

SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING
UNIVERSITI SAINS MALAYSIA

**PROCESSING OF HEAVY MINERALS FROM TERENGGANU
SILICA SAND**

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Dissertation submitted in partial fulfillment
of the requirements for the degree of Bachelor of Engineering with Honours
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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertations entitle 'Processing of Heavy Minerals from Terengganu Silica Sand'. I also declare that is has not been previously submitted for the award of any degree or diploma or other similar title for any other examining body or University.

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PENCIRIAN AMANG DARIPADA PASIR SILIKA DARI TERENGGANU SILICA CONSORTIUM

ABSTRAK

Pengenalan:

Tujuan kajian ini adalah untuk mencirikan sampel gangue mineral silika dan mendapatkan mineral berharga yang diperoleh daripada mineral buangan di Terengganu Silica Sand. Terdapat enam kaedah pencirian yang dilaksanakan dalam kajian ini seperti saiz zarah analisis, kehilangan dalam pembakaran, analisis komposisi unsur (menggunakan XRF), kaedah mikroskop, mendulang dan pemisahan magnetik. Daripada kajian ini, Korundum (Al_2O_3), Silika (SiO_2), Rutile (TiO_2), dan Hematite (Fe_2O_3) telah dijumpai sebagai Amang di Terengganu Silica Sand. Saiz sampel yang telah untuk penyelidikan adalah di antara julat daripada +600 μm hingga -212 μm . Merujuk kepada analisis komposisi; komposisi Korundum meningkat apabila saiz zarah berkurangan. Walau bagaimanapun, walaupun sampel ini berasal dari mineral silika buangan, namun masih terdapat kandungan silika yang tinggi di dalam sisa buangan tersebut.

CHACTERIZATION OF SILICA SAND AMANG FROM TERENGGANU SILICA SAND CONSORTIUM

ABSTRACT

Introduction:

The purpose of this research is to characterize sample silica and to recover the valuable minerals found in silica samples obtained from in Terengganu Silica Sand. There are six methods of characterization were implemented in this research such as Particle Size Distribution, Loss on Ignition, X-ray Fluorescence (XRF), Microscopic Study, Panning and Magnetic Separation. From this study, corundum (Al_2O_3), silica (SiO_2), rutile (TiO_2), and hematite (Fe_2O_3) were found in amang Terengganu Silica Sand. The size of the sample that was chosen for this the research is the ranges from +600 μm to -212 μm . As the result, from composition analysis, the composition of corundum was increase when the size of particle is decrease. However, this sample is from the gangue mineral of silica, but there is still have a higher amount of silica that are still present in the waste.

CHAPTER 1

INTRODUCTION

1.1 Introduction

Sand is used in great types of products produced, and the term 'quartz sand' is used for the essential raw material for making glass and foundry casting industries, as well as in other industries, such as ceramics and chemical manufacture and for water filtration purposes' according to BGS Mineral Planning Factsheet (September 2009). Although quartz has a low specific gravity than other heavy minerals, but it still can be defined as sand used for applications other than construction aggregates and they are valued for their physical and chemical properties. This definition has been recognized by the British Geological Survey (BGS) by the significance of the end use rather than the nature of the sand in the ground.

The important part of the rock cycle which is weathering, has produced quartz sand as their final product. The weathering of any quartz-bearing rock creates sand: igneous, sedimentary, or metamorphic. It is involved in a continuous cycle of rock formation and erosion that started with the Earth's formation and continues today. Weathered grains become separated from inter-grown or cemented minerals that make up hard rocks. Silica sand deposits are usually mature or super-mature. Super-mature sands often are more than 95% quartz with some natural deposits containing 98% quartz.

Silica sand mining is one of the biggest mining around the world and it is not too difficult to get it. There are a lot of silica sand mining around the world for example; Australia which is Cape Flattery Mine Site operated by MC has the largest, high purity silica sand within the others. However, in 2011 foreign industry markets are important as producers to the four largest exporters of industrial silica such United States of America, Australia, Italy, and Germany.

In Asia, Terengganu Silica Sand Consortium is one of the largest silica sand producer and its product provided a purity range from 99.55% to 99.9% of SiO₂. There are a few type of valuable mineral that had been extracted such as Corundum (Al₂O₃), Phosphorite (P₂O₅), Rutile (TiO₂), Ilmenite(FeTiO₃), Monazite(Ce[PO₄,SiO₄]) and Hematite (Fe₂O₃) but there is still have a higher percentage of silica pass to the tailing sump. The minerals weight give some economics value of a cheaper and efficient processes can be develop to recover the minerals. The sample from Terengganu Silica Sand has been taken from the tailing sump and the separation and recovery process had been run in a wet and dry way. In this research, there were no chemical used and only physical separation are been done. In addition, there are a few types of process be used in this research and it was separate by two, which is for process, and another one for characterization. For the process, such as, Panning, X-ray fluorescence, Magnetic Separator, Microscopic Study was used and for the characterization analysis, it was used X-ray fluorescence, Particle Size Analysis and Microscopic Study. In the same time, through this several processes, researcher can know and learned what is the better way or method to separate all these heavy mineral as the recovery and how much these mineral can be recovered from the Silica sand processing.

1.2 Problem Statement

Approximately 10% of feed was reported contain heavy fraction and discarded as waste in the processing plant. Visually this fraction contains mostly free discrete particles with black particle in colour. Technically, this fraction contains minerals with the density is higher than silica which is may contain valuable mineral such as Ilmenite, Monazite, Rutile, Corundum, Magnetite and etc. Therefore, study on this fraction is crucial so that the values that may treated as waste could give some revenue to the company.

1.3 Objective of Study

The main objectives of this research project:

1. To characterize sample of gangue minerals of silica sand process.
2. Recovery of valuable minerals from the gangue minerals by physical processing method.

1.4 Thesis Outline

This thesis has been divide in five chapter, which is for the Chapter 1 is Introduction, Chapter 2 is Literature Review, Chapter 3 is Methodology, Chapter 4 is Result and Discussion and lastly for the final chapter, which is Chapter 5, is Conclusion and Recommendation.

