# Physical and Chemical Properties of Carbonaceous Shale and Their By --Product along Seputeh -- Batu Gajah New Road

By

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A project dissertation submitted to the Chemical Engineering Programme Universiti Teknologi PETRONAS in partial fulfilment of the requirement for the BACHELOR OF ENGINEERING (Hons) (CHEMICAL ENGINEERING)

Approved by,

(Assoc Professor Askury Abd Kadir)

#### UNIVERSITI TEKNOLOGI PETRONAS

#### TRONOH, PERAK

January 2010

## **CERTIFICATION OF ORIGINALITY**

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

ANGGA PRATAMA BIN HERMAN

## ABSTRACT

The carbonaceous shale of Batu Gajah experiencing weathering and acid rock drainage phenomenon doe to exposure to water and atmosphere during the construction of the Seputeh - Batu Gajah new road. As a result, severe soil cancer and land slide occur around the affected area. Sets of sample were collected according to the weathering profile in order to determine the change of physical and chemical properties of the carbonaceous shale before and after experiencing weathering and acid rock drainage. Several experiments such as colour chart. physical observation, X - Ray Diffraction (XRD), X - Ray Fluorescence (XRF), Scanning Electron Microscope (SEM), CHNS elemental experiment and density calculation was carried out in order to meet the project objective. It is found that the physical and chemical properties of the carbonaceous shale change during weathering and acid rock drainage. The density gradually decreased whiles the colour of the rock which dark becomes lighter. The carbonaceous shale which initially hard becomes more brittle and porous as it weathered. The chemical properties of the carbonaceous shale such as carbon and sulphur are significantly decreases because the elements are leach out during the weathering and acid rock drainage process. As a result, the change in physical and chemical properties due to weathering and acid rock drainage are heavily affected the area which cause soil cancer and land slide.

Keyword: Carbonaceous Shale, Acid Rock Drainage, Weathering

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## ABBREVIATION AND NOMENCLATURE

- ARD Acid Rock Drainage
- XRD X Ray Diffraction
- XRF X Ray Fluorescence
- SEM Scanning Electron Microscope
- UTP Universiti Teknologi PETRONAS

## **CHAPTER 1**

## INTRODUCTION

### 1.1 Background of Study

This project is about to determine the physical properties of the carbonaceous shale that can be found along Seputeh – Batu Gajah road. Along this road, there were some environmental issues at the left and the right side of the road. Due to the construction of the new road, the carbonaceous shale is exposing to the atmosphere when the hill is excavated during this process.

Due to the exposure to the environment and atmosphere, the carbonaceous shale can cause rock weathering and also Acid Rock Drainage. Rock weathering happens naturally while acid rock drainage is produced by the oxidation of sulphides minerals such as pyrite and marcasite (FeS<sub>2</sub>). Both of these phenomenons can affect the physical and chemical properties of the carbonaceous shale

Acid Rock Drainage is found around the world both as a result of naturally occurring processes and activities associated with land disturbance such as highway construction and mining where acid-forming minerals exposed to the air and water. These acidic can cause metal in geologic material to dissolve, which can cause a very serious contamination and damage the environment for the flora and fauna around the area.

Rock weathering also happens naturally which it can divide into physical and chemical weathering. Since physical weathering only occurs in a very arid location, chemical weathering is the only factor that contributes to the change of chemical and physical properties of the carbonaceous shale. The importance of this project is to give the idea about what cause the environmental issues in the physical and chemical properties point of view and give the solid analysis about this problem.

#### 1.2 Problem Statement

Carbonaceous shale generally strong in nature, however due to the construction of the Seputeh - Batu Gajah new road, the carbonaceous shale were expose to the surface because of the hill excavation during the construction. The exposure to the water and atmospheric oxygen can cause the properties of the carbonaceous shale along the Seputeh – Batu Gajah new road changed.

This acidic condition can cause metals in geologic material to dissolve, which can affect ground water quality and also can lead to what known as 'soil cancer'. As a result, the carbonaceous shale that initially is black in colour will change to rusty brown and this acidic condition will prevent the growth of plants around the area. This phenomenon can affect the strength of the soils and also lead to landslide and other land problems

The exposed carbonaceous shale also experience rock weathering and it indicates that the grass can grow at the weathered shale. Since the rock weathering takes some time to occur, the land structure or the exposed carbonaceous shale that affected by the ARD earlier are weak and exposed the area to the environmental issues as stated earlier. The project is about to indicate the Physical and Chemical properties of the carbonaceous shale of fresh to weathered sample in order to determine cause of the environmental issues happens at the area.

#### 1.3 Objective and Scope of The Study

The scope of study is around the physical and chemical properties of the carbonaceous shale and it's by – product. Sets of experiment are planned where the results of the experiment will be compared with the reference properties that found at the literature review. The reference properties are referred as the collection data from the various carbonaceous shale data that gathered around the world.

The significant difference in the results may determine the cause of the environmental issues around the study area which also as the by-product of the carbonaceous shale itself. Thus, the objectives of the study are:

- To determine the physical and chemical properties of carbonaceous shale for fresh and weathered sample.
- Compared and analysed the results obtain with the reference data and determine the by-product of the carbonaceous shale after ARD and weathering.

## **CHAPTER 2**

## LITERATURE REVIEW

#### 2.1 Geology of the Project Area

This project area is located at Batu Gajah, Perak. Batu Gajah is on of the territory of Kinta District which was well known as the important tin producing area in Malaysia. Kinta Valley tin output of ore in 1950 was half of the total Malayan output and about 17 per cent of the world's total production during that time [1].

Kinta Valley not only famous by tin production but also famous with the attractive geological structure. Kinta Valley is well known as the place where there has been disagreement in the interpretation amongst the geologist. The famous subjects of the argument were the origin of the Gopeng Beds and the succession of the sedimentary rocks.

Geographically, Kinta Valley is located in between two granite mountain range which is the Kledang Range to the west and the Main Range to the east which the valley gradually widening towards the south. The valley is watered by the Kinta River which flows through the valley to the south and the drainage pattern of the Kinta Valley can be described as 'herringbone pattern'. [1]

#### 2.2 Black Shale

Black shales are fissile, and has many splits readily into thin, slightly flexible sheets of large size. They are exceptionally rich in organic material and they also tend to rich in iron sulphide, usually pyrite, which replaces fossils, forms nodules, or occurs as finely disseminated grains [2].

Black shales contains an unusual amount of organic or carbonaceous matter in table 1. The average shale has about 1 % carbon where the black shales organic content ranges from 3 to 15 %. Some rare or unusual trace elements such as Vanadium (V), Uranium (U), Nickel (Ni), and Copper (Cu) can be found inside the black shale and some black shale even has been exploited because of their unusual metal content such as the Kupferschiefer shales at Mansfeld in Germany [3]. The oxidation of the iron sulphides can cause a swelling and disintegration of the rock in outcrops and covers the surface of the rocks with a white efflorescence.

The black shales contains of coarse and fine detrital materials which all of these components will form the bulk of the rock, pyritic matter, and a carbonaceous fraction inside the black shale. This characteristic gives the black shale its distinct character such as one third of the black shales is silt, another third is the clay material while the remaining third is the pyritic matter. However the pyritic fraction varies greatly in abundance, forming in extreme cases as much as 38 percent, as in the Wauseca "graphitic slate" in Iron County, Michigan [3].

Black shales occur in many places in the world and formed many times during the geologic past. Some famous black shale is the older Precambrian "graphitic slates" of Michigan, the younger Precambrian Nonesuch shale of Michigan, and Permain Mansfeld Kuperschiefer of Germany [2].

Constituent	A	В	С	D	E	F
SiOž	51.03	60.65	36.67	58.03	63.09	33.15
TiO <sub>2</sub>		0.62	0.39	0.64	0.99	
Al <sub>2</sub> O <sub>3</sub>	13.47	11.62	6.90	15.00	18.58	19.3
Fe <sub>2</sub> O <sub>3</sub>	8.06	0.36	a	3.67	2.17	2.60 <sup>6</sup>
FeO			2.35 <sup>a</sup>	5.82	2.73	2.60 <sup>b</sup>
MnO	• • • •	0.04	0.002	0.09	0.22	0.25 <sup>c</sup>
MgO	1.15	1.90	0.65	1.64	2.67	1.00
CaO	0.78	1.44	0.13	0.26	1.11	10.4
Na <sub>2</sub> O	0.41	0.60	0.26	3.52	4.54	1.00
K <sub>2</sub> O	3.16	3.10	1.81	3.60	0.54	3.00
$H_2O + 1$		c 3.77	1.25	3.46		
Į	0.81	Į		ļ	2.69	1 <b>.70</b>
H <sub>2</sub> O -		1.19	0.55	0.84		
$P_2O_5$	0.31	0.18	0.20	0.16	0.12	• • • •
CO <sub>2</sub>		1.65	• • • •	0.03	• • • •	9.24
S	7.29	3.20 <sup>d</sup>		0.04	Trace	2.31
С	13.11	9.20	7.28	3.27		9.06 <sup>e</sup>
FeS <sub>2</sub>			38.70	••••		
V <sub>2</sub> O <sub>5</sub>	• • • •		0.15	••••	****	
Total	102.90 <sup>f</sup>	99.52	100.21	100.24 <sup>g</sup>	99.45	91.01

The table below shows the chemical composition of some of the famous known black shale around the world.

#### Table 1: Chemical Composition Carbonaceous Shales (per cent) [3]

- A. Black shale (Devonian), Dry Gap, Walker County, Georgia. L. G. Eakins, analyst. Includes 3.32 hydrocarbons (Diller, 1898).
- B. Ohio shale (Devonian), Logan County, Ohio. Downs Schaaf, analyst (lamborn, Austin and Schaaf, 1938, p. 20).
- C. "Graphitic slate" Wauseca member Dunn Creek slates (Precambrian) 10<sup>th</sup> level Buck Mine, Iron River District, Michigan. C. M. Warshaw, analyst (James, 1951, p 255).
- D. Black slate, Dunn Creek slates (Precambrian), Crystal Falls district, Michigan. R. H. Nanz, Jr., analyst (Nanz, 1953).
- E. Nonesuch shale (Keweenawan), Michigan (Lane, 1911).
- F. Kupferschirfer, Mansfeld, Germany (Stelzer and Bergerat, 1904).

<sup>a</sup> Direct determination not possible because of organic matter; iron in excess of pyrite reported as FeO.

<sup>&</sup>lt;sup>b</sup> Iron.

<sup>°</sup> Manganese.

<sup>&</sup>lt;sup>d</sup> FeS<sub>2</sub>.

<sup>&</sup>lt;sup>e</sup> Bitumen.

<sup>&</sup>lt;sup>f</sup>Less O = S; total loss becomes 100.17.

<sup>&</sup>lt;sup>g</sup> Includes 0.14 SO<sub>3</sub>.

The origin of the black shale has been much debated. However some claim that the black shale certainly deposited under anaerobic conditions but how such condition were achieve is less certain. More study need to be conducted in order to determined the origin of the black shale.

#### 2.3 Acid Rock Drainage

ARD formation is recognised as a complicated process and it happen naturally as shown in Figure 1. The reaction of pyrite with oxygen and water will produce a solution of ferrous sulphate and sulphuric acid. Ferrous ion can further be oxidized to produce additional acidity. Iron and sulphur oxidizing bacteria are known to catalyse these reactions at low pH thereby increasing the rate of reaction by several orders of magnitude [4].

When pyrite is exposed to the oxygen and water is oxidized, resulting in hydrogen ion release, acidity, sulphate ions and soluble metal ions as shown in equation (1).

$$2FeS_2(s) + 7O_2 + 2H_2O \rightarrow 2Fe_2 + + 4SO^{2-} + 4H^+$$
(1)

Further oxidation of ferrous ions to ferric ions occurs when sufficient oxygen is dissolve in the water or when the water exposed to the sufficient atmospheric oxygen as shown in equation (2). Equation (3) shows the precipitates of ferric hydroxides and release more acidity. At this stage, a red – orange precipitate seen in waters affected by the ARD.

$$4Fe^{2+} + 4H^+ + O_2 \rightarrow 4Fe^{3+} + 2H_2O$$
<sup>(2)</sup>

$$\operatorname{Fe}^{3+} + 3\operatorname{H}_2\operatorname{O} \rightarrow \operatorname{Fe}(\operatorname{OH})_3(s) + 3\operatorname{H}^+$$
 (3)

When ferrous ion is produce, equation (4) and sufficient dissolve oxygen is present in the cycle of reactions (2) and (3) is perpetuated. Without dissolve oxygen equation (4) will continue to completion and waters will shows elevated levels of ferrous ions [4].

$$FeS_2(s) + 14Fe^{3+} + 8H_2O \rightarrow 15Fe^{2+} + 2SO^{2-}_4 + 16H^+$$
 (4)

The rates of chemical reactions in equation (2), (3), (4) can be significantly accelerated by bacteria, especially *Thiobacilus Ferrooxidans*. Another microbe, *Ferroplasma Acidarmanus*, has been identified in the production of acidity in mines water [4].

