measurements, this fingerprint allows identification of the crystalline sample.

The measurement were performed under the following conditions: PANalytical X’Pert PRO MPD PW 3040/60 diffractometer at Nuklear Malaysia was used for phase identification of crystalline REE and its mineral samples as shown in Fig. 19. Cu K $\alpha$ 1 radiation source ( $\lambda = 1.54060 \text{ \AA}$ ) with a  $2\theta$  range of  $20^\circ$  to  $80^\circ$  with a step size of  $0.0334^\circ$  under a voltage of 40 kV and a current of 30 mA were applied. The diffraction data was analyzed using PANalytical’s X’Pert HighScore Plus Version 2.2b (2.2.2) software. Diffraction pattern in samples was observed and matched to calculated profile in ICSD (Inorganic Crystal Structure Database) database Version 2007-2 from FIZ-NIST.

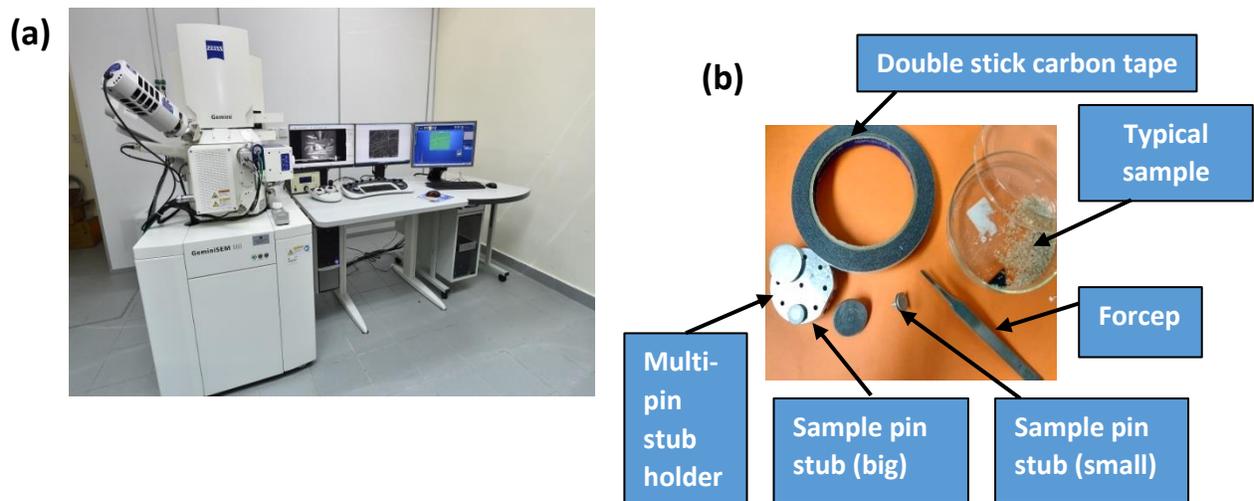


**Figure 19.** The PANalytical X’Pert PRO MPD PW 3040/60 diffractometer (a) and XRD sample preparation apparatus (b).

### 2.3.3 Field Emission Scanning Electron Microscope (FESEM).

A FESEM is microscope that works with electrons instead of light. These electrons are liberated by a field emission source and accelerated in a high electrical field gradient. These so-called primary electrons are focused and deflected by electronic lenses within the high vacuum column to produce a narrow scan beam that bombards the object. As a result, secondary electrons are emitted from each spot on the object. The angle and velocity of these secondary electrons relates to the surface structure of the object. A detector catches the secondary electrons and produces an electronic signal. This signal is amplified and transformed to a video scan-image that can be seen on a monitor or to a digital image that can be saved and processed further. FESEM is more suitable to be used in nanoscience and nanomaterial studies.

The measurement was performed under the following conditions: Microstructural observations of REE and its mineral samples were conducted on a Carl Zeiss Gemini SEM 500 FESEM at Nuklear Malaysia using a secondary electron detector whilst operating at 15 kV (Fig. 20). The microscope was coupled with an Oxford energy dispersive X-Ray spectrometer (EDS) for elemental analysis. Samples were not coated prior to imaging in the FESEM. Samples were prepared by sprinkling the powders onto carbon tape fixed to a FESEM sample holder.