In the first chapter, it is about the introduction of the types of heavy mineral that trapped in the Amang sample, problem statement that involved in this research, followed by objective of this research, overview briefly of the studied areas, scope of research and the thesis outline.

Next, Chapter 2 is about literature review. In this chapter explain about theoretical of the heavy mineral extraction in the Amang sample, described the related past works on the research of characterization of heavy minerals and the properties and characteristic of heavy minerals.

For the following chapter, which is, Chapter 3 is about methodology that is use in this research. It consist the steps and stages of work that already done to achieved the objective of the research. Flowcharts also has been apply in this chapter for a better understanding of the processes that had been done.

For the Chapter 4, it is about the results and discussion of the overall study was done. In the discussion, it was explain about the reason why the result show like this and like that. At last but not lease, Chapter 5 is all about the conclusion and the recommendation of this study and the recommendation for future works.

Chapter 2

Literature Review

2.1 Amang

Normally the tailing tin mining area are rich in Amang that contained heavy minerals, which in turn will be economically beneficial to the mineral industries. However, here in silica sand mining also contain the same Amang as the tin mining area. Heavy minerals, which are having a specific gravity of higher than 3.0 was found, appear in result of silica sand. This heavy mineral usually used in minerals industries such as Ilmenite, Zircon, Monazite, etc and they have a high value in the market.

There are many uses of these heavy mineral in the industries. For the example, Zircon used in various industries such as foundry, ceramic production and casting applications. China represents the largest market worldwide, supported by the massive domestic production capacity for Zirconium sponge. The major manufacturer of zircon worldwide have been top by Australia and South Africa and Asia-Pacific were ranks as the fastest growing market exclude China.

Ilmenite, which is contain titanium dioxide, are famous in white pigment for paint, plastic, paper and fabrics industries. China's are the biggest titanium dioxide (TiO₂) producer.

Ilmenite Mine Production Globally

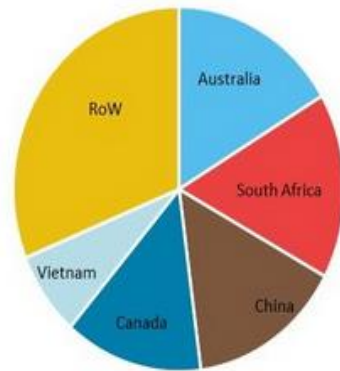


Figure 2.1(S.Platis,K.G.(N.d)

The concentration of heavy minerals from Amang involves a gravitational separation process that will produce a layer, which can be easily differentiated from mud and sand. The more higher density of materials, it will sink more deeper.

2.2 Heavy Mineral Associated with a Mixture of Heavy Minerals.

As mention previously, these Amang samples contain various types of heavy mineral that are high demand in world market price. There are a few types of heavy minerals such as Ilmenite, Zircon, Monazite, Rutile, Magnetite, Corundum etc. A mineral is define as any naturally occurring inorganic solid that possesses an orderly crystalline structure and a definite chemical composition that allows for some variation.

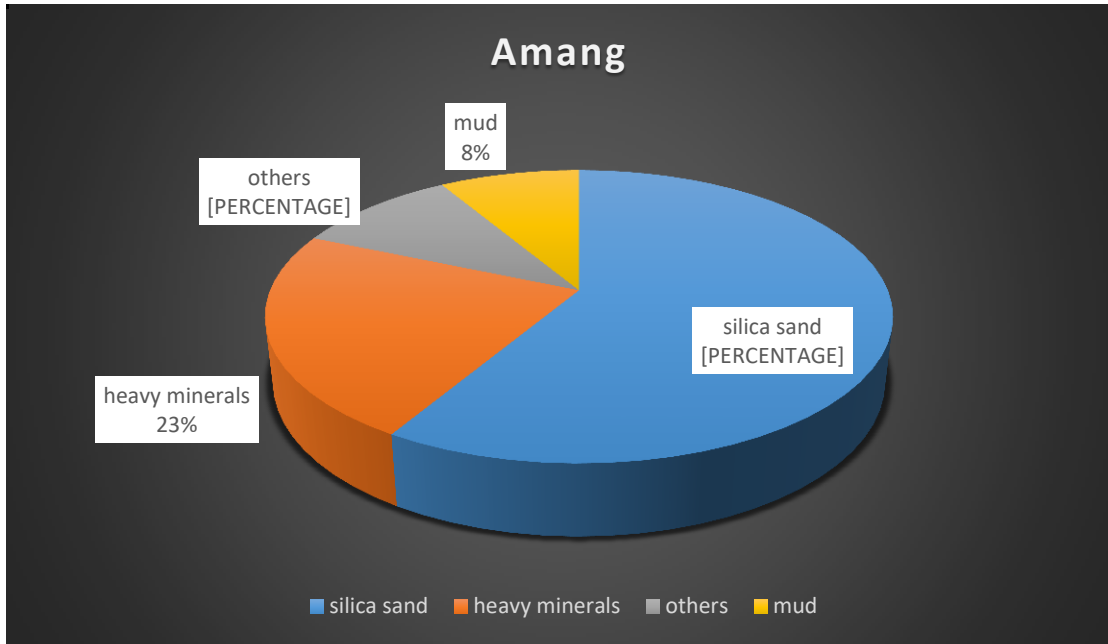


Figure 2.2: Pie chart of percentage Amang minerals that associated in the silica sand. (S.Platis, K.G.(N.d).

2.3 Characterization of Amang

The mineral composition was identified by a few method to make sure the exactly result of the mineral itself. In this research, particle size distribution, elemental composition (using X-ray fluorescence) and microscope study used to identify the mineral composition of the sample. This process is importance to identify of ore for the feasibilities study before everything is begin. The purpose of this process is to make sure this research will achieve its objective, which is to characterize sample of gangue minerals of silica sand process and recovery of valuable minerals from the gangue minerals by physical mineral processing. So from this process, when everything about mineral composition has reveal, the next step can be determine easily by referring to the mineral characteristic itself.