The overall rates of acid production is increased by several orders of magnitudes and the bacteria rapidly catalyzed the process by oxidizing more ferrous ions to ferric ions as shown in equation (5). The process will proceed to equation (6) and large amount of acids associated with the release of the heavy metals into the solution. At this stage, the ARD becomes a problem [5].

$$\mathrm{Fe}^{2+} + \mathrm{O}_2(\mathrm{aq}) \to \mathrm{Fe}^{3+} \tag{5}$$

$$Fe^{3+} + FeS_2(s) \rightarrow Fe^{2+} + SO^{2-}$$
 (6)



Figure 1: The effect of ARD on the carbonaceous shale

#### 2.4 Rock Weathering

Weathering is a phenomenon of breakdown and alteration of materials near the earth's surface to products that are more in equilibrium with newly imposed physico-chemical conditions. There are many proposed definition of weathering but the most widely accepted definition is 'Weathering is the response of the material which were in equilibrium within the lithosphere to conditions at or near its contact with the atmosphere, the hydrosphere, and perhaps still more importantly, the biosphere' [6].

The rock weathering is controlled by the porosity and the permeability of the rock itself. The porosity and the permeability will determine how ease the water can enter the rocks and the weathering products can be removed. Some granular sediment which has high porosity can make every mineral grain is exposed to weathering while some rocks that have no intergranular porosity at all, can only weather at the surface of the rocks and along a few widely spaced joint.

Porosity and permeability itself are controlled by the grain packing and the amount of space between grains of the rocks. Black shale is one of the sedimentary rocks and the sedimentary rocks are formed by the compaction and induration of sediments which most of them are marine life. Sedimentary rocks usually made from by a successive layer of sediments called strata or beds. The minerals grains tend to lie flat as the preferred orientation of the grains leads to the direction of water movement through the rock [7].

The beddings planes are planes with fissility between beds which may in contact plane of rock of different composition or rock of one composition. The plane is said to be 'thin bedded' if the planes of the rock are closely spaced and if the planes are wide apart, the beds are said to be thick or massive. In nature, the sedimentary structure may be further complicated by cross bedding and graded bedding [7].

Generally, sedimentary rocks are classified into clastic rocks such as sandstone, shale and conglomerate. The weathering occurs for these types of rock often more intense at the surface of the rock and decrease down the profiles. However the effect will persists along the crack to much greater depth. As a result, the rocks are broken into fragment which has fresh inside but weathered on the surface. The position of the beds also can be a factor how fast the weathering can happen. For instance, a well-bedded horizontal shale is hard for water to penetrate, but if the beds are steeply inclined many shales edges and makes the bedding planes exposed, the water will allowed to penetrated and cause weathering. [7]

Weathering can makes the geological cycle. A rock weathers and the weathering products may be removed mechanically or in solution. The mechanically removed product is called erosion, and the movement materials are called transport. Weathering and erosion together make up the process of denudation and the transport sediment usually deposited in the sea. They accumulated in the sea, compact, experience mineral diagenesis and form sedimentary rocks. Earth movements may later lift such rocks above the sea-level and the cycle can go around again. [8]

Generally, there are two types of weathering which is the physical weathering and chemical weathering. Each type of weathering has unique mechanism on how the weathering happens.

#### 2.5 Physical Weathering

Physical weathering happens when solid rocks are broken into fragments with little or no chemical change happen to the rock itself. Physical weathering occurs in extreme condition such as in a very cold, dry, or very hot places and dry climates. Due to this condition, the splitting of the solid rocks is largely and the physical weathering is more likely to occur. The disintegration of rocks in moist climates region is cause by chemical weathering [8].

Mechanical disintegration of solid rocks without any chemical changes being produced in the mineral components only takes place in a very hot, dry and very cold condition. This extreme condition cause the splitting and fracturing of the solid rocks. The first stage of the disintegration is caused by the joint present in most of the rocks. The joint become weak and vulnerable due to the removal of the pressure when the rocks exposed to the environment [8].

A chemical analysis of the weathered or fragmented rocks will be the same as the solid rocks, however the effect of the disintegration would differ in the various kinds of rocks. The result of the disintegration would be the net of the loose granular mass of mineral material from the compact or solid rocks.

#### 2.6 Chemical Weathering

The earth soil is developed by the weathered rock except at the highest mountains, very cold and very dry region. Each kind of rocks is weathered according to its texture, chemical composition and to the chemical environment. In contrast to physical weathering that occurs at low temperature and arid climates, the chemical weathering dominates at high temperature and abundant water or rainfall [8].

Chemical weathering is a series of surface chemical reaction between the rocks, the atmosphere and water. For instance, a chemical process of silicate reactions to leaching by water occurs at temperature 30 °C and in atmospheric pressure. The end products of weathering are oxidized residuum from the rocks and chemical solutions containing soluble chemical elements of the original rocks. Thus, the chemical weathering can be represented as:

Although the principle processes of the chemical weathering appear to be simple, the weathering processes are affected by numerous variables factors. However the chemical weathering will occur rapidly if there are abundant supplies of the fresh rocks, abundant rain and easily eroded rocks such as sedimentary rocks. [8]

## **CHAPTER 3**

## METHODOLOGY

The project will consist of several experiments which will be conducted in order to determine the properties of the carbonaceous shale. The figure 2 below shows the methodology in executing this project:

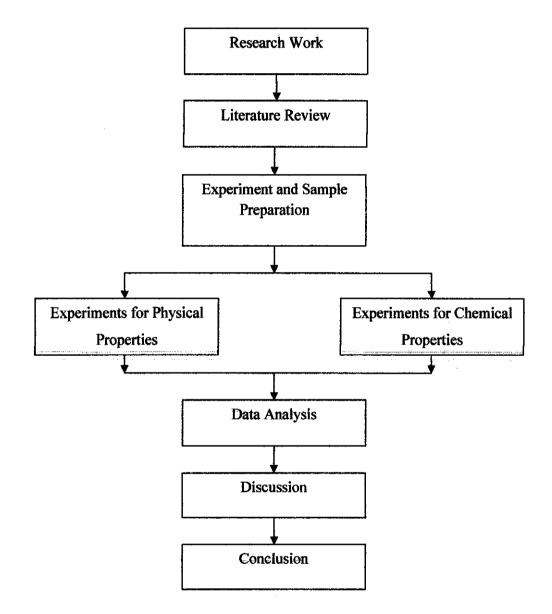


Figure 2: Project methodology in executing the project.

The project starts with a discussion with the supervisor to enlighten the understanding about this project and followed by the research of the related journal, article and books. The sources of the literature are mainly from the UTP library, Minerals and Geosciences Department library and internet.

Preparations for the experiment are carefully planned with all the required procedures are reviewed while the locations of the project are survey and choose accordingly. There are two groups of experiment which is the physical properties experiments and the chemical properties experiments. The table 2 shows the procedures experiments for the project:

Physical Properties Experiment	Chemical Properties Experiment
1. Physical Observation	1. XRD
2. Density Calculation	2. XRF
3. Colour Chart	3. CHNS Experiment
4. SEM	

#### Table 2: List of planned experiments

The locations of the project area need to be determined and three possible locations are chosen around the Batu Gajah – Seputeh road as the possible sampling area. Six samples at each location are gathered and each sample is collected according to specification of the project. The sample is a profile of the carbonaceous shale itself where it gathered by the range of fresh sample to half weathered, to weathered, and to top soil.

The samples later divided accordingly to the experiment requirement, some of the samples need to be air dried and grind into powder. The samples are labeled and arrange to its respective experiments. The results of the experiment will be recorded according to each experiment and analyzed. The results later will be discussed and relate the results with the literature review obtain.

Last step of the project is concluding the findings of all the experiments and determine whether the results of the project meet the objectives of the final year project or not. Some recommendations also need to be given in order to improve the research for future studies.

## **CHAPTER 4**

## THE EXPERIMENTS

## 4.1 Sample Collecting

There are many affected carbonaceous shale located along the slope of the Batu Gajah – Seputih new road, but three locations have been identified as the sampling location for this project. Each location is far away from each other but still inside the area for the project. The locations are labeled as BG 1 for the first location, BG 2 for the second location and BG 3 for the third location as shown in figure 3 below:



Figure 3: The map of Batu Gajah, location of the project and some landmark around the area [9].

Figure 3 above shows the location of the project area. Since the Seputeh = Batu Gajah road is newly constructed road, the exact location of the project area is hard to determine since the actual map of the road are yet to be made. However, the legend of the figure 3 above that consists of some landmark of the area is shown in the table 3 below:

Legend	Landmark
A	Batu Gajah town.
В	• SOA (M) Sdn. Bhd.
С	Clearwater Sanctuary Resort.
D	Kellie's Castle.
E	• Location of BG 1 area, near Taman Bemban Jaya and SOA Sdn. Bhd.
F	• Location of BG 2 area, after the traffic light before Clearwater Santuary.
G	• Location for BG 3 area, near the Kellie's Castle.

Table 3: The legend and the landmark around the project area



Figure 4: The location for BG 1, near the Taman Bemban Jaya.

The first location is near the Taman Bemban Jaya right after the first traffic light. There are large amount of carbonaceous shale exposed at the slope and it indicate that there are no grass grow at the shale in figure 4. At the drainage system along the road, it is found that there are brownish stained at the drain and road as the effect of the acid rock drainage. Along the BG 1 location, there are also the weathered carbonaceous shale at the right hand side and the top of the slope.

The second location, BG 2 located right after the traffic light on the way to Clear Water Sanctuary. The location is an abandon land after the road construction. It noticed that there are also hardly seen grass grow on the carbonaceous shale. As the carbonaceous shale weathered and become soil, the grass starts to grow on it. Figure 5 shows the carbonaceous shale located at the left hand side of the picture and it weathered as it moves to the right.



Figure 5: The location of BG 2, after the traffic light to the Clear Water Sanctuary.

There are also found a location of the affected carbonaceous shale right after the Kellie's Castle and it makes the third location for the project. The location name as BG 3 and the condition of the carbonaceous shale at BG 3 is almost the same as at BG 1. The fresh carbonaceous shale lies under the weathered carbonaceous shale.



Figure 6: The location and condition of BG 3, near Kellie's Castle.

A profile of sample collected from each location based on weathering profile, where the samples collected from the fresh carbonaceous shale sample to the top soil sample. The sample collected by digging the predetermine spot at each location about 5 inch in depth using a pick. The sample then labeled according to the requirement table 4 below.

	<b>BG</b> 1	BG 2	BG 3
Criteria	Label	Label	Label
Fresh Sample	Spot 1	Spot 1	Spot 1
Half weathered 1	Spot 2	Spot 2	Spot 2
Half weathered 2	Spot 3	Spot 3	Spot 3
Weathered 1	Spot 4	Spot 4	Spot 4
Weathered 2	Spot 5	Spot 5	Spot 5
Top Soil	Spot 6	Spot 6	Spot 6

Table 4: The label for each spot at each location

#### 4.2 Sample Preparation

The sample later prepared for the coming experiments after gathered from the each location. Some of the samples need to be dried in an oven for a day or 24 hours at temperature 105 °C and later grind into powder while some of the samples remain undisturbed. The samples place inside a container and later divided to its respective experiments and ready to be tested such in figure 7.



Figure 7: Samples divided to each experiments and ready to be tested.

#### 4.3 Density Calculation

# 4.3.1 Objective

Density is one of the essential physical properties that differs in every substance, thus the objective of this experiment are:

- a) To determine the density of every samples at every location.
- b) To analysed and compare the pattern of the densities of every sample at every location.

# 4.3.2 Theory

Each substance has its own density and it been consider as one of the physical properties of the substance. Density means the mass per unit volume of the substance. A density of pure substance which is pure solids and pure liquid are independent on pressure and varies slightly on temperature [10]. In the other hands, the density can be shown as:

# Density = Mass / Volume

In this experiment, each sample in every location will be tested to determine its densities. Takes the pressure and temperature to be constant which is at atmospheric pressure and room temperature, the experiment will determine all the sample in every profiles. The result of the experiment will be analysed and the pattern of the densities in every samples will be compared.

# 4.3.3 Experiment Procedure

- 1. Zero the balance of the weight and measure the mass of the rock. Record the value in the table.
- 2. Take a measuring cylinder and fill the measuring cylinder with water. Record the value as 'Volume 1' in the table.
- 3. Slide in the sample and take the reading from the measuring cylinder. Record the value as 'Volume 2' in the table.
- 4. Calculate the density and record in the result table provided.

#### 4.3.4 Results and Discussion

The table 5 below showed the results of the density calculation for BG1 and the complete result can be found in appendix A. The results later being convert into standard unit which in kilogram per unit volume and it being rounded into three decimal points.

Profile	Mass	Volume	1 Volume 2	Volume	Density	Density
	(g)	(ml)	(ml)	(ml)	(g/ml)	(Kg/m3)
Spot 1	5.90	30.00	33.00	3.00	1.97	0.00197
Spot 2	7.20	30.00	34.00	4.00	1.80	0.00180
Spot 3	3.20	30.00	32.00	2.00	1.60	0.00160
Spot 4	5.10	30.00	33.00	3.00	1.70	0.00170
Spot 5	4.60	30.00	33.00	3.00	1.53	0.00153
Spot 6	2.90	30.00	32.00	2.00	1.45	0.00145
		1				

Table 5: The results for density calculation for BG 1

The data in table 5 above plotted into a graph for better representation. For an example, the density for BG 1 is decreasing as it moves from spot 1 to spot 6 as it shows in figure 8 below. It appears the weathered carbonaceous shale has lower density compare to the fresh carbonaceous shale. As the rock weathered, some of chemical elements inside the carbonaceous shale leach out by water with the helps of temperature and atmospheric pressure. The end products of the weathering are oxidized residuum from the rock and chemical solution containing soluble chemical elements of the carbonaceous shale.