**Figure 20.** The Carl Zeiss Gemini SEM 500 microscope (a) and FESEM sample preparation apparatus (b).

## REFERENCES

- [1] Mat Suli, L., Ibrahim, W. H. W., Abdul Aziz, B., Deraman, M. R., & Ismail, N. (2017). *A Review of Rare Earth Mineral Processing Technology*. Chemical Engineering Research Bulletin. 19, 20-35.
- [2] Sadri, F., Nazari, A. M., & Ghahreman, A. (2017). *A review on the cracking, baking and leaching processes of rare earth element concentrates*. *Journal of Rare Earths*, 35(8), 739–752.
- [3] End of Report for Thorium Flagship Project DSTIN (FP0214D052-01) “*Development of Innovative Nuclear Reactor Technology (With the Spin-Off) Based on Thorium*” (2014 – 2019).
- [4] Roshasnorlyza, H., Jacqueline, K., Khaironie, M. T., Nur Aqilah, S., Norhazirah, A., & Wilfred, P. (2019). *Recovery of Thorium and Rare Earth Element (REE) from Different Particle Size of Xenotime Mineral*. *J of Nuc and Related Tech*. Vol 16(2).
- [5] Md. Jaffary, N. A., Khoo, K. S., Mohammed, N. H., Yusof, M. A. W., & Mohd Fadzil, S. (2019). *Malaysian monazite and its processing residue: Chemical Composition and Radioactivity*. *J of Radioanalytical and Nuc Chem*. 322, 1097-1105.
- [6] Sadri, F., Nazari, A. M., & Ghahreman, A. (2017). *A review on the cracking, baking and leaching processes of rare earth element concentrates*. *Journal of Rare Earths*, 35(8), 739-752. doi:10.1016/s1002-0721(17)60971-2.
- [7] Kumari, A., Panda, R., Jha, M. K., Kumar, J. R., & Lee, J. Y. (2015). *Process development to recover rare earth metals from monazite mineral: A review*. *Minerals Engineering*, 79, 102–115.
- [8] Gupta, C.K., & Krishnamurthy, N. (2015) *Extractive Metallurgy of Rare earths*; CRC Press: Boca Raton, Florida
- [9] Zhu, Z., Pranolo, Y., Cheng, Y.C. (2015). *Separation of Uranium and Thorium from Rare Earths for Rare Earth Production – A review*. *J of Minerals Engineering*, 77, 185-196.
- [10] Meor Yusoff, M. S., Khaironie, M.T., Nursaidatul, K., Ahmad Khairulikram, Z., & Nur Aqilah, S. (2016) *An Alternative Alkaline Fusion Process for the Production of Heavy Rare Earth, Thorium, Uranium and Phosphate from Malaysian Xenotime*. *Recent Advances in Environment, Ecosystems and Development*. 163-167
- [11] Jacqueline, K., Norhazirah, A., Nur Aqilah, S., & Khaironie, M T., (2019). *Alkaline Fusion of Malaysian Monazite and Xenotime for the Separation of Thorium and Uranium*. *Jurnal Sains Nuklear Malaysia*. 31(1), 37 – 41
- [12] Khaironie, M.T., Jacqueline, K., & Shahida, S. (2020). *Selective Recovery of Rare Earth Elements from Malaysian Monazite Concentrate*. Poster presentation on Seminar R&D Nuklear Malaysia.
- [13] Norhazirah, A., Khaironie, M. T., Jaqueline, K., Nur Aqilah, S., & Ahmad Khairulikram, Z. (2019). *Cracking of Malaysia Xenotime Concentrate in Sodium Hydroxide Using Fusion Technique*. *J Sains Nuklear Malaysia*. 31(1), 11-17.

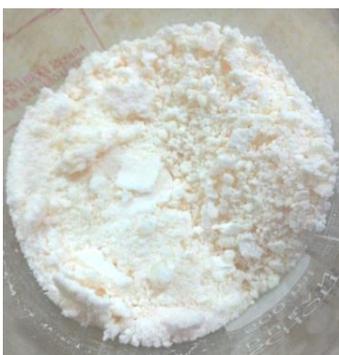
[14] Amer, T.E., Abdella, W.M., Wahab, G.M., & El-Sheikh, E.M. (2013). *A Suggested Alternative Procedure for Processing of Monazite Mineral Concentrate*. International Journal of Mineral Processing, 106,125.

# CHAPTER 3: Trisodium Phosphate

---

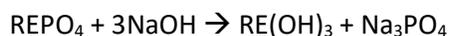
## 3.1 Introduction to TSP

TSP is a by-product obtained from cracking process. It is a non-toxic white crystal that has pH of 12 to 14 in solution (Fig. 21). It is highly soluble in water and forms an alkaline solution when mixed.



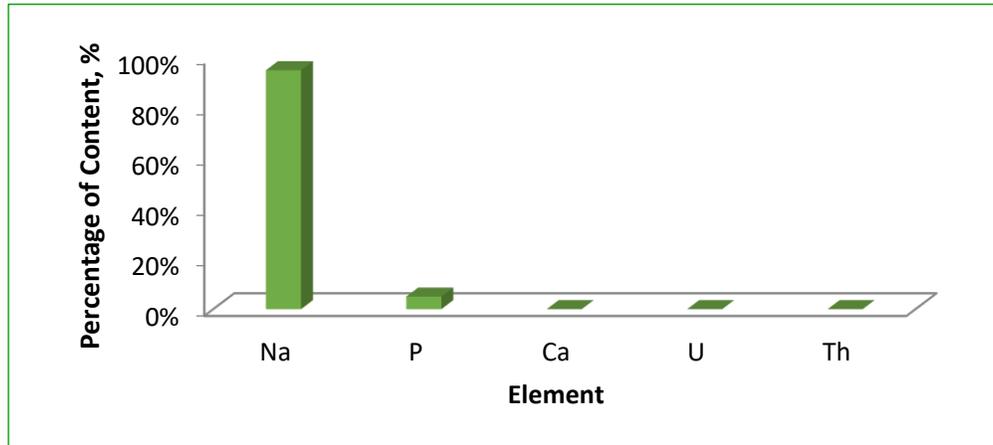
**Figure 21.** TSP powder from alkaline fusion cracking of xenotime.