2.31 Particle Size Distribution (PSD)

Through the particle size distribution, it can determine the particle size of particles ranges in a minerals sample. To study the liberation size of the minerals its importance to know the particle size distribution first. The distribution pattern in sample can be determine by this PSD. The gradient shows the range size of mineral sample, which is the steep gradient; indicate the small size and less steep shows the minerals varied with size range. Rutile production has a very high demand in Australia and follow up with the other Asia and worldwide country. The main uses for rutile are manufacture of refractory ceramics, as a pigment and for the production of titanium metal. It also was used in paints, plastics, paper, foods, and other applications that call for a bright white colour industry.

Asia Pacific dominates the global rutile market and the trend is expect to continue during the forecast period. Increasing industrial investment and high GDP growth in the emerging economies of India and China is expect to drive market growth in Asia Pacific. (S.Plantias,K.G.(N.d). Asia Pacific is also expect to be the fastest growing region for rutile demand during the forecast period.

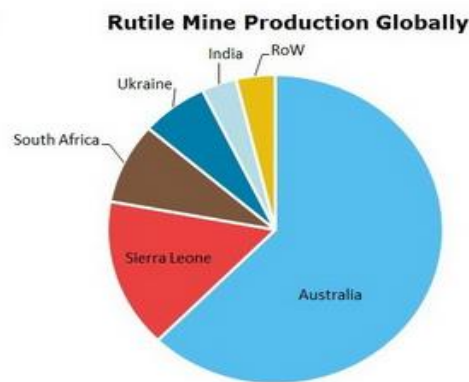


Figure 2.3(S.Plantias,K.G.(N.d).

For the Monazite minerals, there is a primary ore of several rare earth metals most notably thorium, cerium and lanthanum. These metals give mineral composition of $(\text{Ce,La})\text{PO}_4$ and these metals have various industrial uses. Malaysia, Brazil, Vietnam and India are among the countries that monazite has been mined and this shows that monazite have a valuable value in the industry market price. (*S.Platis,K.G.(N.d)*).

Next, for the Corundum which is owing to the extreme hardness of the material (9 out of 10 on the Mohr's hardness scale). There are a few of uses of Corundum based on its hardness, which is used for grinding optical glass and for polishing metals and been made into sandpapers and grinding wheels. For the Magnetite, it contains highest iron content and can be separate through Magnetic Separator. This mineral are highly demand in the industries manufacturing of pigment in paints and as an aggregate in high-density concrete.

Therefore, every minerals that are contain in the Amang have their value and their uses in the industry. That is why this Amang mineral should be recover and this may increase the company profit.

Therefore, by this graph, the value of percentiles shows how the size distributions are spread over interval from smallest to the largest value. The D75, D50 and D25 values represent the 75th, 50th and 25th percentile of cumulative passing size respectively, which are known as third quartile, media and first quartile (*S.Platis,K.G.(N.d)*).

The determination of the gradation of the sample, uniformity coefficient, C_u should be known. So, followed by C_u range, which has been set, more than 6 means the sample is well graded. If the value is less than six, it does not perform mean well in all sizes. Uniformly

graded in sense, the soils have identical size of particle. This is mean the sample that have Cu value more than 6 is a good representation in all sizes. Uniformity coefficient, Cu:

$$Cu = \frac{D_{60}}{D_{10}} \quad (1)$$

Where:

D10= Passing size of mineral at 10%

D60= Passing size of mineral at 60%

(Burt, Charles M.; Styles, Stuart W. (1999).

2.32 Elemental Composition Analysis

X- Ray fluorescence (XRF) spectrometry is a based on the principle that individual atoms, when excited by an external energy source, emit X-ray photons of a characteristic energy or a wavelength. By counting the number of photons of each energy emitted from a sample, the elements present may be identified and quantitated. The purpose of using energy-dispersive X-ray fluorescence (EDXRF) spectrometry is because it is a rapid, multi-element alternative to current methods using acid digestion and inductively, coupled plasma optical emission spectrometry (ICP-OES). *(K.D. (2017)*

Before this process was taken, loss on ignition (LOI) must be done because the value of LOI is used to determine the organic substance in the sample before the sample is sent to the XRF process. In the LOI process, the sample was taken originally from the reference sample then be weight around 30 grams per crucible. This process was done 3 crucible for each sample to get the accurate value. The temperature used is 950°C to allow evaporation of organic substance and volatile materials due to low boiling point of organic substance and volatile materials. This process take 24 hour to finish it until the temperature back to zero Celsius. The sample then is weight after the ignition is done and the difference in weight before and after will show the amount of organic substances and volatile materials. Calculated the % weight loss on ignition using the following formula:

$$\%LOI = \frac{W_f - W_a}{W_f - W_b} \times 100\% \text{ weight loss on ignition} \quad (2)$$

Where:

W_f = weight container filled with the representative sample before ignition

W_b = weight container

W_a = weight container filled with representative sample after ignition

The percentage of LOI that were achieved was used in the XRF elemental analysis

(K.D. (2017)).

2.33 Microscopic Study

Ore microscope is the basic instrument for the petrographic examination of the large and economically important group of minerals referred to collectively as 'ore' or 'opaque' minerals. It was similar to conventional petrographic microscopes in the systems of lenses, polarizer, analyser, and various diaphragms employed, but differs in that its primary method of illumination is a light source above the sample to allow examination by light reflected from polished surface.

The increasing interest in ore-gangue relationships and the recognition that much textural information can be derived from the examination of translucent ore minerals in polished thin section now commonly leads to the use of microscopes equipped for both reflected- and transmitted-light study.

The sample usually can be observe by using either Crossed Polarize Light (XPL) or Plane Polarize Light (PPL). In PPL, mineral can be identify by its colour, reflection pleochroism, reflectance bireflectance and hardness of the ore. Anisotropism and internal reflection can be observe in XPL technique. The minerals that have same properties in all directions is call isotropic and vice versa called anisotropic. For isotropic, which is the light passes through them in the same way, with the same velocity, no matter what direction the light is travelling. So for the anisotropic, it just vice versa with the isotropic.