The decreases in density also help by the effect of acid rock drainage which can found around the study area which is BG 1, BG 2 and BG 3. The ARD drains the iron and sulphur element from the carbonaceous shale. As a result, the weathered carbonaceous shale density becomes much lower as it goes down the profile due to the weathering and ARD.

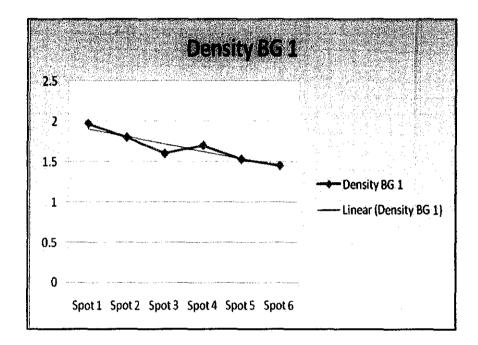


Figure 8: The graph of density of BG 1 profile.

# 4.3.5 Conclusion

In summary, the density of each sample of every profile can be determine as the complete results is shown in the appendix A. Overall, the density of the carbonaceous shale decreases as it weathered because of the weathering and the effect of the ARD.

#### 4.4 Physical Observation and Colour Determination

# 4.4.1 Objective

Physical observation is important in order to describe the physical description of the rock. Generally, each rock has its own physical properties such as colour, porosity and grain size. In this experiment, the samples will be describing physically and classed based on its colour group using the Rock Colour Chart. Thus, the objectives of the experiment are:

- a) To observe and describe the physical of the samples.
- b) To class the samples according the Rock Colour Chart.

# 4.4.2 Theory

The Rock Colour Chart are prepared by The Rock-Color Chart Committee which representing already been the indispensable tool for geologist since 1948. The Rock Colour Chart is widely used around the world as a standard references for rock colours.

The Rock Colour Chart contains a forms and arrangements of rock colour chips based on Munsell system. In this system, there are three important terms such represented in figure 9 which have been used to classify the colour of the rock which are:

- 1. Value which represent the properties of lightness.
- 2. Hue which represent the colour of the rock.
- 3. Chroma which represent the degree of saturation of the colour.

**Final Year Project** 

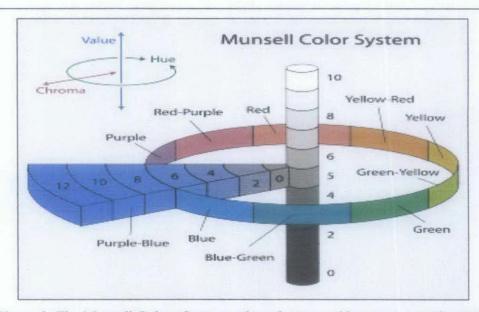


Figure 9: The Munsell Colour System value, chroma and hue representation [11].

The Hue designated by a number and letter such as 5R or 10YR while the Value and Chroma are numbered so the colour of the rock can be given by a numerical designation such as 5R 6/4 and 10YR 8/2. There are cases where the colour of the rock is lying between two colour chips on the chart, it also can be determined. For instance, if the rock colour lying between light red (5R 6/6) and moderate red (5R 4/6), it can be designated as 5R 5/6.

The Rock Colour Chart can be used either with dry rocks or wet rocks specimens. The wet rock usually will decrease the value where it will make the specimens darker, but do not change the chroma. [12]

# 4.4.3 Experiment Procedure

The samples are taken from three different locations which labelled as BG 1, BG 2 and BG 3. Each location has a profile of six or seven samples. Thus, the experiment procedures are:

- The rock samples are labelled before being dried inside an oven at 105 °C for 24 hours.
- 2. The samples one by one describe its physical characterization and record the findings.
- Place the sample at The Rock Colour Chart and determine the colour of the sample.
- 4. Record the results along with the physical characteristics earlier.

# 4.4.4 Results and Discussion

After observing the result such in appendix B, it is found that the colour of the carbonaceous shale is changing due to weathering in quite a similar way but there is some difference in the colour between the solid sample and powder sample. Take results for BG 3:

Samples	Picture	Description
Spot 1		<ul> <li>Medium grey which indicated as N5 in the rock colour chart.</li> <li>Physically hard and indicates as fresh sample for BG 3.</li> <li>Do not have obvious layers.</li> </ul>
Spot 2		<ul> <li>Medium light grey which indicated as N6 in the rock colour chart.</li> <li>Slightly weathered as white stain at some part of the rock.</li> <li>Physically hard and do not have layers.</li> </ul>
Spot 3		<ul> <li>Medium light grey which indicated as N6 in the rock colour chart.</li> <li>Slightly weathered as some white and brown stain at some part of the rock.</li> <li>Not too hard and do not have layers.</li> </ul>

Table 6: Physical observation and colour determination for solid BG 3

Spot 4	<ul> <li>White which indicated as N9 in the rock colour chart.</li> <li>Weathered, porous and not too hard. Do not have layers also.</li> </ul>
Spot 5	<ul> <li>Yellowish grey which indicated as 5Y 8/1 in the rock colour chart.</li> <li>Weathered, porous and brittle, do not have layers.</li> </ul>
Spot 6	<ul> <li>Very pale orange which indicated as 10YR 8/2 in the rock colour chart.</li> <li>Top soil of the profile, brittle, porous and do not have layers.</li> </ul>

Table 6: Continue, the physical observation and colour determination for solid

# BG 3

From table 6 above, in is found that the value which represent the properties of lightness of the carbonaceous shale is increasing from N5 (Medium Grey) for fresh sample to N6 (Medium Light Grey) for spot 2 and 3. The value becomes N9 (white) at spot 6 where the carbonaceous shale is completely weathered.

At spot 5, the colour of the carbonaceous shale becomes more 5Y 8/1 (Yellowish Grey) and the colour change again as it reach top soil condition which is 10YR 8/2 (Very Pale Orange). The results for BG 3 such in the table 6 above is almost the same with the other two profiles BG1 and BG 2 which can be view at appendix B.

It is not only the colour of the carbonaceous shale changes as it weathered and experience ARD, the physical properties of the carbonaceous shale changes too. According to table 6, spot 1 and spot 2 samples are hard and the hardness of the sample decreasing from spot 3 and the pattern continues as it moves to weathered sample. Weathered sample physically brittle, porous and easy to destroy which can contribute to the landslide at the area.

However, the colour of the carbonaceous shale can better represent in powder form. The colour of the carbonaceous shale more vibrant compare with solid sample because of the powder sample free from any stain. The results for powder sample for BG 3 can be represented in table 7 below:

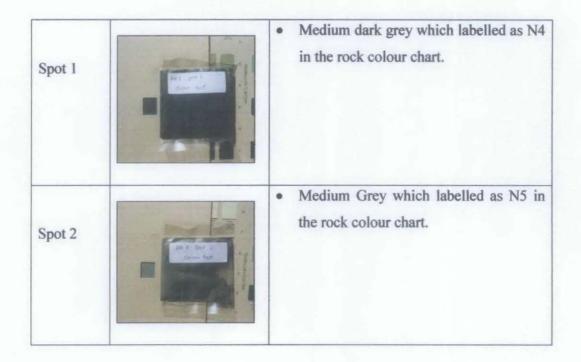


Table 7: The physical observation and colour determination for powder BG 3

Spot 3		Medium light grey which labelled as N6     in the rock colour chart.
Spot 4		White which labelled as N9 in the rock colour chart.
Spot 5	ALL DE T	• Yellowish grey which labelled as 5Y 8/1 in the rock colour chart.
Spot 6		• Very pale orange which labelled as 10YR 8/2 in the rock colour chart.

Table 7: Continue, the physical observation and colour determination for powder

BG 3

The fresh sample colour for powder sample is N4 (Medium Dark Grey) and it continuously increase in the value which shows the colour of the sample become lighter, N5 (Medium Grey) for spot 2, N6 (Medium Light Grey) for spot 3 and N9 (White) for spot 4. Similar to solid sample, at spot 5 and spot 6, the carbonaceous shale appear to have orange colour such 5Y 8/1 (Yellowish Grey) and 10YR 8/2 (Very Pale Orange).

# 4.4.5 Conclusion

The carbonaceous shale colour are initially dark in colour gradually becomes lighter as the carbonaceous shale experience weathering and ARD. The weathering and ARD phenomenon also affected the physical condition of the shale which initially the carbonaceous shale is hard becomes more brittle and porous.

#### 4.5 CHNS Elemental Experiment

# 4.5.1 Objective

CHNS is an experiment that can determine the composition of carbon, hydrogen, nitrogen and sulphur for a homogeneous organic material. The CHNS analyser provides the determination for various kinds of materials such as solids, liquids, volatile and even viscous samples.

The composition of carbon, hydrogen, nitrogen and sulphur of the black shale can be determined using this method. The samples were taken from three different locations at Batu Gajah and a profile of samples was taken for every locations. The samples need to be grind first and then dried before start the experiment.

The result of the experiment will be analysed and discussed later. Thus, the objective of this experiment is:

- a) To analyse and to determine the composition of carbon, Hydrogen, nitrogen and sulphur of the black shale.
- b) To describe the composition pattern among the samples.

#### 4.5.2 Theory

High temperature combustion is used in removing the elements from the tested sample. In the combustion process, a maximum 2 milligram (mg) sample is encapsulated in silver capsule. The sample is then placed in the sample loading chamber and held until a dosage of oxygen is released. The sample is then dropped into the furnace at the same time the oxygen arrives. The sample is combusted by the heated oxygen – rich environment.

High temperature is used in removing the elements from the tested samples. A maximum 2 milligram (mg) sample is encapsulated in a silver vial and then placed in the sample loading chamber and held until a dosage of oxygen is released. The sample later dropped into the combustion chamber and combusted by the heated oxygen rich environment.

At 1000 °C, carbon is converted to carbon dioxide, hydrogen is converted to water, nitrogen to oxides of nitrogen and sulphur to sulphur dioxide. The combustion product then swept away from the combustion chamber by the carrier gas such as helium and passed over heated high purity copper. The copper is used to remove any oxygen that not combusted during the first combustion and also to convert any oxides of nitrogen to nitrogen gas.

The product gasses then passes through the absorbent trap to make sure only carbon dioxide, water, nitrogen gas and sulphur dioxide left for the detection. For over thirty years, the CHNS elemental analyser has been used in analytical laboratories. This method is used extensively through a wide range of application such as pharmaceutical, geology and chemical. The analyser is a important tool for elemental analysis which can handle many types of samples in wide range of application fields [13].

#### 4.5.3 CHNS Experiment Procedures

# 4.5.3.1 Experiment Preparation

- 1. Switch on the CHNS equipment.
- 2. Run 3 blank analyses before starting the experiment.
- 3. Prepare three sets of standard samples and run the samples analyses.
- 4. Proceed the experiment with the samples if the results of the standard analyses meet the standard requirement.
- 5. Record the result after finished the analyses.

# 4.5.3.2 Sample Preparation

- 1. The samples need to be grind as small as possible and also need to be dried before the sample can be prepared.
- 2. First weight the silver capsule and record the weight.
- 3. Take the Sulfamethazine and place it inside the silver capsule that we weight earlier.
- 4. Make sure the weight of the sample inside the not exceed 2 mg.
- 5. Crimp the top of the capsule, then flatten and compact it before weight it again to get the actual weight and record the weight.
- To prevent contamination, wear gloves, and handle the capsule with a tweezers during the experiment.

# 4.5.3.3 CHNS Experiment

- 1. Enter the weight to the equipment and load first the standard sulphonamide into the loading head or carousel.
- 2. Begin the experiment for the standard sulphonamide
- 3. Record the result for the standard sulphonamide and compare with the standard CHNS chemical reading. If the result is acceptable, then only can proceed with the actual sample.
- 4. The standard chemical for CHNS reading is shown as follows [13]:
  - Carbon : 51.78%
  - Hydrogen : 5.07%
  - Nitrogen : 20.13%
  - Sulphur : 11.52%

#### 4.5.4 Results and Discussion

Before the experiment can be started, the CHNS elemental analyser must first being tested by the standard solution which is sulphonamide. The results of the standard solution will determine whether the equipment is ready to be operated or not. The experimental results should be as close as possible to the theoretical results.

The table 8 below shows the experimental results for the standard sulphonamide after three times run. From the readings below, it was found that the experimental value is in good agreement with the theoretical value. If the results of the standard solution testing are not acceptable, the experiment can not be conducted and further calibration of the elemental analyser need to be conducted.