During the alkaline treatment of RE mineral by using NaOH, the production of TSP is in the following equation:



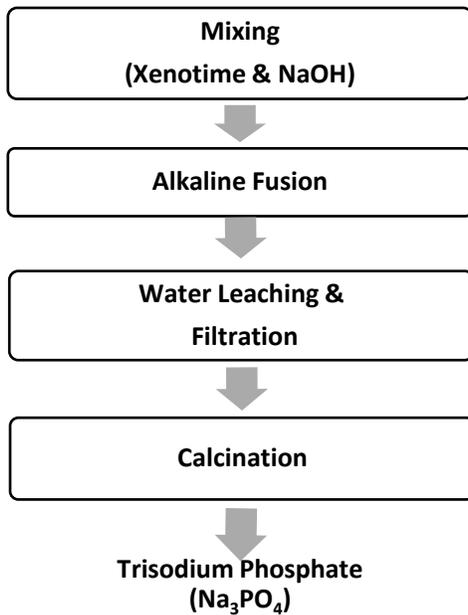
Both monazite and xenotime concentrates are able to produce TSP throughout the alkaline treatment route. From initial study on RE and Th recovery from Malaysian xenotime mineral for Thorium Flagship Project FP0214D052 DSTIN, it was found that alkaline fusion cracking of xenotime was able to produce radioactive free TSP which was containing about 95% of sodium (Na) and 5% of phosphorus (P) <sup>[1-4]</sup>. The elemental content of TSP from that process is shown in Table 6.

**Table 6:** Elemental content of TSP from alkaline fusion cracking of xenotime <sup>[5]</sup>



**Figure 22.** TSP from the studied process is called as TRINAF

After fusion cracking, the fused product which comprises of REE and TSP will undergo a leaching process. Owing to its highly water solubility property, the separation of TSP in fused product was made by leaching with generous amount of water until pH constant <sup>[5]</sup>. After leaching, the collected solution will undergo a dehydration process by heating up the solution until white crystal formed. Finally, the solid crystal was calcined under high temperature in a furnace to remove excessive water until TSP powder formed. The powder was stored in the air-tight container to prevent moisture absorption. Basic flow process for TSP production from xenotime through alkaline fusion cracking is show in Fig. 23.



**Figure 23.** Process flow of TSP generation.

### 3.2 Application

Because of its extreme alkalinity property, TSP is mostly used in agriculture as seed treatment to protect and disinfect seeds from potential diseases, soil conditioner to treat soil and promote plants growth, pesticide or fungicide to kill fungal diseases and mold, and disinfectant to disinfect gardening tools that been used to handle diseased plants. Other than, it is also used in household routine such as for cleaning, sanitizing, and removing stains from various substrates.



**Figure 24.** Common applications of TSP.

## REFERENCES

- [1] Meor Yusoff, M. S., Khaironie, M.T., Nursaidatul, K., Ahmad Khairulikram, Z., & Nur Aqilah, S. (2016) *An Alternative Alkaline Fusion Process for the Production of Heavy Rare Earth, Thorium, Uranium and Phosphate from Malaysian Xenotime*. Recent Advances in Environment, Ecosystems and Development. 163-167
- [2] Khaironie, M.T., Roshasnorlyza, H., Nur Aqilah, S., Ahmad Khairulikram, Z., Wilfred, P., Norhazirah, A., Jacqueline K., Mohd Izzat Fahmi, M. R., & Siti Salwa, Z. A. (2018). *Production of TRINAF from Xenotime via Alkaline Fusion Process*. Poster presentation on Technology Preview and Showcase Nuklear Malaysia.
- [3] Khaironie, M.T., Roshasnorlyza, H., Nur A. S., Ahmad Khairulikram, Z., Wilfred P., Norhazirah A., Jacqueline K., Mohd Izzat Fahmi, M. R., & Siti Salwa, Z. A. (2018). *TRINAF: Gardening aid from mineral digestion process*. Poster presentation for Hari Inovasi Nuklear Malaysia.
- [4] Khaironie, M.T. Jacqueline, K., Roshasnorlyza, H., Norhazirah, A., Nur Aqilah, S. & Wilfred, P. (2019). *Decomposition of Xenotime Concentrate Using Alkaline Fusion Method*. ALTA 2019 Uranium REE Proceedings
- [5] Khaironie, M.T., Roshasnorlyza, H., Nur Aqilah, Sapiee., and Ahmad Khairulikram, Z. (2018). *Standard Operation Procedure: Process for Production of Trisodium Phosphate ( $Na_3PO_4$ ) From Malaysian Xenotime Using Alkaline Fusion Digestion Method*. NUKLEARMALAYSIA/M/2018/21.

# CHAPTER 4: Achievement

---

## 4.1 Introduction to Achievement

This chapter report the human capital building for Malaysia REE and its mineral's study. It is considered to be one of the most important factors for the achievements in this study. This process requires creating the necessary training to learn know-how, apply innovative ideas, acquire new competencies, and develop skills, behaviors and attitudes. Seven training programs related to REE and its mineral have been attended locally and internationally. Knowledge in this field has also been transferred to students at higher institutions, industries and government agencies through training, meetings and conferences.

This chapter also reports the achievements of Malaysia REE and its mineral's study. The achievements are evaluated within the context of a few select categories; innovation, patents, and publications. A total of two research applications have been produced successfully. Innovation in this study is visible when two golds, three silvers and three bronzes have been awarded in the Malaysian Technology Expo (MTE), Exposition on Islamic Innovation (i-INOVA), Anugerah Harta Intelek Negara 2016 (AHIN2016) and Nuklear Malaysia Innovation competition. 42 publications which include journal articles, as well as international and national working papers for conferences, technical reports and proceedings have been published. This achievement can certainly boost up the recognition and spirit of the Malaysia REE and its mineral's study for producing more useful and innovative products in the future. More importantly, this achievement will benefit and have positive impacts on the community.