They travel in the different ways with different velocities while depending on the direction of travel through a grain. These both of the properties can easily distinguished because of

minerals does not transmit light which means always black in isotropic when viewed under XPL. There are two result shown when the minerals is under microscope and it depend to the technology and minerals size. (D.J.Vaughan,I.(N.d).

2.34 Identified Mineral from Amang Onn Sdn Bhd

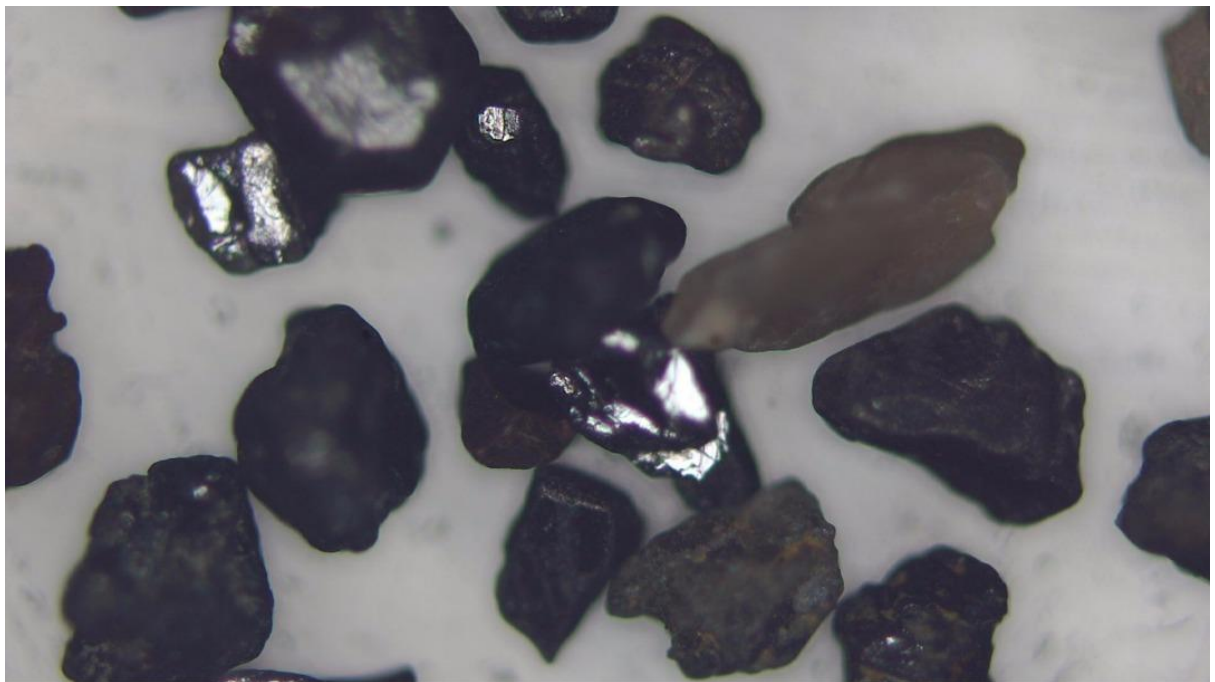


Figure 2.4:Ilmenite



Figure 2.5: Manganese

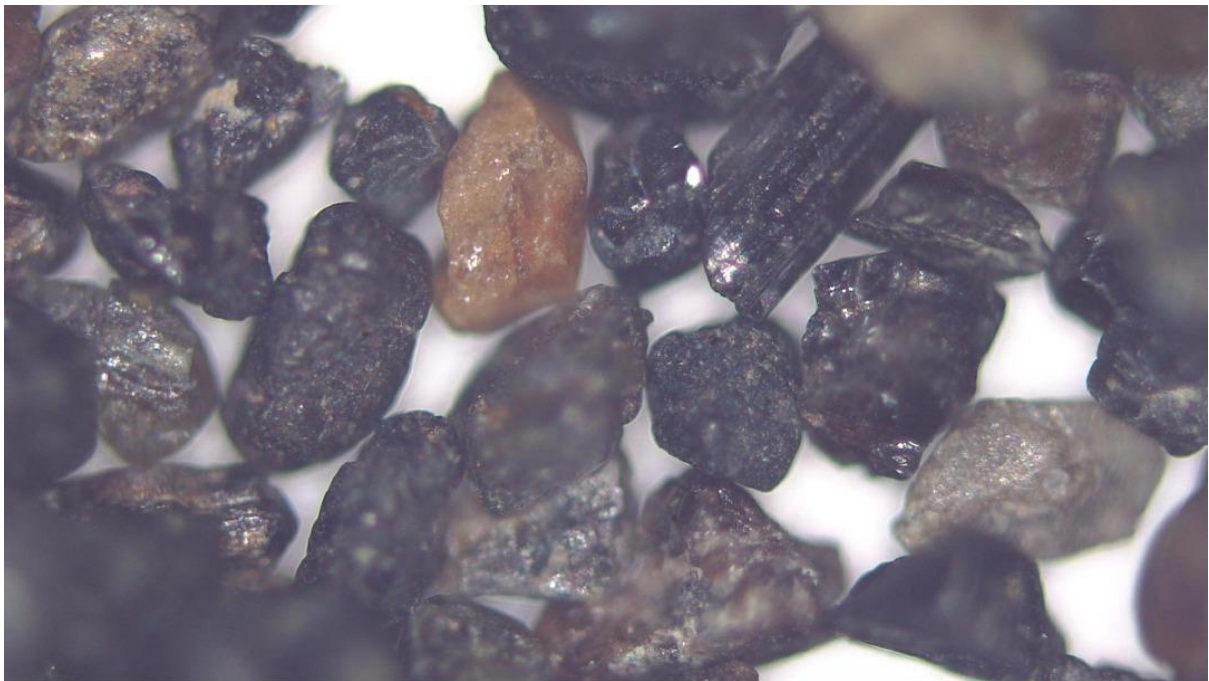


Figure 2.6: Rutile