#### Sulphonamide

No	Sample	%С	%Н	%N	% S	wt (mg)
1	sulphonamide	50.14	4.367	16.52	12.21	1.756
2	sulphonamide	50.86	4.882	20.65	12.33	1.646
3	sulphonamide	51.02	4.913	20.67	12.49	1.95
	STANDARD	51.78	5.07	20.13	11.52	

# Table 8: The experimental results and the theoretical value of the standard sulphonamide solution

The results of the elemental analyser show the percentage of the carbon, hydrogen, nitrogen and sulphur content inside the carbonaceous shale. The complete results can be found in appendix C. Generally, there are changes in properties in the carbonaceous shale along the profile. Take results of BG 3 for instance:

No	Sample	% C	%Н	%N	% S	wt (mg)
1	BG 3 Spot 1	5.594	0.628	0.289	0.107	1.937
2	BG 3 Spot 1	6.741	0.797	0.368	0.105	1.899
3	BG 3 Spot 1	8.320	0.726	0.373	0.125	1.851
	Average Spot 1	6.885	0.717	0.343	0.112	1.895
1	BG 3 Spot 2	2.234	0.451	0.224	0.047	1.914
2	BG 3 Spot 2	5.556	0.403	0.272	0.007	1.860
3	BG 3 Spot 2	2.060	0.491	0.229	0.004	1.805
	Average Spot 2	3.283	0.448	0.242	0.019	1.859
1	BG 3 Spot 3	1.916	0.723	0.250	0.026	1.865
2	BG 3 Spot 3	1.484	0.490	0.198	0.039	1.803
3	BG 3 Spot 3	1.817	0.716	0.230	0.026	1.928
	Average Spot 3	1.739	0.643	0.226	0.030	1.865
1	BG 3 Spot 4	0.106	0.683	0.196	0.064	1.976
2	BG 3 Spot 4	0.048	0.706	0.216	0.009	1.783
3	BG 3 Spot 4	0.041	0.663	0.227	0.045	1.904
	Average Spot 4	0.065	0.684	0.213	0.039	1.888
1	BG 3 Spot 5	0.117	0.765	0.163	0.012	1.947
2	BG 3 Spot 5	0.150	0.757	0.156	0.003	1.838
3	BG 3 Spot 5	0.083	0.775	0.167	-0.015	1.844
	Average Spot 5	0.117	0.766	0.162	0.000	1.876
1	BG 3 Spot 6	0.256	0.749	0.190	0.005	1.857
2	BG 3 Spot 6	0.184	0.728	0.180	0.011	1.712
3	BG 3 Spot 6	0.434	0.748	0.193	-0.002	1.879
	Average Spot 6	0.291	0.742	0.188	0.005	1.816

Table 9: the results of elemental analysis of BG 3

The table 9 above shows the complete result for BG 3. Each spot tested three times and the average of the result later calculated manually. The result were rounded into three decimel points and the results can be briefly intepreted using graph. The data later plotted into graph to make the interpretation more easy and useful.

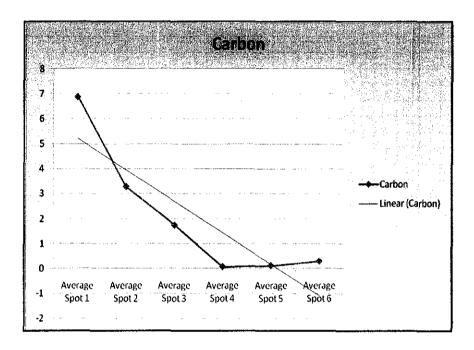


Figure 10: The graph for carbon percentage along the BG 3 profile.

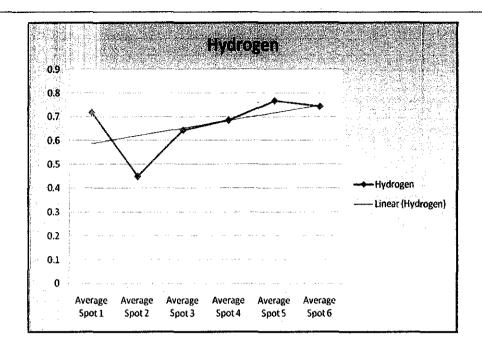


Figure 11: The graph for hydrogen percentage along the BG 3 profile.

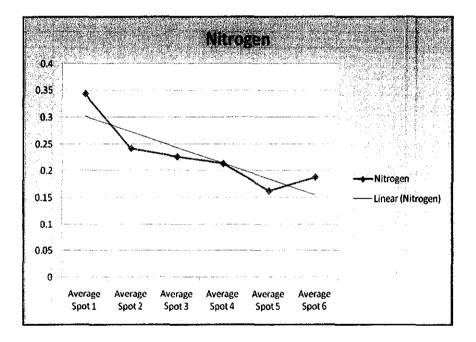


Figure 12: The graph for nitrogen percentage along the BG 3 profile.

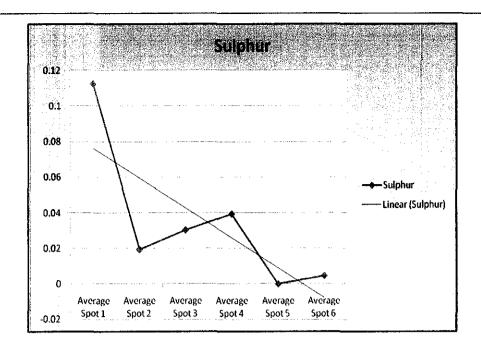


Figure 13: The graph for sulphur percentage along the BG 3 profile.

The data collected from the table 9 are represented in form of graph such as figure 10 for carbon, figure 11 for hydrogen, figure 12 for nitrogen and figure 13 for sulphur. The graph later constructed a trending line using linear regression method to determine the trend of the results obtained for BG 3 samples.

The carbon percentages of the fresh carbonaceous shale are initially 6.885% and as the carbonaceous shale experience weathering, the percentages of the carbon are decreasing as in table 10 and almost depleted at spot 4. The carbon increase again at spot 5 and 6 but the percentage of the carbon at spot 5 and spot 6 are very low.

It is found that the carbon percentage inside the carbonaceous shale in figure 10 decreasing from spot 1 to spot 4. However, there are slightly increase in carbon percentage at spot 5 and spot 6 but the increment are so little. The graph in figure 10 shows a better represented by implementing the trending line and the trending line shows the carbon percentage is decreasing from spot 1 to spot 6.

Chemical weathering dominates at high temperature and abundant water or rainfall [8]. These factors can be applied at the Batu Gajah area as the areas always receive heavy rainfall. As the carbonaceous shale is exposed to the atmosphere and water, the carbonaceous shale experience chemical weathering where in figure 10, the carbon content inside the carbonaceous shale is decreasing. The carbon react with oxygen, water and pressure to produce oxidized residuum and also soluble compound [8].

The hydrogen content of the carbonaceous shale is increasing as shown in figure 11 but the increasing of the hydrogen is not significant or in the other words, the results are more or less the same. From the result obtain in table 9, the percentage of hydrogen is range from 0.4% to 0.8% which means the hydrogen content in the carbonaceous shale are not effected much by either weathering or acid rock drainage.

The nitrogen shows in figure 12 is decreasing in trend lines as the nitrogen in the carbonaceous shale decrease from spot 1 to spot 6. From results table 9 shows the highest percentage of nitrogen is 0.343% and the lowest is 0.162% which means the hydrogen does not severely affected by the weathering and also acid rock drainage.

Sulphur is one of the main elements that involve in the acid rock drainage. Sulphur can react with iron in presence of oxygen and water to produce hydrogen ions, acidity, sulphate ions and soluble metal ions in the ARD [4]. This is the reason why is the percentage of sulphur in the carbonaceous shale are decreasing.

From figure 13, the sulphur decrease from 0.112% at spot 1 to 0% at spot 5 and increase again at spot 6. This trend shows that the sulphur that initially abundant in fresh sample, spot 1 and gradually decreased with respect to weathering and depleted at the end of the profile.

# 4.5.5 Conclusion

As a summary, the percentage of carbon, hydrogen, nitrogen and sulphur successfully determined. It is found that the percentage of carbon significantly decreased and almost depleted at the end of the weathering profile. The percentage of sulphur also gradually decreased as the rock weathered due to ARD and heavily affected the area surround the carbonaceous shale.

#### 4.6 XRF Experiment

#### 4.6.1 Objective

XRF is a powerful tool to qualitatively and quantitatively elemental analysis of environmental, geological, biological and also industrial types of sample. The advantage of using XRF is the method doesn't destroy the samples. It also provides a uniform detection limit along the periodic table and also cover a wide range of concentration which from 100% to few parts per million (ppm).

Thus, the objective of the XRF experiment is to:

- a) To determine the chemical composition of the carbonaceous shale.
- b) Compare the chemical composition of the carbonaceous shale with the reference composition and determined the difference.

# 4.6.2 Theory

XRF instruments works by exposing a sample to be measured to the beam of X – rays. The atoms of the sample absorb the energy from the X – rays, become temporarily excited and emit secondary X – rays. Each chemical element emits a unique X – rays at a unique energy. By measuring the intensity and characteristics energy of the emitted X – rays, an XRF analyser can provide qualitative and quantitative analysis regarding the composition of the tested material [14].

An electron can be ejected from its atomic orbital by the absorption of a light wave (photon) of sufficient energy. The energy of the photon (hv) must be greater than the energy with which the electron is bound to the nucleus of the atom. When an inner orbital electron is ejected from an atom, an electron from a higher energy level orbital will be transferred to the lower energy level orbital. During this transition a photon maybe emitted from the atom. This fluorescent light is called the characteristic X-ray of the element [14].

The energy of the emitted photon will be equal to the difference in energies between the two orbitals occupied by the electron making the transition. Because the energy difference between two specific orbital shells, in a given element, is always the same characteristic of a particular element, the photon emitted when an electron moves between these two levels, will always have the same energy. Therefore, by determining the energy (wavelength) of the X-ray light (photon) emitted by a particular element, it is possible to determine the identity of that element [14].

# 4.6.3 Results and Discussion

The XRF tests successfully determine the chemical composition of the carbonaceous shale at all profiles. The full results can be viewed at the appendix D where the change of chemical composition can be seen significantly at certain elements. Take example at BG 2 profiles for instance:

					1		
Symbols	Name	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6
MgO	Magnesium Oxide	1.020	0.773	0.44	0.26	0.498	0.45
Al <sub>2</sub> O <sub>3</sub>	Aluminium Oxide	20.01	27.33	30.4	14.1	21.22	21.74
SiO <sub>2</sub>	Silicon Dioxide	68.90	60.99	47.45	79.31	70.79	61.94
P <sub>2</sub> O <sub>5</sub>	Phosphorus Pentoxide	0.872	0.852	0.849	0.835	0.812	0.874
SO₃	Sulphur Trioxide	0.409	0.362	0	0	0	0.316
K <sub>2</sub> O	Pottasium Oxide	6.154	6.91	3.135	2.269	4.558	3.021
CaO	Calcium Oxide	0.168	0.144	0.187	0.154	0.139	0.133
TiO <sub>2</sub>	Titatium Dioxide	1.250	1.32	1.22	0.671	0.914	1.01
V <sub>2</sub> O <sub>5</sub>	Vanadium (V) Oxide	0.154	0.024	0.025	0	0	0
Cr <sub>2</sub> O <sub>3</sub>	Chromium (III) Oxide	0.025	0.0148	0.0166	0.009	0.006	0.003
Fe <sub>2</sub> O <sub>3</sub>	Iron (III) Oxide	0.925	1.128	16.2	2.366	0.9494	10.39
NiO	Nickle Oxide	0.0086	0	0	0	0	0
Rb <sub>2</sub> O	Rubidium Oxide	0.0243	0.0376	0.0218	0.0102	0.0274	0.0183
ZrO <sub>2</sub>	Zirconium Oxide	0.0304	0.0239	0.0419	0.0136	0.0173	0.0289
SrO	Strontium Oxide	0	0.0077	0.0074	0.004	0.0058	0.0056
ZnO <sub>2</sub>	Zinc Peroxide	0	0	0.0051	0	0	0.0049
CuO	Copper Oxide	0	0	0	0.0033	0	0.014
MnO	Manganese Oxide	0	0	0	0	0	0.047

Table 10: The chemical composition for BG 2 profile

The table 10 above shows the chemical composition of the carbonaceous shale for BG 2 profile. The carbonaceous shale for BG 2 contains the quite similar composition with the reference carbonaceous shale such in table 1. The main elements for the fresh carbonaceous shale are MgO,  $Al_2O_3$ ,  $SiO_2$ ,  $P_2O_5$ ,  $SO_3$ ,  $K_2O$ , CaO, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>. These elements presence in the carbonaceous shale from fresh to weathered and up to top soil sample except for SO<sub>3</sub> which completely leach out as the carbonaceous shale weathered.

It is also found that the Fe<sub>2</sub>O<sub>3</sub> also depleting as a results of the acid rock drainage where the iron react with oxygen and water in this phenomenon. There are also some minor elements presences in the carbonaceous shale such as NiO, CuO,  $V_2O_5$  and MnO. However the minor elements such as ZnO, CuO and MnO do not exist in all samples along the profiles, where these elements only presence at the end of the profile which is spot 6, the top soil.

# 4.6.4 Conclusion

In summary, the chemical properties of the carbonaceous shale are quite similar with the chemical properties of the references. The major and minor element that contain in the carbonaceous shale can be determine.