## 4.2 International linkage, Expertise and Training.

As a measure to build expertise in the field of extraction and mineral product development processes comprehensively, the IAEA Technical Corporation Fellowship project EVT1805736-0001-MYS has been implemented. Through this project, about 10 researchers have been trained

to conduct research in related fields. Awareness of the training needs in this field is being given priority as the demand for REE and mineral products is very high especially in the manufacturing of generator motors, automobiles, hard disc drives and many other electronics industries.

**Table 7:** List of linkage, expertise and training.

No.	Title
1.	Technical Cooperation Fellowship (EVT1805736-0001-MYS), 3-28 June 2021, Thailand Institute of Nuclear Technology.
2.	IAEA Expert Mission (TC MAL2007) on Solvent Extraction Technology For Laboratory Scale, 10 - 13 September 2019, Selangor. Malaysia.
3.	Third Research Coordination Meeting (RCM) on Uranium/Thorium fuelled High Temperature Gas Cooled Reactor Applications for Energy Neutral and Sustainable Comprehensive Extraction and Mineral Product Development Processes. 2-6 July 2018, International Atomic Energy Agency, Vienna International Centre, Vienna, Austria.
4.	RCA/KAERI Introductory Training Course on Radiation Technology and its Applications, 28 October - 8 November 2019, Daejeon, Korea.
5.	Inter-Regional Training Course on State Systems of Accounting for and Control of Nuclear Material, 1 - 5 October 2018, Bahadurgarh, India.
6.	Technical Cooperation Fellowship (TC MAL 2007) on Mineral Characterization and Ore Processing, 1-28 February 2017, National Nuclear Energy Agency (BATAN), Jakarta, Indonesia
7.	Latihan Pemrosesan Mineral, 17-19 November 2015, Pusat Penyelidikan Mineral, Jabatan Mineral dan Geosains, Ipoh, Perak.



**Figure 25.** Technical Cooperation Fellowship in Thailand.



**Figure 26.** IAEA Expert Mission by Dr. Ahmed El Sayed Mady Hussein from Nuclear Material Authority of Cairo, Egypt.



**Figure 27.** Third Research Coordination Meeting (RCM) for Coordinated Research Project (CRP).



**Figure 28.** Last day presentation for Introductory Training Course on Radiation Technology and its Applications.



**Figure 29.** Member state presentation during sharing experience session.



**Figure 30.** IAEA Fellowship Training on Mineral Characteristic and Ore Processing.



**Figure 31.** Mineral Processing Training in Ipoh, Perak.

### **4.3 Industrial Training Supervision**

Human capability building in the field of REE and its mineral has been conducted by supervising students from international and local institutions (University of Queensland (UCSI), Universiti Kebangsaan Malaysia (UKM), Universiti Putra Malaysia (UPM), Universiti Sains Islam Malaysia (USIM), Universiti Malaysia Kelantan (UMK), Universiti Malaysia Pahang (UNIMAP), Universiti Sains Malaysia (USM), Universiti Teknologi Mara (UiTM)).

**Table 8:** List of Industrial training students.

No.	Name	Institution
1.	Nor Hasmida Zainy	UITM
2.	Muhamad Ariff Amir Hamzah	UITM
3.	Nur Izzati Jaffery	USIM
4.	Ahmad Hazim Ahmad Ahfrai	UPM
5.	Mohd Shahiran Mohd Sakri	UPM
6.	Mohamad Noor Nu'aim	UPM
7.	Sanjith Udayakumar	USM
8.	Hazwani Mohd Noor	UMK
9.	Syaheera Putri Hasan	UMK
10	Shahida Salih	UCSI
11	Natrah Shafiqah Rosli	UPM
12	Nor Atikah Jembari	UiTM
13	Mariani Ahmad	UMT
14	Muhammad Faris Rosdidi	UMT
15	Muhammad Azri Muhamad Yusop	UPM
16	Any Mastura Zulpakar	UiTM
17	Nurfatin Izwani Rosli	IIUM
18	Muhammad Ishraff Idris	IIUM
19	Noor Hanani Hanis Jaidi	UiTM
20	Mohamad Hakimi Azhar	UiTM
21	Muhammad Irsyad Roslan	UiTM



**Figure 32.** Briefing on XRF sample preparation to industrial students.

#### 4.4 Innovation.

In realizing the potential of Malaysia REE and its mineral, the outputs of this study have been generated and disseminated at the national levels. As such, several awards have been won in innovation competitions:

**Table 9:** Malaysia REE and its mineral innovation achievement.

No.	Programme	Award	Title
1.	Malaysia Technology Expo 2019 (MTE2019).	Bronze	TRINAF: Gardening Solution from Rare-Earth Mineral.
2.	Pertandingan Inovasi Nuklear Malaysia 2018.	Gold Medal Anugerah Saintis Nuklear	TRINAF: Gardening Aids from Mineral Digestion Process.
3.	Pertandingan Inovasi Nuklear Malaysia 2017	Silver Medal	Graphite Doped TiO <sub>2</sub> Nanoparticles (GTN) - The Excellent Smoke Degradation.
4.	Seminar R&D Agensi Nuklear Malaysia 2016.	3 <sup>rd</sup> Place (Best Poster Award)	Effect of Graphite Doped TiO <sub>2</sub> Nanoparticles on Smoke Degradation.
5.	Anugerah Harta Intelek Negara 2016 (AHIN2016)	Silver Medal (Paten Category)	Preparation of Nanoscale Anatase and Nanoscale Rutile and Uses Thereof.
6.	Malaysian Technology Expo 2016 (MTE2016)	Bronze Medal.	Nanocoated Facemask for Haze Protection Application.
7.	6 <sup>th</sup> Exposition on Islamic Innovation 2015 (i-INOVA2015)	Al-Khawarizmi Award Gold Medal.	Nanotitania Antibacterial Coating.
8.	Pertandingan Inovasi Nuklear Malaysia 2015	Komoditi Inovasi 2015 Award Silver Medal.	Haze Facemask: Nano Titania Coated Face Mask to Solve Haze Problem.