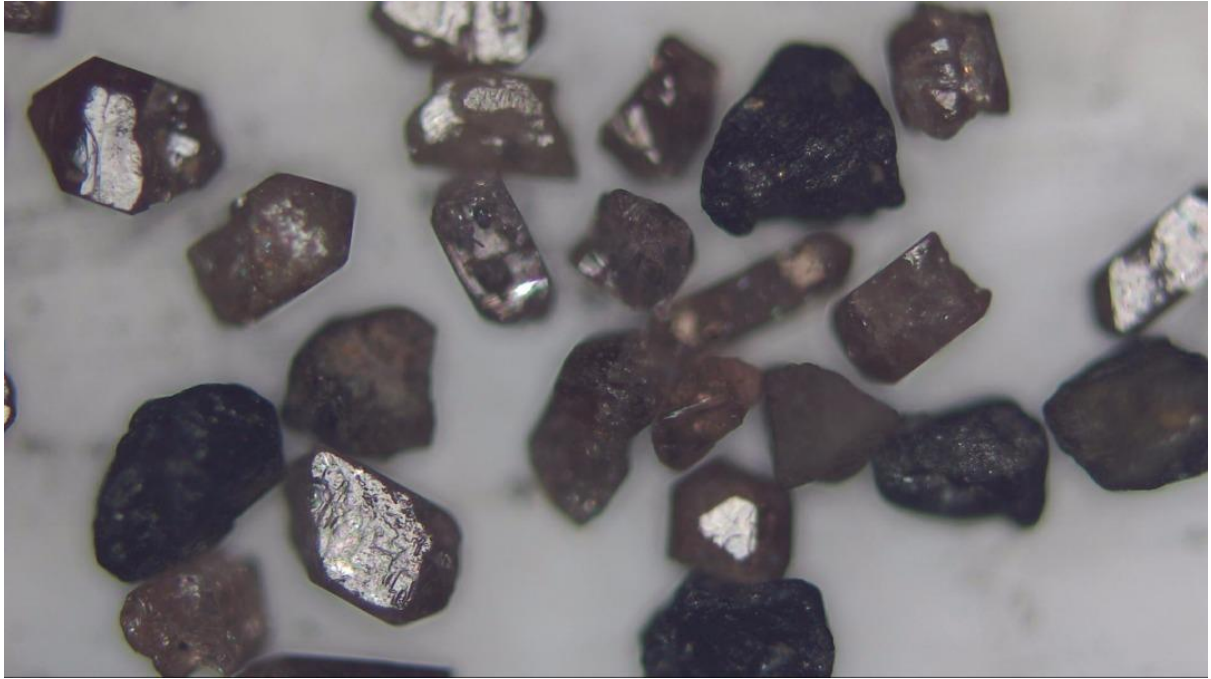


Figure 2.7: Zircon

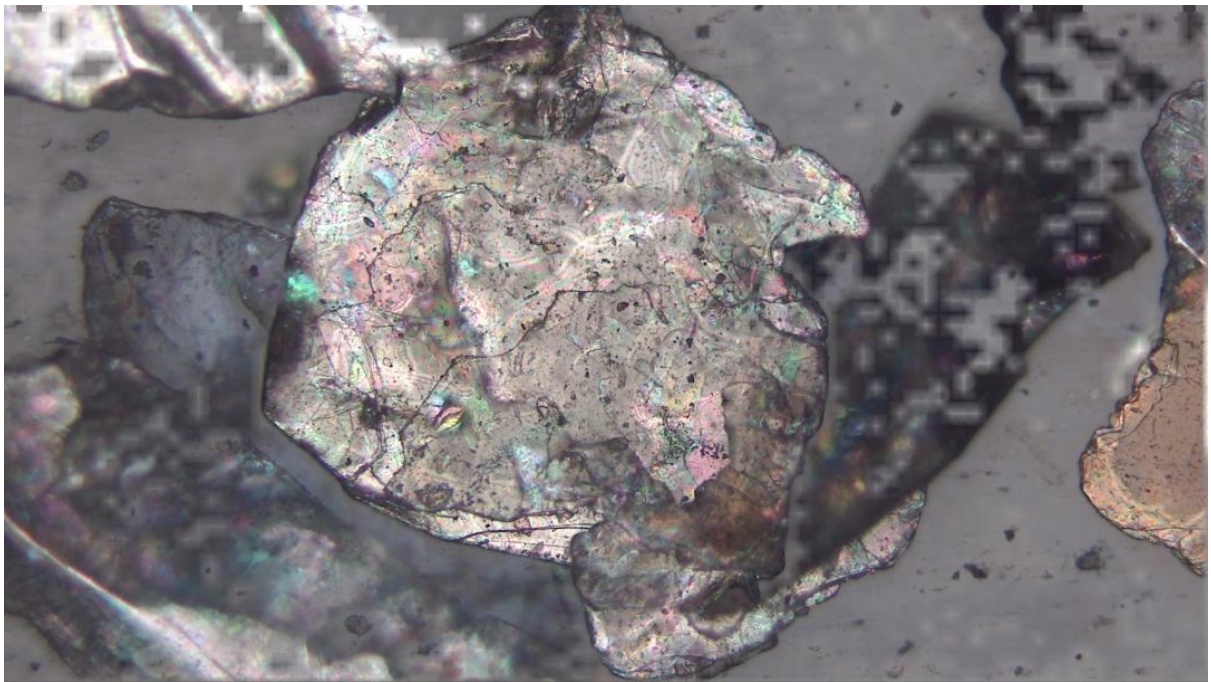


Figure 2.8: Mica

Minerals	Descriptions
Ilmenite	<ul style="list-style-type: none"> • Black colour • Black streak • Metallic • Opaque • Hexagonal
Manganese	<ul style="list-style-type: none"> • Silvery grey • Hard and very brittle
Rutile	<ul style="list-style-type: none"> • Black or reddish brown in colour • Tetragonal
Zircon	<ul style="list-style-type: none"> • Brown or red in colour • Colourless streak • Sometimes oily
Mica	<ul style="list-style-type: none"> • Cleavage flakes • Can be split or delaminated into thin sheets.