#### 4.7 XRD Experiment

# 4.7.1 Objective

According to the literature, iron sulphides such as pyrite and marcasite  $(FeS_2)$  are the main components that help the ARD phenomenon. In this experiment, the samples will be tested and the results will be analyzed whether the carbonaceous shale sample contains pyrite or marcasite. The objectives of the XRD are:

a) To determine the mineral constituent of the carbonaceous shale samples.

#### 4.7.2 Theory

XRD is a versatile, non – destructive technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactures materials. Every crystalline materials gives a pattern and the same substance will produce the same pattern. In a mixture of a substance each produces its pattern independently of the other. Like fingerprint, the powder diffraction is ideal suit for characterization and identification of a polycrystalline phases [14].

A diffracted beam is produced by such scattering only when certain geometrical conditions are satisfied where represented by either Bragg's law or the Laeu Equations. The resulting diffraction patterns of the crystal are comparing both the positions and the intensities of the diffraction effects and also can produce quick elucidation of the crystal. Analysis of the positions of the diffractions effects lead immediately to a knowledge o the size, shape, and the orientation of the unit cell [15].

#### 4.7.3 Results and Discussion

The figure 14 below is one of the XRD results of the fresh carbonaceous shale for BG 3 profiles. It is found that quartz is the main minerals constituent for the entire sample along the BG 3 profile. The trend also shared by the BG 2 profiles and BG 1 profile. It is because quartz is a very stable mineral and also the main element of the carbonaceous shale.

However, at some spot, it is found that there are some other mineral constituent found in the sample such as kaolite (clay mineral), montmorillonite (clay mineral) lavendulan (secondary mineral in oxidized zone of some copper deposits)[18] and potassium manganese but the existence of these minerals are random. For instance, lavendulan can be found at spot 1 and spot 4 while at spot 2, kaolite and muscovite are found. The full results of XRD can be found in appendix E.

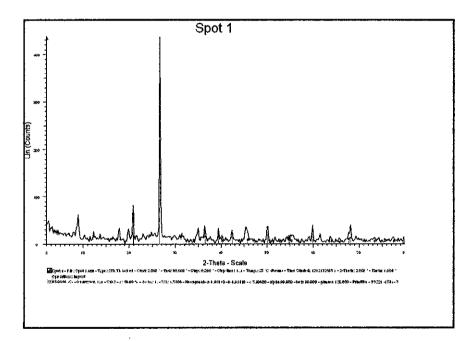


Figure 14: The XRD results for the fresh carbonaceous shale, BG 3 spot 1.

# 4.7.4 Conclusion

In summary, the XRD results are successfully determine the mineral constituent of the carbonaceous shale samples. The minerals contain in the carbonaceous shale are quartz and clay minerals such as kaolite and montmorillonite.

#### 4.8 SEM Experiment

# 4.8.1 Objective

SEM is a type of electron microscope that images the sample of surface by scanning it with high energy beam of electron in a raster scan pattern. The electron interact with the atom that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electron conductivity.[16]

The objective of this experiment is to:

a) To determine and analyzed the crystal morphology of the minerals.

### 4.8.2 Theory

SEM is mainly design for imaging rather than analysis. The images are produced by scanning the beam in a television like raster and displaying the image on the screen. The spatial resolution can better than 10nm in topographic and 100nm in compositional. The SEM images usually 100 times greater than an optical microscope of comparable resolution.

The advantages of SEM such as high spatial resolution, large depth of field and simple specimen preparation makes the SEM an important tool in geology. Some of the usages in geology are: [17]

- a) Palaeontology: SEM is suite to study the fossil morphology and the studies of micro-fossils.
- b) Sedimentology: SEM can scan the individual sediment grains and intergrowth can be viewed in three dimensions.
- c) Mineralogy: SEM is effective to study crystal morphology on a micro scale.

Accelerated electron in SEM carry a large amount of kinetics energy and this energy will dissipate as a variety of signals by electron-sample interaction when the incident electron are decelerate in a sample. The signals include secondary electron that produce the SEM images, backscattered electrons, and diffracted backscattered electrons that produce the crystal structure and orientation of minerals where the photons characteristics X-rays are used in elemental analysis [17].

#### 4.8.3 Results and Discussion

Since the regulation of the SEM laboratory can only take only eight samples per student, the sample are being choose and divided into four groups which each groups consists of two samples from any profiles. The groups can be dividen into tables 11 below:

Groups	Descriptions
Groups 1	• Fresh sample from BG 2 and 3
Groups 2	Half weathered from BG 2 and 3
Groups 3	Weathered sample from BG 2 and 3
Groups 4	Top soil sample from BG 2 and 3

Table 11: The groups and the description of samples for SEM test.

The figure 15 below shows the surface morphology of the fresh sample at 1000 times magnifying. There are 'flake' found at the surface of the fresh sample and also the surface seems to be solid and less pores found around the area. As the samples weathered, the 'flake' disappear from the surface and more pores form at the surface of the sample.

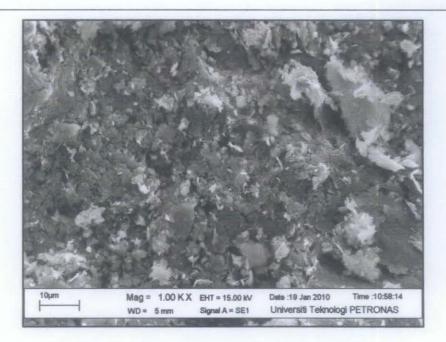


Figure 15: The surface morphology for fresh sample from BG 2.

Weathering and acid rock drainage not only change the chemical properties of the carbonaceous shale, the physical properties of the also changes. The weathered sample such in figure 16 below have more pores compare to the fresh sample where the pores increase the porosity of the weathered carbonaceous shale which can contribute to the disintegration of the rock. The full SEM figure can be view at the appendix F.

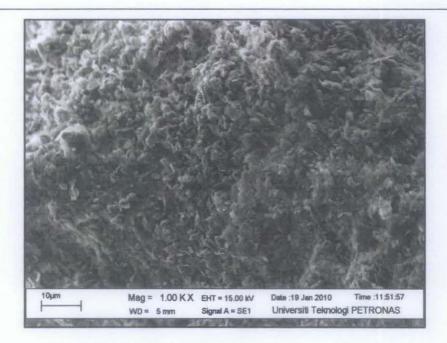


Figure 16: The surface morphology of the weathered sample of BG 3.

# 4.8.4 Conclusion

The surface morphology of carbonaceous shale changes due to the effect of the weathering and acid rock drainage. It is found that, the fresh carbonaceous shale has some 'flaky' effect which indicates the organic content inside the shale. As the shale weathered, the morphology of the carbonaceous shale does not have 'flaky' effect and becomes more pores. It can conclude that, the weathering and ARD can cause the surface morphology of the carbonaceous shale change.

# **CHAPTER 5**

# **OVERALL DISCUSSION**

There are three project areas around Batu Gajah where profiles of sample collected for each project area. Exposed carbonaceous shale founded at the project area which is BG 1, BG 2 and BG 3. The carbonaceous shale experience weathering and acid rock drainage due to the exposure to the environment and it affect the surrounding area such as 'soil cancer' and land slide.

Six samples collected from each profiles area which ranges from the fresh sample, half weathered sample, weathered sample and top soil sample. The sample later prepared to be tested by several experiments that being planned before. The results of each experiment later analyze and discussed in order to meet the objective of the project.

The physical properties that tested for this project are the density, colour and physical observation and SEM test. It is found that the physical properties of the carbonaceous shale change significantly as the carbonaceous shale experience weathering and acid rock drainage. The trend shows for density calculation, all profiles which is BG 1, BG2 and BG 3 experience decreasing of density as the sample move from the fresh sample to weathered and lastly to top soil.

The results are also the same to the colour test and physical observation. The carbonaceous shale that initially black in colour slowly changes its colour as it weathered. Physically, the carbonaceous shale is hard slowly become brittle as the rock weathered. The SEM photos also proof that the pores of the carbonaceous shale increases which also contributes to the decreasing of the density and the strength of the carbonaceous shale itself.

As the carbonaceous shale becomes more brittle and porous, the water can easily enter the rock to remove the weathering product. The rock will be easily disintegrate as the carbonaceous shale becomes brittle and will easily cause the land slide which happens at the project area. The weathering effect at the project area more rapidly happens by the increasing of the rainfall at the area. The carbonaceous shale also called black shale because of the carbon content inside the carbonaceous shale itself. CHNS experiment results proof that the carbon content inside the carbonaceous shale decrease significantly as the rock weathered. The theory also supported by the colour test results where the carbonaceous shale colour becomes lighter as the rock experience weathering.

The sulphur content inside the carbonaceous shale also decreases due to the acid rock drainage. The sulphur is leach out by water during the acid rock drainage and this result also supported by the XRF data which also shown the decrease of sulphur as the sample move down the profile. As a results, the area that effected by the ARD do not have any grass grow at the area.

The XRF data shows that the chemical compositions of Batu Gajah carbonaceous shale are quite similar to the reference data. However, the mineral constituent that obtain from the XRD shows that carbonaceous shale consists of quartz and some clay minerals such as kaolite. However, there are some other mineral such as potassium manganese and lavendulan exist randomly at the samples.

#### **CHAPTER 6**

#### CONCLUSION AND RECOMMENDATION

#### 6.1 Conclusion

The carbonaceous shale along Seputeh – Batu Gajah new road experience weathering and ARD as a result of the road construction a few months ago. There are land slide occur around the affected area and it also found that there are no grass can live around the area which also known as the 'soil cancer'. For the project, three locations were selected as the project area where the sample were taken from the area to be tested physical and chemically.

It can conclude that, the physical properties of the carbonaceous shale change due to weathering and ARD. The density of the carbonaceous shale decreases from fresh to weathered sample as a result of the decomposition of carbon from the carbonaceous shale. The colour of the shale also changes which initially dark gradually decreased becomes lighter as the shale weathered. Physically, the carbonaceous shale is hard and the shale becomes more brittle and porous as the rock weathered. The SEM results show the surface morphology of the weathered sample is more porous than the fresh sample.

In CHNS elemental analysis, it is found that the percentage of carbon gradually decreased and almost depleted at weathered sample which also affected the colour of the carbonaceous shale. The percentage of sulphur also decreased gradually and depleted at the weathered sample due to ARD. In XRF, all the element that contain in the carbonaceous shale can be determine. It is found that element contain inside the carbonaceous shale are quite similar with the reference. The XRD show the mineral constituent of the carbonaceous shale, it is found that the carbonaceous shale contain quartz and other clay mineral.

Lastly, it can conclude that the physical and chemical properties of the carbonaceous shale are change due to weathering and ARD where severely affected the environment surround it.

#### 6.2 Recommendation

As the project complete, there are still some recommendations can be suggested in order to improve the project. First recommendation is to add more books related to geosciences in UTP library improve the literature review. For better results of the experiment, it is recommend to increase the number of samples and also to carefully select the sample. The better criteria for sample collection need to be review in order to come out with a better requirement for sample collection.

Other than that, the equipment inside the lab also needs to be maintained carefully so that the equipment can operate during the semester. Less breakdown means the project can be done on time and less interruption of experiment flow can be obtain and better results for the experiment. Lastly, further study on carbonaceous shale need to be done in order to makes the project better and useful to future needs.

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#### **APPENDIX A: Density Calculation Results**

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Profile	Mass	Volume	1	Volume 2	Volume	Density	Density
	(g)	(ml)		(mi)	(ml)	(g/ml)	(Kg/m3)
Spot 1	5.90	30.00		33.00	3.00	1.97	0.00197
Spot 2	7.20	30.00		34.00	4.00	1.80	0.00180
Spot 3	3.20	30.00		32.00	2.00	1.60	0.00160
Spot 4	5.10	30.00		33.00	3.00	1.70	0.00170
Spot 5	4.60	30.00		33.00	3.00	1.53	0.00153
Spot 6	2.90	30.00		32.00	2.00	1.45	0.00145

Table 12: The results for density calculation for BG 1.

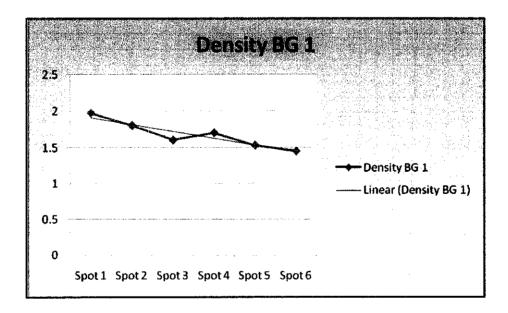


Figure 17: The graph of density of BG 1 profile.

	BG	2
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Profile	Mass	Volume 1	Volume 2	Volume	Density	Density
	(g)	(ml)	(ml)	(ml)	(g/ml)	(Kg/m3)
Spot 1	3.40	30.00	31.50	1.50	2.26	0.00226
Spot 2	4.20	30.00	32.00	2.00	2.10	0.00210
Spot 3	4.46	30.00	32.00	2.00	2.23	0.00223
Spot 4	5.40	30.00	33.00	3.00	1.80	0.00180
Spot 5	2.90	30.00	32.50	2.50	1.45	0.00145
Spot 6	4.60	30.00	32.50	2.50	1.84	0.00184
						1

Table 13: The results for density calculation for BG 2.