**Figure 33.** Awards giving ceremony during the innovation competitions.

#### 4.5 Patent

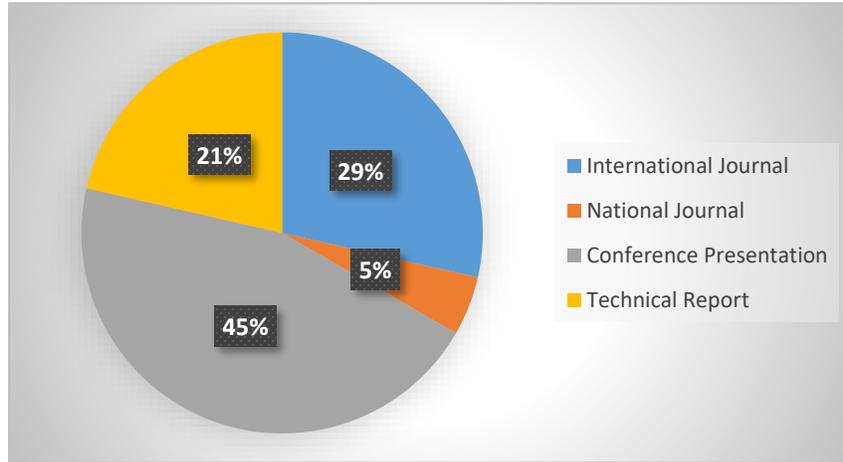
The study of Malaysia REE and its mineral has been granted four patents. These patents are detailed below:

**Table 10:** List of patents filed.

No.	Patent Name	Patent
1.	Method of producing photocatalytic rutile titanium dioxide.	Filing No. PI2012003514
2.	Preparation of nanoscale anatase and nanoscale rutile and uses thereof.	Filing No. PI2012004623
3.	Method of fabricating titanate nanowire.	Filing No. PI2013000771
4.	Method of processing xenotime for the recovery of thorium, uranium and phosphate	Filing No. PI 2015704208

## 4.6 Publication

Publication is one of the Knowledge Management (KM) methods implemented in this study. This is to ensure that all information can be managed well and then can be used as a continuation for future reference. Malaysia REE and its mineral's study successfully produced 42 publications [1-42].



**Figure 34.** Total number of publications.

## REFERENCES

- [1] Norhazirah, A., Khaironie, M. T., Roshasnorlyza, H., Wilfred, S. P., Nur Aqilah, S. and Jacqueline, K. 2020. Solvent extraction of Thorium from Malaysian xenotime using Tributyl Phosphate (TBP). IOP Conf. Series: Materials Science and Engineering 785; 012015
- [2] Roshasnorlyza, H., Muhammad Azri, M. Y., Wilfred, P. and Khaironie, M. T. 2020. Obtaining  $TiO_2$  and  $Fe_2O_3$  from Ilmenite via Alkaline Fusion Method. Materials Science Forum 1010: 385-390.
- [3] Roshasnorlyza, H., Jacqueline, K., Khaironie, M. T., Nur Aqilah, S., Norhazirah, A. and Wilfred, P. 2019. Recovery of thorium and rare earth element (REE) from different particle size of xenotime mineral. Journal of Nuclear and Related Technologies 16: 25-30.
- [4] Norhazirah, A., Khaironie, M. T., Jacqueline, K., Nur Aqilah, S. and Ahmad Khairulikram, Z. 2019. Cracking of Malaysian Xenotime Concentrate in Sodium Hydroxide Using Fusion Technique. Jurnal Sains Nuklear Malaysia 31(1): 11 – 17.