Table 2.9: The physical properties of minerals at Amang Onn Sdn Bhd

All of the figures above show the minerals from Amang Onn Sdn Bhd under the microscope. These figures above as the reference to identify the mineral physical properties of the sample from Terengganu Silica Consortium Sdn Bhd, which is also test under the microscopic study.

CHAPTER 3

METHODOLOGY

3.1 Location of Samples in this Study

This location was taken from Google Maps and it show the location of the Terengganu Silica Consortium Sdn Bhd. Both figures are showing where the company belong. In Figure 3.1 shows overall area of the company and the surrounding of the area and in Figure 3.2 show the overview of the Terengganu Silica Consortium Sdn Bhd.

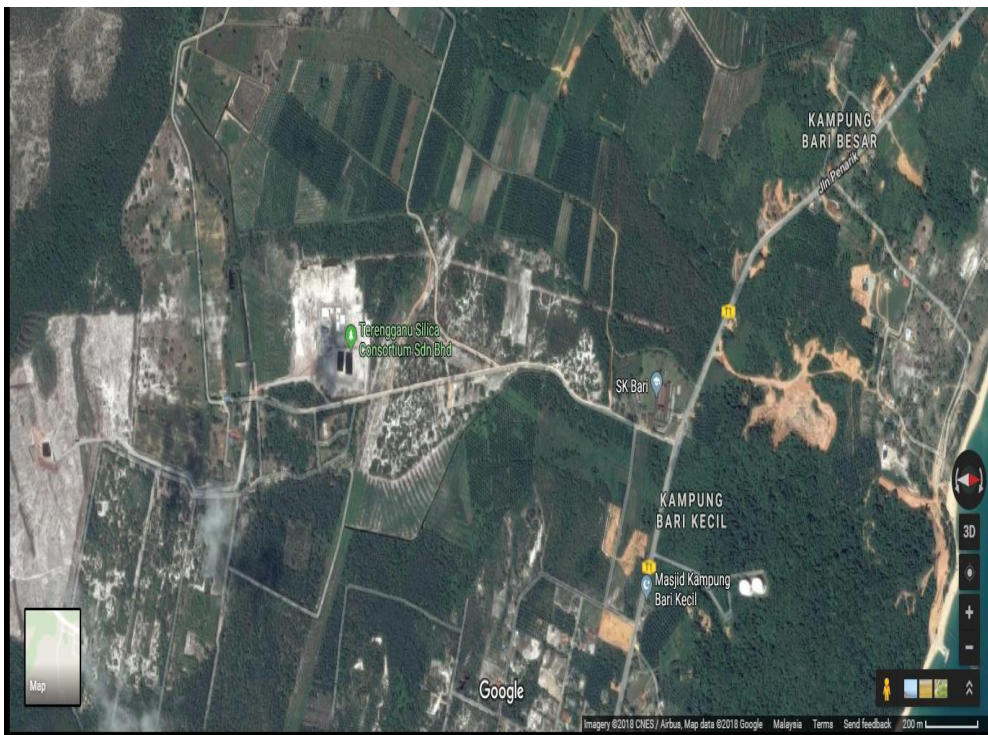


Figure 3.1 Map of Location

Overview of Location.



Figure 3.2 Overview of Location

Based on the location above, the sample of research has been taken from Terengganu Silica Sand at Kampung Bari, Setiu that is one of the largest silica sand producer in Asia.

3.2 Scope of Research

This Amang sample was separate and characterized based on Gilson Sieve mesh size which is starting from $(-1.18\text{ mm}+600\ \mu\text{m})$, $(-600+425)\ \mu\text{m}$, $(-425+300)\ \mu\text{m}$, $(-300+212)\ \mu\text{m}$, and $-212\ \mu\text{m}$. Then the analysis and test was done based on this following sample size. All of this sample size has been separate into two category, which is size-by-size analysis, and for reference. Based on this following size, crushing and grinding are not required for the further characterization process.

For the further research, the sample was been testing by using X-ray Fluorescence technique to determine the elemental composition of minerals. Before this technique been apply, the sample should be ground until it form a powder size $-75\ \mu\text{m}$ as much as 25-30 g per sample. Ring mill, agate mortar and planetary mill are been used to grind the sample based on sample's classification.

Next, microscopic study also been apply for the optical identification and mineral characterization study. In this process, the image of the mineral is capture under microscope and the identification is do based on the mineral characteristic itself.

For more detailed, the sample was separated by using magnetic separator to attract the magnetic mineral and separate it from a mixture of Amang sample. Panning technique also are been used for separated the sample based on its specific gravity so that all of this Amang mixture can be identify one by one based on its physical characteristic.

3.3 Introduction

This chapter discussed the method or technique used to study the stages characteristics and the possibility of concentration of valuable minerals from Terengganu Silica Sand Consortium Sdn Bhd. Figure 3.1 shows the flowchart of the research methodology:

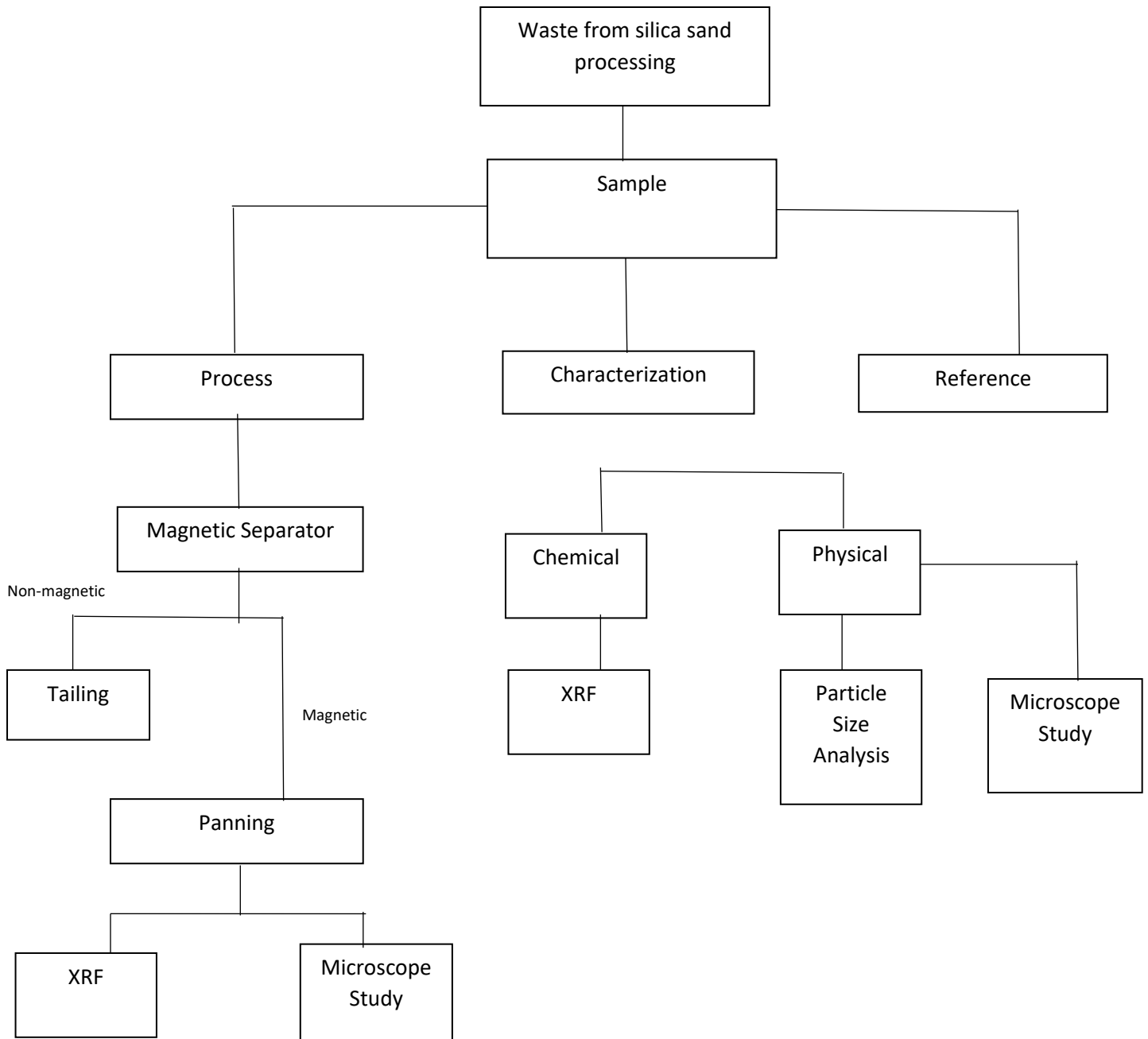


Figure 3.3: Flowchart show the stages of mineral characterization

3.4 Sample

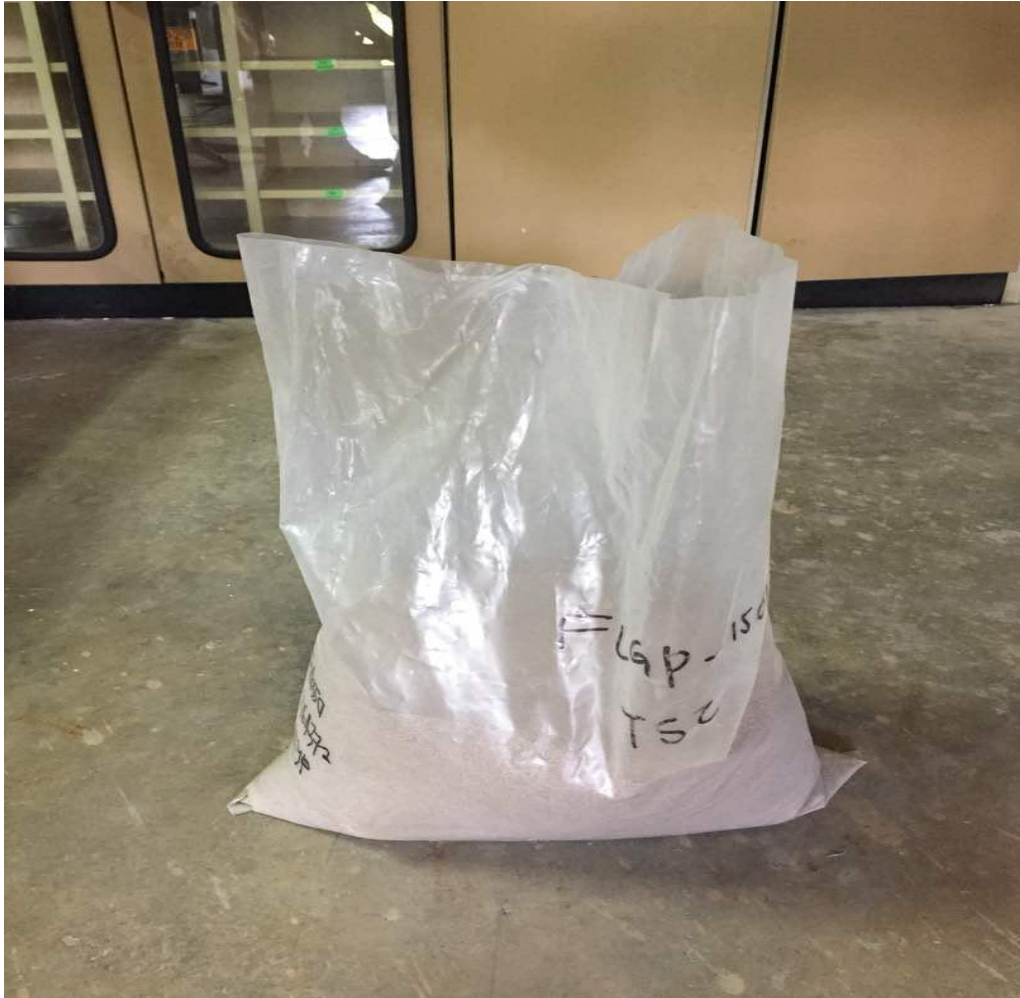


Figure 3.3: The sample taken from Terengganu Silica Consortium

Figure 3.3 shows the Amang sample that was taken from Terengganu Silica Consortium. The weight of the sample is around 11 kg. This sample was a mixed of size from course to fine grain. The coding wrote at the plastic bag of the sample, which is FLGP-150218 means the sample is come from final low-grade product, and the date of the sample taken.