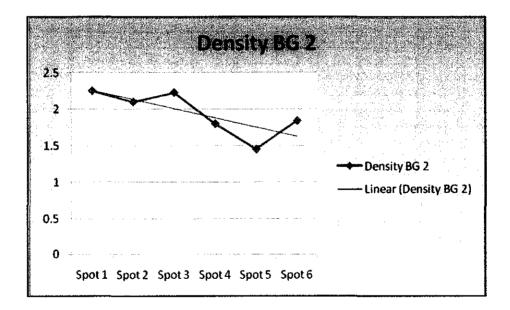
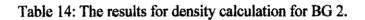


Figure 18: The graph of density of BG 1 profile.

Mass	Volume 1	Volume 2	Volume	Density	Density
(g)	(ml)	(ml)	(ml)	(g/ml)	(Kg/m3)
4.20	30.00	32.00	2.00	2.10	0.00210
6.20	30.00	32.50	2.50	2.48	0.00248
7.30	30.00	33.00	3.00	2.21	0.00221
4.70	30.00	32.00	2.00	1.70	0.00170
3.40	30.00	32.50	2.50	1.88	0.00188
3.90	30.00	32.50	2.50	1.56	0.00156
	(g) 4.20 6.20 7.30 4.70 3.40	(g)       (ml)         4.20       30.00         6.20       30.00         7.30       30.00         4.70       30.00         3.40       30.00	(g)(ml)(ml)4.2030.0032.006.2030.0032.507.3030.0033.004.7030.0032.003.4030.0032.50	(g)(ml)(ml)(ml)4.2030.0032.002.006.2030.0032.502.507.3030.0033.003.004.7030.0032.002.003.4030.0032.502.50	(g)(ml)(ml)(ml)(g/ml)4.2030.0032.002.002.106.2030.0032.502.502.487.3030.0033.003.002.214.7030.0032.002.001.703.4030.0032.502.501.88



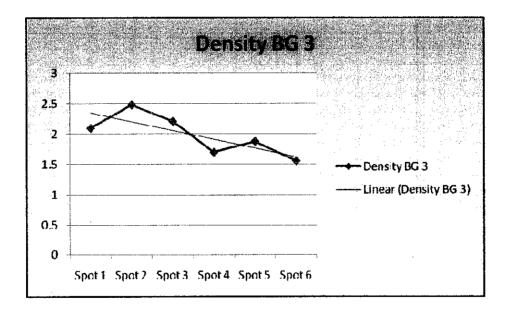


Figure 19: The graph of density of BG 1 profile.

# **APPENDIX B: Colour Test and Physical Observation Results**

## **BG 1 Solid**

Samples	Picture	Description
Spot 1		<ul> <li>Medium dark grey which symbolled as N4 in the colour chart.</li> <li>Physically hard, layer structure and choose as fresh sample for BG 1</li> </ul>
Spot 2		<ul> <li>Medium dark grey which symbolled as N4 in the colour chart.</li> <li>Physically hard, slightly weathered as white stain found at some part of the rock.</li> </ul>
Spot 3		<ul> <li>Dark grey which symbolled as N3 in the rock colour chart.</li> <li>Obvious white layer found in between layers.</li> <li>Slightly weathered and physically not too hard.</li> </ul>

Table 15 : BG 1 solid results for colour determination and physical descriptions.

Spot 5	<ul> <li>White in colour as it symbolled as N9 in the rock colour chart.</li> <li>Slightly hard, still have layer and weathered carbonaceous shale.</li> </ul>
Spot 6	<ul> <li>Yellowish grey which symbolled as 5Y 8/1 in the colour chart.</li> <li>Do not have layer, porous, weathered and not as hard as the fresh sample.</li> </ul>
Spot 7	<ul> <li>Light brown which symbolled as 5YR 5/6 in the rock colour chart.</li> <li>The top soil which the carbonaceous shale completely converted into soil.</li> <li>Porous, and not physically hard.</li> </ul>

Table 15: Continue BG 1 solid results for colour determination and physical descriptions.

#### BG 2 Solid

Samples	Picture	Description
Spot 1		<ul> <li>Greyish black which symbolled as N2 in the rock colour chart.</li> <li>Shiny black, does not have layer and physically hard.</li> </ul>
Spot 2		<ul> <li>Medium grey which indicated as N5 in the rock colour chart.</li> <li>The sample is hard but no obvious layer indicated.</li> <li>Slightly weathered as white stain at some point of the sample.</li> </ul>
Spot 3		<ul> <li>Brownish black which indicated as 5YR 2/1 in the rock colour chart.</li> <li>Slightly weathered and cemented by the Acid Rock Drainage which give the brownish colour.</li> <li>Physically hard and do not have layer.</li> </ul>

Table 16: Physical observation and colour determination for solid sample BG 2

Spot 4	<ul> <li>White which indicated as N9 in the rock colour chart.</li> <li>Weathered, porous and have some pinkish stain.</li> <li>Physically soft and do not have obvious layer.</li> </ul>
Spot 5	<ul> <li>Very pale orange which symbolled as 10 YR 8/2 in the rock colour chart.</li> <li>Weathered rock, porous and physically not hard compare to the fresh sample.</li> </ul>
Spot 6	<ul> <li>Dark yellowish orange which indicated as 10YR 6/6</li> <li>Top soil of the profile which physically soft, porous and do not have layer.</li> </ul>

Table 16: Continue physical observation and colour determination for solid sample BG 2

## BG 3 Solid

Samples	Picture	Description
Spot 1		<ul> <li>Medium grey which indicated as N5 in the rock colour chart.</li> <li>Physically hard and indicates as fresh sample for BG 3.</li> <li>Do not have obvious layers.</li> </ul>
Spot 2		<ul> <li>Medium light grey which indicated as N6 in the rock colour chart.</li> <li>Slightly weathered as white stain at some part of the rock.</li> <li>Physically hard and do not have layers.</li> </ul>
Spot 3		<ul> <li>Medium light grey which indicated as N6 in the rock colour chart.</li> <li>Slightly weathered as some white and brown stain at some part of the rock.</li> <li>Not too hard and do not have layers.</li> </ul>

Table 17: Physical observation and colour determination for solid BG 3

Spot 4	<ul> <li>White which indicated as N9 in the rock colour chart.</li> <li>Weathered, porous and not too hard. Do not have layers also.</li> </ul>
Spot 5	<ul> <li>Yellowish grey which indicated as 5Y 8/1 in the rock colour chart.</li> <li>Weathered, porous and brittle, do not have layers.</li> </ul>
Spot 6	<ul> <li>Very pale orange which indicated as 10YR 8/2 in the rock colour chart.</li> <li>Top soil of the profile, brittle, porous and do not have layers.</li> </ul>

Table 17: Continue. Physical observation and colour determination for solid BG 3

#### **BG 1 Powder**

Spot 1	•	Medium dark grey which also indicated as N4 in the rock colour chart.
Spot 2	•	Medium grey which also indicated as N5 in the rock colour chart.
Spot 3	•	Medium light grey which symbolled as N6 in the rock colour chart.

Table 18: Physical observation and colour determination for powder BG 1

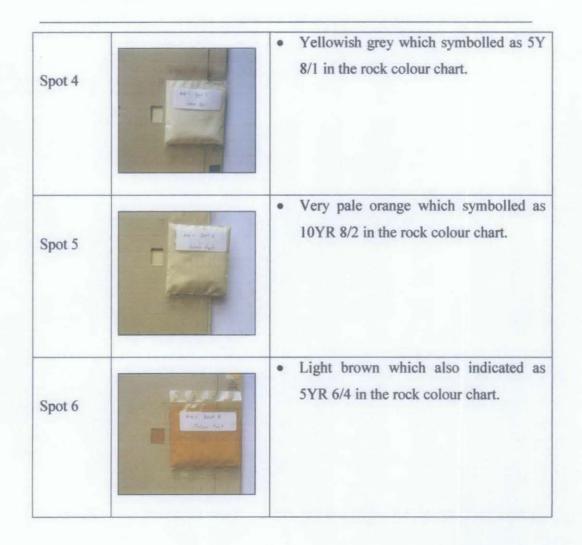


Table 18: Continue. Physical observation and colour determination for powder BG

1

## **BG 2 Powder**

Spot 1	•	Dark grey which labelled as N3 in the rock colour chart.
Spot 2	•	Medium Grey which labelled as N5 in the rock colour chart.
Spot 3	•	Medium light grey which also labelled as N6 in the rock colour chart.

Table 19: Physical observation and colour determination for powder BG 2

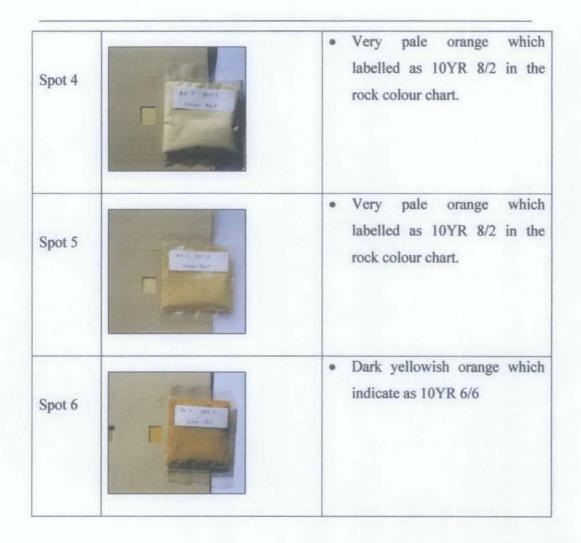


Table 19: Continue. Physical observation and colour determination for powder BG

2

#### **BG 3 Powder**

Spot 1	•	Medium dark grey which labelled as N4 in the rock colour chart.
Spot 2	•	Medium Grey which labelled as N5 in the rock colour chart.
Spot 3	•	Medium light grey which labelled as N6 in the rock colour chart.

Table 20: Physical observation and colour determination for powder BG 3

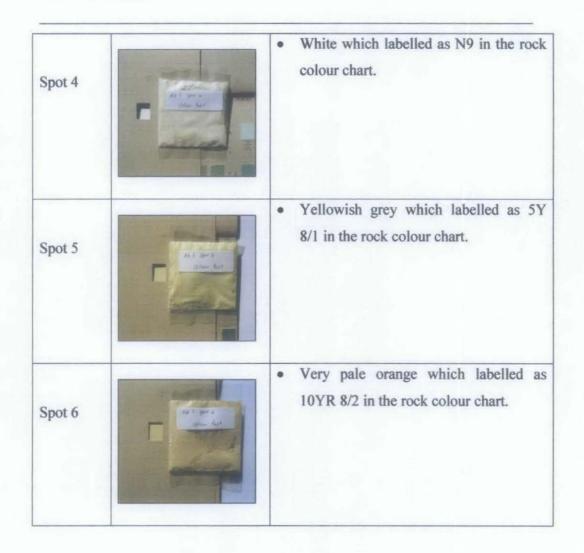


Table 20: Continue. Physical observation and colour determination for powder BG

3

#### **APPENDIX C: CHNS Results**

## **BG** 1

No	Sample	%C	% H	%N	% S	wt (mg)	
1	BG 1 Spot 1	1.225	0.505	0.151	0.039	1.895	
2	BG 1 Spot 1	1.065	0.394	0.129	0.030	1.932	
3	BG 1 Spot 1	0.941	0.334	0.123	0.031	1.864	
	Average Spot 1	1.077	0.411	0.134	0.033	1.897	
1	BG 1 Spot 2	1.093	0.920	0.172	0.243	1.916	
2	BG 1 Spot 2	1.158	0.849	0.170	0.379	1.765	
3	BG 1 Spot 2	1.769	0.793	0.186	0.126	1 <i>.</i> 958	
	Average Spot 2	1.340	0.854	0.176	0.249	1.880	
1	BG 1 Spot 3	0.426	0.646	0.150	0.277	1.927	
2	BG 1 Spot 3	0.511	0.541	0.123	0.222	1.867	
3	BG 1 Spot 3	0.420	0.558	0.142	0.209	1.818	
	Average Spot 3	0.452	0.582	0.138	0.236	1.871	
1	BG 1 Spot 4	0.078	0.886	0.122	-0.002	1.878	
2	BG 1 Spot 4	0.072	0.755	0.095	0.019	1.931	
3	BG 1 Spot 4	0.055	0.771	0.099	-0.005	1.825	
	Average Spot 4	0.068	0.804	0.105	0.004	1.878	
1	BG 1 Spot 5	0.043	0.703	0.101	0.028	1.950	
2	BG 1 Spot 5	0.033	0.485	0.074	0.012	1.886	
3	BG 1 Spot 5	0.039	0.552	0.090	0.021	1.838	
	Average Spot 5	0.038	0.580	0.088	0.020	1.891	
1	BG 1 Spot 6	0.130	1.683	0.094	0.020	1.782	
2	BG 1 Spot 6	0.185	1.560	0.111	0.011	1.875	
3	BG 1 Spot 6	0.122	1.406	0.084	0.029	1.819	
	Average Spot 6	0.146	1.550	0.096	0.020	1.825	