- [5] Khaironie, M. T., Jacqueline, K., Roshasnorlyza, H., Norhazirah, A., Nur Aqilah, S. and Wilfred, P. 2019. Decomposition of Xenotime Concentrate using Alkaline Fusion Process. Australian Language Technology Association (ALTA2019) Proceeding.
- [6] Nur Aqilah, S., Khaironie, M. T., Roshasnorlyza, H., Ahmad Khairulikram, Z. and Wilfred, P. 2019. Extraction of thorium from Malaysian xenotime for preparation of nuclear grade thorium. AIP Conference Proceeding 2068: 020012.
- [7] Ahmad Khairulikram, Z., Nuraqilah, S., Khaironie, M. T., and Roshasnorlyza, H. 2019. The upscaling of the alkaline fusion process. AIP Conference Proceeding 2068: 020015.
- [8] Natrah Shafiqah, R., Che Azurahaman, C. A. and Roshasnorlyza, H. 2018. Synthesis, characterization and investigation of photocatalytic activity of nano-titania from natural ilmenite with graphite for cigarette smoke degradation. Results in Physics Vol. 11; pp. 72-78.
- [9] Sanjith, U., Sheikh, A. R., Ahmad Fauzi, M. N., Teuku Andika, R. P., Khaironie, M. T., Roshasnorlyza, H. 2018. Characterization of Malaysian Monazite Concentrate for the Recovery of Thorium Dioxide. IOP Conference Series: Journal of Physics 1082: 012090.
- [10] Meor Yusoff, M.S., Nursaidatul, S.K. and Ahmad Khairulikram, Z. 2016. Standardless EDXRF analysis methods for Malaysian tin slag waste. Materials Science Forum, vol. 840, pp410-415.
- [11] Meor Yusoff, M. S., Khaironie, M. T., Nursaidatul, K., Ahmad Khairulikram, Z. and Nuraqilah, S. 2015. An Alternative Alkaline Fusion Process for the Production of Heavy Rare Earth, Thorium, Uranium and Phosphate from Malaysian Xenotime. Journal of Recent Advances in Environment, Ecosystem and Development. pp. 163-167. 2015.
- [12] Khaironie, M. T., Masturah, M. and Meor Yusoff, M. S. 2015. Solvent Extraction of Light Rare Earths from Acidic Medium by Di-(2-ethylhexyl) Phosphoric Acid in Kerosene. Journal of Engineering Vol. 27(1); 031.
- [13] Khaironie, M. T. 2015. Course on State Systems of Accounting for And Control of Nuclear Material. Nuclear Bulletin of Malaysia 16; p. 15- 16; ISSN:13945610.
- [14] Khaironie, M. T., Masturah, M., Meor Yusoff, M. S. and Nazaratul Ashifa S. 2014. Solvent Extraction of Light Rare Earth Ions Using D2EHPA from Nitric Acid and Sulphuric Acid Solutions. Advanced Materials Research, Vol. 970, pp. 209-213.
- [15] Khaironie, M. T., Roshasnorlyza, H., Wilfred, P., Norhazirah, A., Jacqueline, K., Nur Aqilah, S. 2019. End of Project Report – Production of Nuclear-Grade Thorium and Uranium from Local Sources for Reactor Fuel. NUKLEARMALAYSIA/L/2019/10
- [16] Roshasnorlyza, H., Khaironie, M. T., Muhamad Nor Nu'aim, Z. and Nur Aqilah, S. 2019. Alkaline Fusion of Malaysian Xenotime: Prospect of High Temperature Gas Cooled Reactor. NUKLEARMALAYSIA/L/2019/36.

- [17] Roshasnorlyza, H., Hazwani, M. N. and Khaironie, M. T. 2019. High Temperature Gas Cooled Reactor Application for Alternative Recovery of Thorium from Malaysian Monazite Sands. NUKLEARMALAYSIA/L/2019/38.
- [18] Roshasnorlyza, H., Muhammad Azri, M. Y., Natrah Syafiqah, R. and Wilfred, P. 2019. Utilization of Heat from High Temperature Gas Cooled Reactor to Produce Advanced Materials from Malaysian Ilmenite. NUKLEARMALAYSIA/L/2019/39.
- [19] Roshasnorlyza, H. 2018. Procedure for Ball Mill System (MyTech-BM3). NUKLEARMALAYSIA/M/2018/18.
- [20] Roshasnorlyza, H., Wilfred, P., Ahmad Khairulikram, Z., Khaironie, M. T., Sarala, S. and Nor Azillah Fatimah, O. 2018. Quantitative Analysis Using A Calibration Curve For Thorium Concentration. NUKLEARMALAYSIA/M/2018/19.
- [21] Roshasnorlyza, H. and Wilfred, P. 2018. Standard Operating Procedures Renishaw Invia Confocal Raman Spectrometer (Single Point Measurement). NUKLEARMALAYSIA/M/2018/20.
- [22] Khaironie, M. T., Roshasnorlyza, H., Ahmad Khairulikram, Z., Nur Aqilah, S. 2018. Procedure for production of trisodium phosphate (TSP) from Malaysian xenotime by alkaline fusion method. NUKLEARMALAYSIA/M/2018/21.
- [23] Ahmad Khairulikram, Z., Norhazirah, A., Jacqueline, K., Nur Aqilah, S., Mohd 'Izzat Fahmi, M. R., Nasasni, N. and Narimah, A. B. 2017. Wavelength Dispersive X-Ray Spectrometer (WDXRF) Analysis of Borehole Samples for presence of Uranium and Thorium Resources in Sg. Chalit, Raub Pahang (BSC-01). NUKLEARMALAYSIA/L/2017/ 137.1-40 (S).
- [24] Roshasnorlyza, H., Nuraqilah, S., Norhazirah, A., Wilfred, P., Khaironie, M. T., Jacqueline, K. and Muhamad Nor Nu'aim, Z. 2020. Different Particle Size of Xenotime on Alkaline Fusion for Yttrium Recovery. Seminar R&D 2020, Bangi, Selangor, Malaysia - poster presentation (online).
- [25] Roshasnorlyza, H., Jacqueline, K., Khaironie, M. T., Nur Aqilah, S., Norhazirah, A. and Wilfred, P. 2019. Recovery of Thorium and Rare Earth Element (REE) from Different Particle Size of Xenotime Mineral. International Nuclear Science, Technology and Engineering Conference 2019 (iNuSTEC2019), Bangi, Selangor, Malaysia – poster presentation.
- [26] Norhazirah, A., Khaironie, M. T., Nur Aqilah, S., Roshasnorlyza, H., Jacqueline K. and Wilfred P. 2019. Solvent Extraction of Thorium from Local Mineral Xenotime Using Tributyl Phosphate (TBP). International Nuclear Science, Technology and Engineering Conference 2019 (iNuSTEC23019), Bangi, Selangor, Malaysia – oral presentation.
- [27] Roshasnorlyza, H., Muhammad Azri, M. Y., Wilfred, P. and Khaironie, M. T. 2019. Obtaining  $TiO_2$  and  $Fe_2O_3$  from Ilmenite via Alkaline Fusion Method. Conference on Advanced Materials Characterization Technique 2019, 23-24 July 2019, Kangar, Perlis, Malaysia – poster presentation.