3.5 Sampling

Sampling is an art of securing a representative fraction in a small weight or sample from a relatively large lot. In order to make sample representative, it must be selected in a fair way. Practically, before the sample start to be process to the next stage, the sample should be separate into a two or more portion, which is sample that will be process to continue the research and the other one is reference sample. Reference sample was used if anything with the experiment goes wrong and this references sample presence as a backup sample. In order to separate, the sample into small fraction, Cone and Quartering method is used. This is the listing of apparatus used; plastic mat, shovel, spatula and sample. In this process, the entire sample was put it on a plastic mat. Then mix it by using the shovel and spatula. Make sure it was blend well. After that, the grain mixed and coned it. The grain cone was divide into four fraction by using the spatula. The opposite quarter was take for mixing and forming. Other portion was keep for reference.

3.6 Sieving

Screening or sieving is one of the simplest and direct sizing process. In this, each particle is subject to pass through an aperture of particular size and shape. The sample separated by different type of size based on the aperture size used. After separate the sample into small portion by Cone and Quartering method, the sample is weight and record it. The sample then sieve by vibration screen. In this work, Gilson Sieve is being used because of the portion of sample is quite larger. The total sample weight used to continue as a research sample is about 10 kilograms. So in this case, Gilson Sieve is accommodate for this weight. In this step, put 10 kg of sample into Gilson Sieve, tight it well, then turn it on and left it about 5 minute and off. There are 6 types of sieve size that were used which is (-1.18 mm

+600 μm), (-600+425) μm , (-425+300) μm , (-300+212) μm , and -212 μm . This sample is quite fine, so there is no need to be crush or grind anymore. The sieve is arrange by top to bottom followed by mesh size, as the efficiency decreases rapidly with fineness. At a given capacity, the effectiveness depends on the nature of screening operation, i.e. on the overall chance of a particle passing through the screen once it has reached it. Plugging of minerals particles will cause blinding because of the particle size have a same size with the sieve aperture. Therefore, sieve aperture should be clean after used to avoid blocking the aperture opening.

3.7 Particle Size Distribution

After 10 minutes of sieving, the sample retained were weight one by one based on the sieves size. The sample is then is move into a plastic bag separately according to its size. The mass of the sample were be recorded. Then, plot a graph of the percentage of passing size of sample against the sieves size to obtain the uniformity coefficient, Cu.

3.8 Sample Preparation for XRF.

3.8.1 Loss on Ignition

This process allowing volatile substance to escape until its mass ceases to change. The empty porcelain crucibles were weight first before put the sample inside it and record. Then put the sample inside around 30 grams and weight it. Record. The heating process is then were conducted with increasing temperature of 10°C/min until the soaking temperature of 950°C and the time takes is 95 minute. For the soaking time, it takes 4 hours. Furnace temperature with decrease with same temperature with is 10°C/min and takes 95minute to

cooling down. The reason why it have to be cool down slowly, it is that to avoid the porcelain crucible from thermal shock and it can cause crack on the crucible.



A



B

Figure 3.4: the image A and image B shows the condition of the sample before and after LOI test.

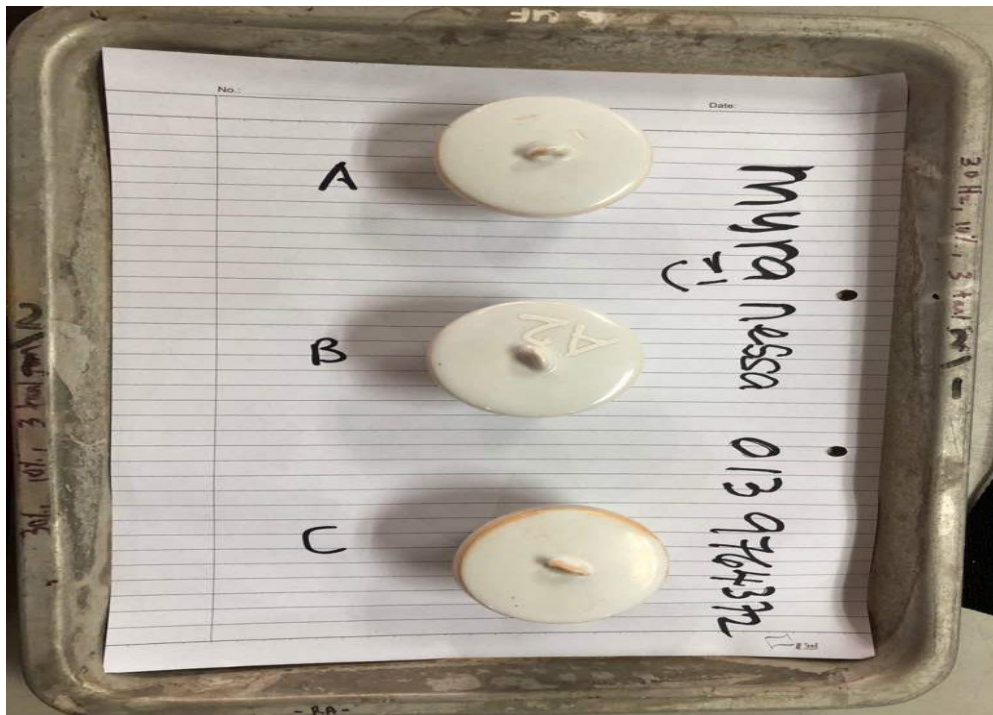


Figure 3.4: The sample was mark after recorded its weight