Table 21: CHNS results for BG 1

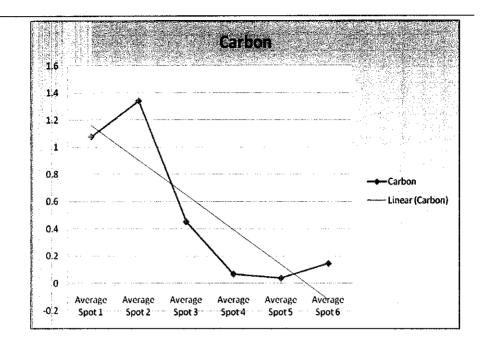


Figure 20: The graph of carbon percentage for BG 1

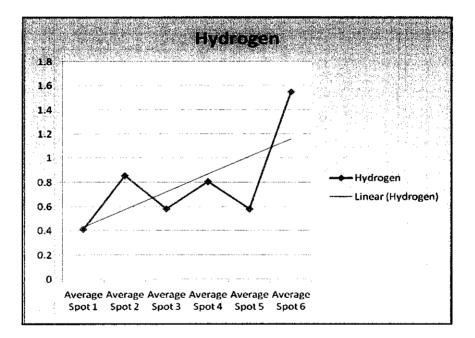


Figure 21: The graph of hydrogen percentage for BG 1

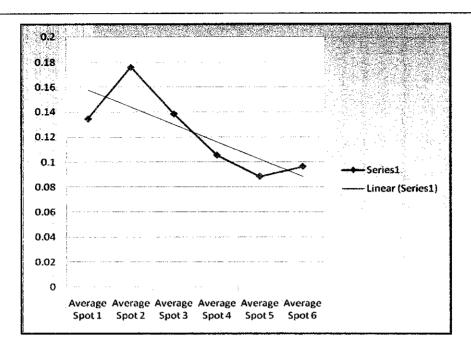


Figure 22: The graph of nitrogen percentage for BG 1

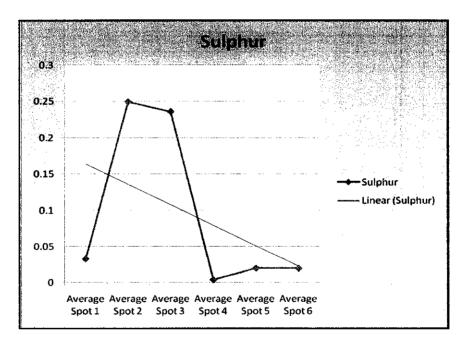


Figure 23: The graph of sulphur percentage for BG 1

#### BG 2

No	Sample	% C	%Н	%N	% S	wt (mg)
1	BG 2 Spot 1	3.532	0.286	0.146	0.014	1.926
2	BG 2 Spot 1	5.339	0.450	0.211	0.041	1.808
3	BG 2 Spot 1	4.483	0.374	0.183	0.035	1.957
	Average Spot 1	4.451	0.370	0.180	0.030	1.897
1	BG 2 Spot 2	0.987	0.675	0.145	0.066	1.847
2	BG 2 Spot 2	-	-	-	-	1.954
3	BG 2 Spot 2	0.690	0.707	0.173	0.052	1.765
	Average Spot 2	0.839	0.691	0.159	0.05 <del>9</del>	1.806
			·			
1	BG 2 Spot 3	0.458	1.306	0.135	0.026	1.662
2	BG 2 Spot 3	0.528	1.215	0.121	0.003	1.802
3	BG 2 Spot 3	0.506	1.299	0.132	0.012	1.785
	Average Spot 3	0.497	1.273	0.129	0.014	1,750
1	BG 2 Spot 4	0.124	0.299	0.082	-0.002	1.772
2	BG 2 Spot 4	0.081	0.273	0.062	0.008	1.846
3	BG 2 Spot 4	0.096	0.32	0.074	0.015	1.706
	Average Spot 4	0.100	0.297	0.073	0.007	1.775
			,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,			
1	BG 2 Spot 5	0.058	0.673	0.127	0.003	1.895
2	BG 2 Spot 5	0.078	0.639	0.096	-0.002	1.847
3	BG 2 Spot 5	0.059	0.572	0.099	0.022	1.788
	Average Spot 5	0.065	0.628	0.107	0.008	1.843
1	BG 2 Spot 6	0.054	0.815	0.082	0.076	1.916
2	BG 2 Spot 6	0.059	0.862	0.085	0.097	1.836
3	BG 2 Spot 6	0.071	0.797	0.078	0.032	1.870
	Average Spot 6	0.061	0.825	0.082	0.068	1.874

Table 22: The CHNS results for BG2

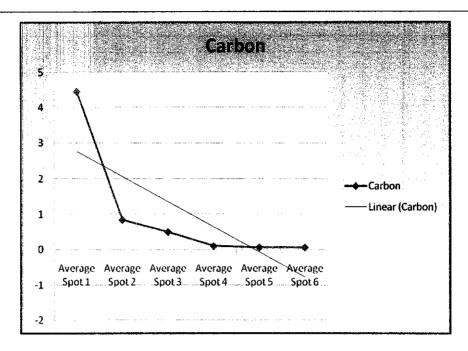


Figure 24: The graph of carbon for BG 2

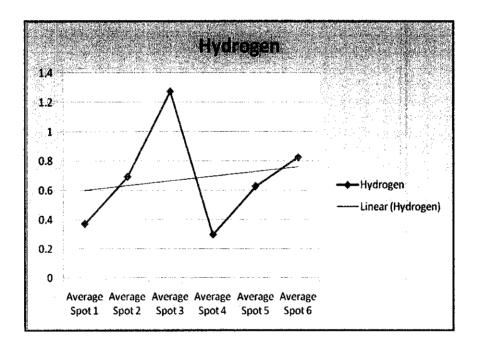


Figure 25: The graph of hydrogen for BG 2

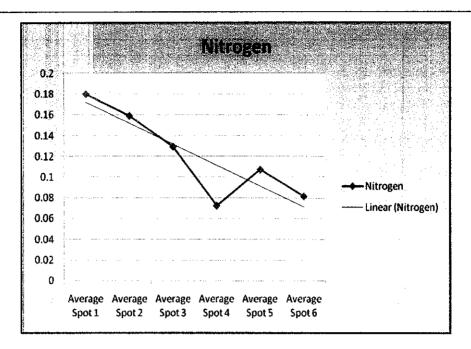


Figure 26: The graph of nitrogen for BG 2

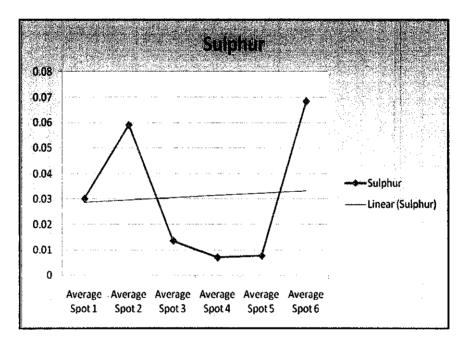


Figure 27: The graph of sulphur for BG 2

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No	Sample	%С	%Н	%N	% S	wt (mg)
1	BG 3 Spot 1	5.594	0.628	0.28 <del>9</del>	0.107	1.937
2	BG 3 Spot 1	6.741	0.797	0.368	0.105	1.899
3	BG 3 Spot 1	8.320	0.726	0.373	0.125	1.851
	Average Spot 1	6.885	0.717	0.343	0.112	1.896
1	BG 3 Spot 2	2.234	0.451	0.224	0.047	1.914
2	BG 3 Spot 2	5.556	0.403	0.272	0.007	1.86
3	BG 3 Spot 2	2.060	0.491	0.229	0.004	1.805
	Average Spot 2	3.283	0.448	0.242	0.019	1.860
1	BG 3 Spot 3	1.916	0.723	0.25	0.026	1.865
2	BG 3 Spot 3	1.484	0.49	0.198	0.039	1.803
3	BG 3 Spot 3	1.817	0.716	0.23	0.026	1.928
	Average Spot 3	1,739	0.643	0.226	0.030	1.865
		•••••••				
1	BG 3 Spot 4	0.106	0.683	0.196	0.064	1.976
2	BG 3 Spot 4	0.048	0.706	0.216	0.009	1.783
3	BG 3 Spot 4	0.041	0.663	0.227	0.045	1.904
	Average Spot 4	0.065	0.684	0.213	0.039	1.888
		,				
1	BG 3 Spot 5	0.117	0.765	0.163	0.012	1.947
2	BG 3 Spot 5	0.150	0.757	0.156	0.003	1.838
3	BG 3 Spot 5	0.083	0.775	0.167	-0.015	1.844
	Average Spot 5	0.117	0.766	0.162	0	1.876
		······				
1	BG 3 Spot 6	0.256	0.749	0.190	0.005	1.857
2	BG 3 Spot 6	0.184	0.728	0.180	0.011	1.712
3	BG 3 Spot 6	0.434	0.748	0.193	-0.002	1.879
	Average Spot 6	0.291	0.742	0.188	0.005	1.816

Table 23: The CHNS results for BG 3

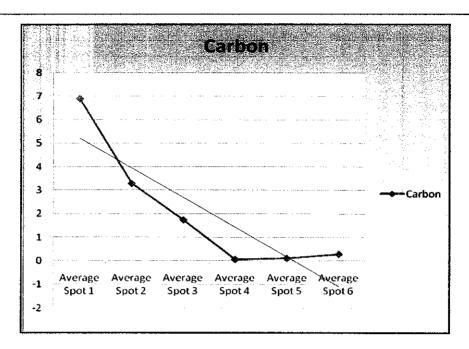


Figure 28: The graph of carbon for BG 3

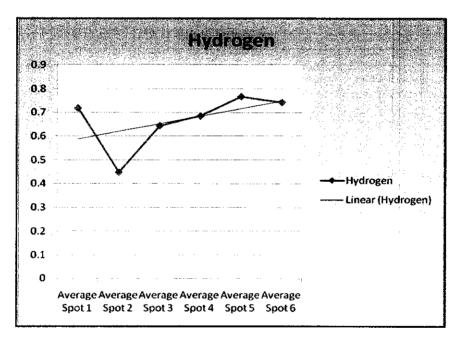


Figure 29: The graph of hydrogen for BG 3

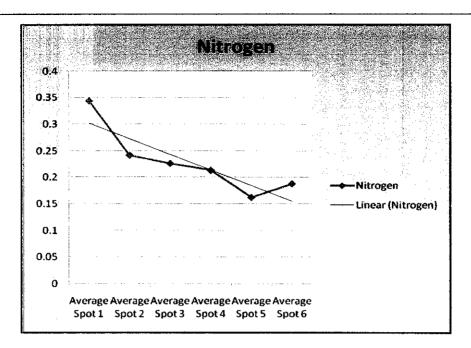


Figure 30: The graph of nitrogen for BG 3

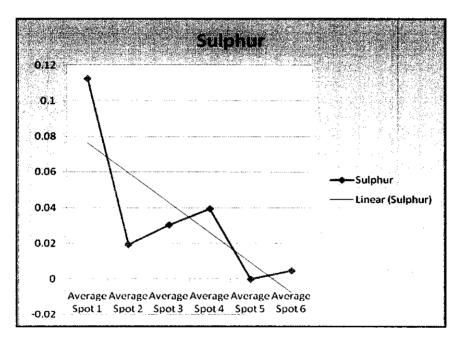


Figure 31: The graph of sulphur for BG 3

## **APPENDIX D: XRF Results**

### **BG 1 XRF Result**

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Symbol	Oxides	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6
MgO	Magnesium Oxide	1.19	1	1.38	0.997	0.628	0.48
AI2O3	Aluminium Oxide	21.64	27.31	21.21	21.59	18.18	35.6
SiO2	Silicon Dioxide	67.16	60.49	66.67	70.07	75.81	43.04
P2O5	Phosphorus Pentoxide	1.06	0.867	0.889	0.917	0.833	0.914
SO3	Sulphur Trioxide	0.361	0.893	1.03	0	0	0.12
к20	Pottasium Oxide	5.422	4.875	5.42	3.772	2.741	2.834
CaO	Calcium Oxide	0.182	0.154	Q.16	0.153	0.138	0.157
TiO2	Titatium Dioxide	1.29	2.303	1.23	1.01	0.679	2.044
V205	Vanadium (V) Oxide	0	0.0442	0	0	0	0.0563
Cr2O3	Chromium (III) Oxide	0.009	0.0107	0.005	0	0	0.0259
Fe2O3	Iron (III) Oxide	1.47	1.881	1.84	1.297	0.8498	14.62
ZnO	Zinc Peroxide	0.0128	0	0.003	0	0.0571	0.0053
Rb2O	Rubidium Oxide	0.0168	0.019	0.0177	0.0156	0.012	0.00136
SrO	Strontium Oxide	0.0174	0.0055	0.0088	0.0135	0.0082	0.0076
ZrO2	Zirconium Oxide	0.0288	0.0351	0.0259	0.0309	0.0201	0.0068
BaQ	Barium Oxide	0.15	Q	Q	0.14	0	Q
NiO	Nickle Oxide	0	0.004	0	0	0	0
CuO	Copper Oxide	0	0.0038	0	0	0	0
MnO	Manganese Oxide	0	0	0.023	0	0	0
Ga2O3	Galium (III) Oxide	0	0	0	0	0	0.008

Table 24: XRF result, chemical composition for BG 1.