- [28] Khaironie, M. T., Wilfred, P., Roshasnorlyza, H., Jacqueline, K., Norhazirah, A., Nuraqilah, S. 2019. Overview of Thorium Research Facilities, Seminar On Development Of Innovative Nuclear Reactor Technology (With The Spin-Off) Based On Thorium, 11-12 July 2019, Bangi, Malaysia - oral presentation.
- [29] Nur Aqilah, S., Khaironie, M. T., Roshasnorlyza, H., Norhazirah, A., Jacqueline, K. and Wilfred, P. 2019. Towards Production of Nuclear Grade Thorium from Malaysian Xenotime and Monazite, Seminar On Development Of Innovative Nuclear Reactor Technology (With The Spin-Off) Based On Thorium, 11-12 July 2019, Bangi, Malaysia - oral presentation.
- [30] Khaironie, M. T., Jacqueline, K., Roshasnorlyza, H., Norhazirah, A., Nur Aqilah, S. and Wilfred, P. 2019. Decomposition of Xenotime Concentrate using Alkaline Fusion Method, Australian Language Technology Association (ALTA2019), 18-28 May 2019, Perth, Australia – oral presentation.
- [31] Roshasnorlyza, H., Muhamad Nor Nu'aim, Z., Nur Aqilah, S., Ahmad Khairulikram, Z., Wilfred, P., Khaironie, M. T., Norhazirah, A., Jacqueline, K. and Mohd 'Izzat Fahmi, M. R. 2018. Influence of Fuse Duration On The Thorium Recovery From Malaysian Xenotime. WiN 3rd Annual Conference 2018, 19 - 20 September 2018, Bangi, Malaysia – poster presentation.
- [32] Norhazirah, A., Khaironie, M. T., Jacqueline, K., Nur Aqilah, S. and Ahmad Khairulikram, Z. 2018. Cracking of Malaysian Xenotime Concentrate in Sodium Hydroxide Using Fusion Technique. WiN 3rd Annual Conference 2018, 19 - 20 September 2018, Bangi, Malaysia – poster presentation.
- [33] Jacqueline, K., Norhazirah, A., Nur Aqilah, S. and Khaironie, M. T. 2018. Alkaline Fusion of Malaysian Monazite and Xenotime for the Separation of Thorium and Uranium. WiN 3rd Annual Conference 2018, 19 - 20 September 2018, Bangi, Malaysia – poster presentation.
- [34] Sanjith, U., Sheikh, A. R., Ahmad Fauzi, M. N., Teuku Andika, R. P., Khaironie, M. T., Roshasnorlyza, H. and Ismail I. 2018. The Dephosphorization Behaviour of Malaysian Monazite Concentrates. 3rd International Conference on Innovative Advancements in Engineering and Technologies (IAET) 2018, Jaipur India, 23-24 March 2018, 2018 – oral presentation.
- [35] Roshasnorlyza, H., Khaironie, M. T., Wilfred, P., Ahmad Khairulikram, Z., Nuraqilah, S., Siti Salwa, Z. A., Norhazirah, A., Jacqueline, K. and Mohd 'Izzat Fahmi, M. R. 2018. Effect of Caustic Soda Ratio On Malaysian Xenotime Mineral. International Symposium on Uranium Raw Material for the Nuclear Fuel Cycle: Exploration, Mining, Production, Supply and Demand, Economics and Environmental Issues (URAM 2018), Vienna, Austria, 25-29 June 2018 – oral presentation.
- [36] Nur Aqilah, S., Khaironie, M. T., Roshasnorlyza, H., Ahmad Khairulikram, Z. and Wilfred, S. P. 2018. Preparation of Nuclear Grade Thorium from Malaysian Xenotime by Solvent Extraction. International Conference on X-ray and Related Techniques in Research and Industry (ICXRI 2018), 18-19 August 2018, Kota Bharu, Kelantan - poster presentation.

- [37] Ahmad Khairulikram, Z., Roshasnorlyza, H., Nur Aqilah, S. and Khaironie, M. T. 2018. Upscaling of the Alkaline Fusion Process. International Conference on X-ray and Related Techniques in Research and Industry (ICXRI 2018), 18-19 August 2018, Kota Bharu, Kelantan - poster presentation.
- [38] Khaironie, M. T., Roshasnorlyza, H., Wilfred, P., Ahmad Khairulikram, Z., Nur Aqilah, S., Siti Salwa, Z. A., Norhazirah, A., Jacqueline, K. and Mohd 'Izzat Fahmi, M. R. 2018. R&D Seminar Nuclear Malaysia 2018, Bangi, Selangor, 30th October – 2nd November 2018 - Poster presentation.
- [39] Khaironie, M. T., Roshasnorlyza, H., Wilfred, P., Nur Aqilah, S., Ahmad Khairulikram, Z., Siti Salwa, Z. A., Norhazirah, A., Jacqueline, K. and Mohd 'Izzat Fahmi, M. R. 2018. Rare Earth for Green Technology. Hari 'Knowledge Management' 2018.
- [40] Roshasnorlyza, H., Nur Aqilah, S., Ahmad Khairulikram, Z., Siti Salwa, Z. A., Wilfred, P. and Khaironie, M. T. 2017. Mineralogical Characteristic of Mineral Phases in Malaysian Monazite. 8<sup>th</sup> SIMPOMIN & JMG 2017 Conference, Ipoh, Perak, Malaysia – poster presentation.
- [41] Natrah Shafiqah, R., Che Azurahaman, C. A. and Roshasnorlyza, H. 2017. Nano-Titania for Photocatalyst Applications. Fundamental Science Congress 2017, Serdang, Selangor, Malaysia - poster presentation.
- [42] Roshasnorlyza, H., Meor Yusoff, M. S., Mohamad Shahrizal, M. Z., Natrah Syafiqah, R., Ahmad Khairulikram, Z., Khaironie, M. T., Nuraqilah, S., Wilfred, P. and Siti Salwa, Z. A. 2016. Effect of Graphite Doped TiO<sub>2</sub> Nanoparticles on Smoke Degradation. Seminar R&D Agensi Nuklear Malaysia 2016, Bangi, Selangor, 8 -10th November 2016 – poster presentation.

# AUTHOR'S BIOGRAPHY

---

## *DR. JULIE ANDRIANNY MURSHIDI*



Julie Andrianny Murshidi conferred her Ph.D. award in Applied Physics from Curtin University, Perth, Australia. Prior to this, she has gained her Master's Degree in Material Science from the National University of Malaysia. With Material Science background, Nuklear Malaysia offered her as a Research Officer in June 2001 serving under the Material Technology Group. She has been appointed as editor, referee, chapter writer and publication committee for several scientific journals and books. She has also served as the Knowledge Management Officers for Industrial Technology Division since 2016.

## *KHAIRONIE MOHAMED TAKIP*

Khaironie Mohamed Takip obtained her Bachelor of Science in Chemical Engineering from the University of Missouri-Columbia USA and presently pursuing Master of Engineering (Chemical) at the National University of Malaysia. Her career began in 1997 as a Chemical Engineer with Borneo Pulp & Paper Sdn Bhd. In 2002, she joined Nuklear Malaysia as a Research Officer with the Technical Support Division before joining the Nuclear Power Division in 2008. She joined the Material Technology Group in 2014 and was appointed as one of the Sub Project Leaders for the DSTIN Thorium Flagship Project in 2017. Her current research focus is in the separation techniques and rare earth elements recovery from local minerals



## *DR. ROSHASNORLYZA HAZAN*



Roshasnorlyza Hazan has joined Nuklear Malaysia as a Research Officer with the Material Technology Group since 2014. She was awarded the Bachelor of Engineering with Honours in Materials Engineering (2007), Master of Science in Materials Engineering (2008), and Doctor of Philosophy (Ph.D.) in Advanced Materials (2014) from Universiti Sains Malaysia. She was the subproject team members of the DSTIN Thorium Flagship Project. She had been also appointed as the Project Leader for IAEA Coordinate Research Project 'Thorium, uranium, rare earth and other advanced materials from Malaysian non-conventional radioactive mineral and waste'. Her areas of interest include rare earth elements recovery from local minerals and metal oxide synthesis. She has published extensively in the peer-reviewed research journal and as a scientific journal reviewer.

### ***WILFRED PAULUS***



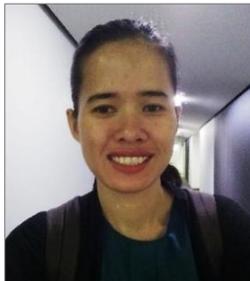
Wilfred Paulus is the Service Manager for Material Technology Laboratory. He started his career as a Research Officer at Nuklear Malaysia in April 2002. He holds Bachelor's Degree in Nuclear Science from the National University of Malaysia and has more than 18 years of experience in R&D projects related to radioactive waste treatment and advanced material. With that extensive experience, he was assigned as the subproject team member for the DSTIN Thorium Flagship Project. He has been trained in theoretical and practical training to operate XRD, EDXRF, Raman spectrometers, and UV-Vis.

### ***NORHAZIRAH AZHAR***

Norhazirah Azhar acquired her Bachelor of Science with Honours in Applied Chemistry from University Technology MARA in Arau, Perlis. She began her career in 2017 as a Trainee in Nuklear Malaysia under the JPA-RSOG program called Talent Acceleration in Public Services (TAPS). In 2018, she officially joined NuklearMalaysia as a Research Officer and serves under the Material Technology Group. She was assigned as the subproject team member of the DSTIN Thorium Flagship Project until project completion. She has also been appointed as one of the personnel in charge of handling XRF and FE-SEM since 2018.



### ***JACQUELINE KONES***



Jacqueline Kones obtained her Bachelor of Science in Chemistry from Universiti Teknologi MARA. Earlier in 2017, she had joined the Talent Acceleration in Public Services program, which was conducted by the JPA-RSOG and Nuklear Malaysia. She officially started her career in Nuklear Malaysia as a Research Officer with the Material Technology Group and was assigned as the subproject team member for the DSTIN Thorium Flagship Project. She has served as one of the personnel in charge of the material characterization method using Particle Size Analyser (PSA) and WD-XRF since 2018.

### ***NURAQILAH SAPIEE***

Nur Aqilah Sapiee's research interest began during her Bachelor's Degree (Environmental Engineering) at the Universiti Malaysia, Perlis. She was a team member for an innovation project named 'sphagSOB: Biodegradable Adsorption for Oil and Grease Trap' which was awarded as Eco-Friendly Award at EINIC 2007 and *Anugerah Inovasi Cemerlang* 2007 from the UNIMAP. During her Master's Degree in the Universiti Putra Malaysia, she was offered as a Research Officer by Nuklear Malaysia. She was one of the subproject team members of the DSTIN Thorium Flagship Project. Presently, she is focusing on hydrometallurgical and XRF characterization techniques.