### **BG 2 XRF Result**

Symbol	· · · · · · · · ·						
S	Name	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6
MgO	Magnesium Oxide	1.02	0.773	0.44	0.26	0.498	0.45
AI2O3	Aluminium Oxide	20.01	27.33	30.4	14.1	21.22	21.74
SiO2	Silicon Dioxide	68.9	60.99	47.45	79.31	70.79	61.94
P2O5	Phosphorus Pentoxide	0.872	0.852	0.849	0.835	0.812	0.874
SO3	Sulphur Trioxide	0.409	0.362	0	0	0	0.316
K2Q	Pottasium Oxide	6.154	6.91	3.135	2.269	4.558	3.021
CaO	Calcium Oxide	0.168	0.144	0.187	0.154	0.139	0.133
TiO2	Titatium Dioxide	1.25	1.32	1.22	0.671	0.914	1.01
V205	Vanadium (V) Oxide	0.154	0.024	0.025	0	0	0
Cr2O3	Chromium (III) Oxide	0.024 5	0.014 8	0.016 6	0.009	0.006	0.003
Fe2O3	Iron (III) Oxide	0.925	1.128	16.2	2.366	0.949 4	10.39
NiO	Nickle Oxide	0.008 6	0	0	0	0	0
Rb2O	Rubidium Oxide	0.024 3	0.037	0.021	0.010	0.027 4	0.018
		0.030	0.023	0.041	0.013	0.017	0.028
ZrO2	Zirconium Oxide	4	9	9	6	3	9
SrO	Strontium Oxide	0	0.007	0.007	0.004	0.005	0.005
				0.005		<u>-</u>	0.004
ZnO2	Zinc Peroxide	0	0	1	0	0	9
CuO	Copper Oxide	0	0	0	0.00 <del>3</del> 3	0	0.014
MnO	Manganese Oxide	0	0	0	0	0	0.047

Table 25: XRF result, chemical composition for BG 2.

#### **BG 3 XRF Result**

· · · · · · · · · · · · · · · · · · ·							
Element	Name	Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6
	a har she a far y da 19 a 1						
MgO	Magnesium Oxide	0.94	0.675	0.762	0.465	0.41	0.636
AI2O3	Aluminium Oxide	19.14	14.3	23.7	17	20.63	23.49
SiO2	Silicon Dioxide	68.89	77.48	65.93	73.93	72.28	66.53
P2O5	Phosphorus Pentoxide	0.864	0.818	0.799	0.759	0.786	0.821
SO3	Sulphur Trioxide	0.826	0.13	0.257	0.169	0	0
K20	Pottasium Oxide	6.362	4.263	6.051	5.081	3.742	5.099
CaO	Calcium Oxide	0.161	0.141	0.145	0.128	0.138	0.167
TiO2	Titatium Dioxide	0.993	0.756	1.11	0.829	0.942	0.998
V205	Vanadium (V) Oxide	0.39	0	0.054	0	0	0
Cr2O3	Chromium (III) Oxide	0.0401	0.004	0.0113	0.006	0.006	0.008
Fe2O3	Iron (III) Oxide	1.192	1.341	1.028	1.464	0.9393	2.107
NiO	Nickle Oxide	0.0148	0	0	0	0	0
Rb2O	Rubidium Oxide	0.0318	0.0209	0.0281	0.0292	0.0238	0.0252
ZrO2	Zirconium Oxide	0.0347	0.0172	0.0224	0.0141	0.0288	0.0215
BaO	Barium Oxide	0.126	0	0	0.122	0	0
SrO	Strontium Oxide	0	0	0	0	0.0043	0.0046

Table 26: XRF result, chemical composition for BG 3.

#### **APPENDIX E: XRD Results**



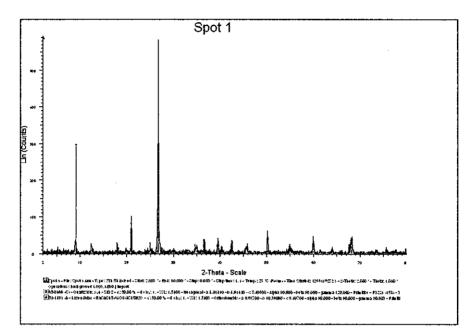


Figure 32: XRD for BG 1 Spot 1

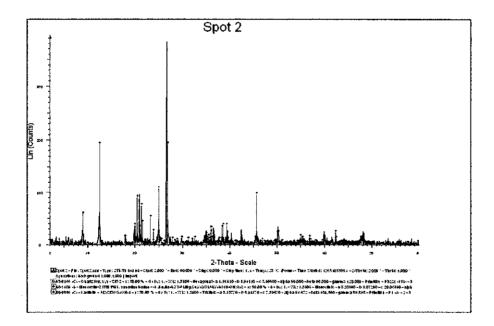


Figure 33: XRD for BG 1 Spot 2

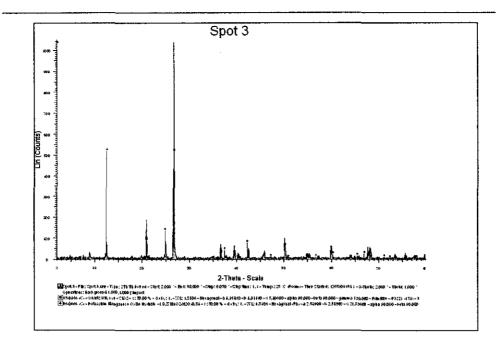


Figure 34: XRD for BG 1 Spot 3

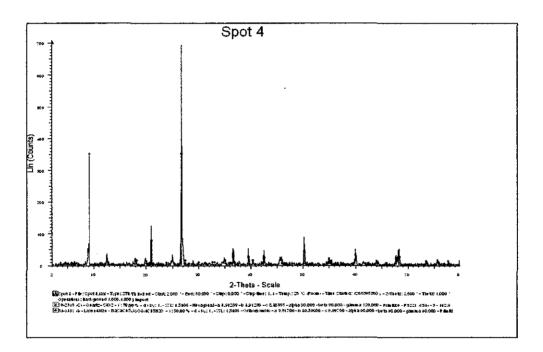


Figure 35: XRD for BG 1 Spot 4

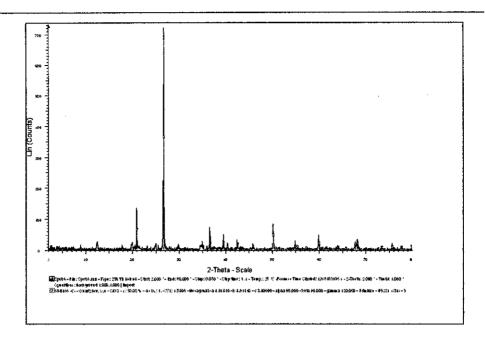


Figure 36: XRD for BG 1 Spot 5

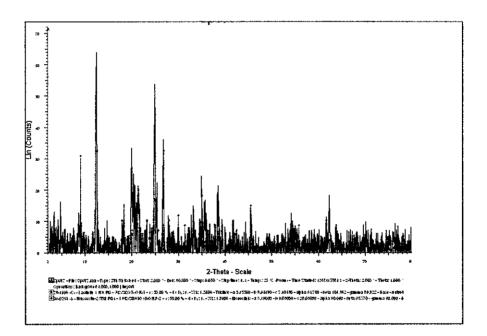


Figure 37: XRD for BG 1 Spot 6

## **XRD Results for BG 2**

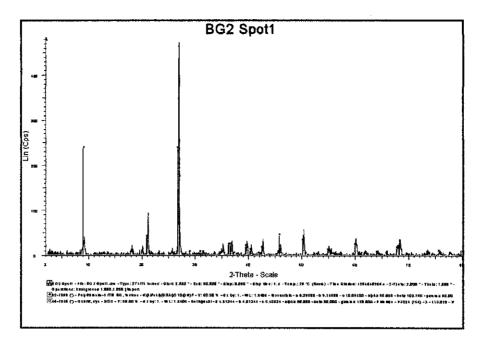


Figure 38: XRD for BG 2 Spot 1

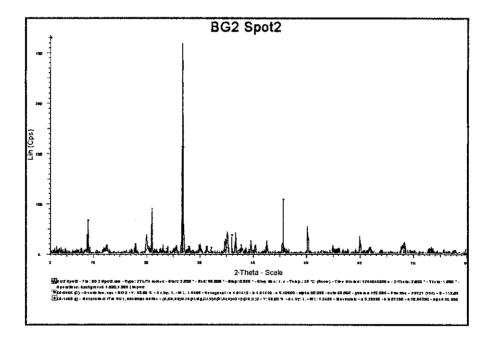


Figure 39: XRD for BG 2 Spot 2

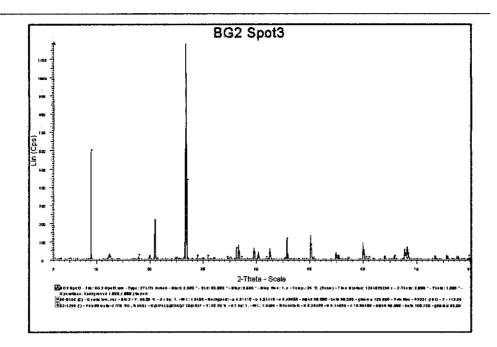


Figure 40: XRD for BG 2 Spot 3

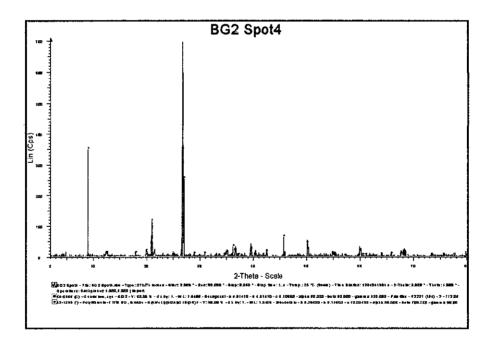


Figure 41: XRD for BG 2 Spot 4

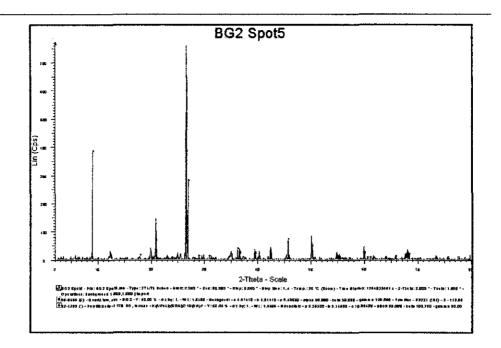


Figure 42: XRD for BG 2 Spot 5

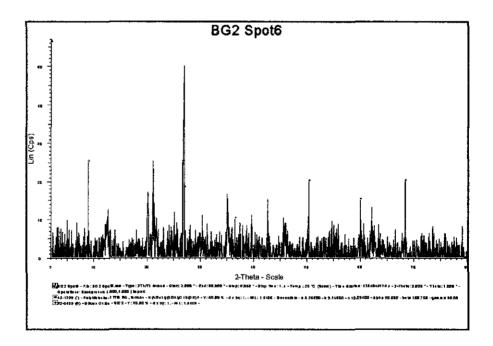


Figure 43: XRD for BG 2 Spot 6

#### **XRD Result for BG 3**

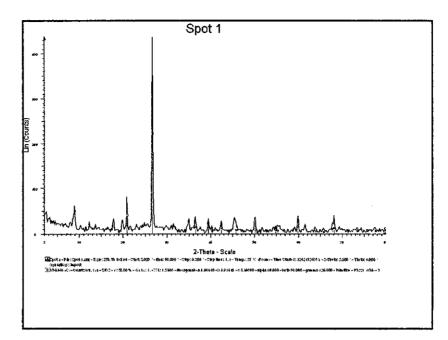


Figure 44: XRD for BG 3 Spot 1

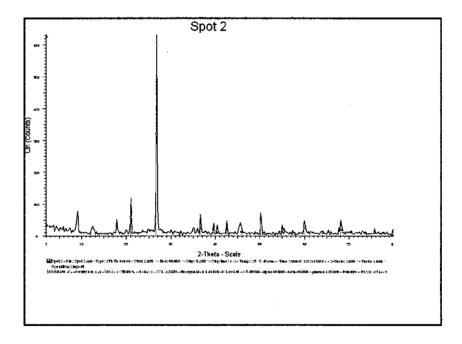


Figure 45: XRD for BG 3 Spot 2

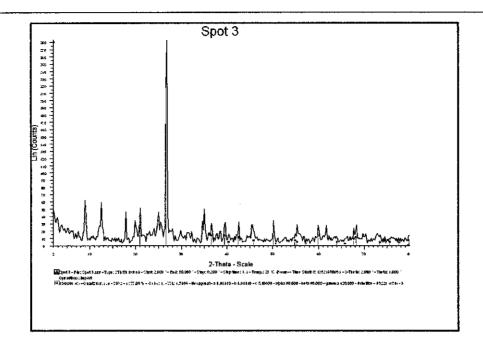


Figure 46: XRD for BG 3 Spot 3

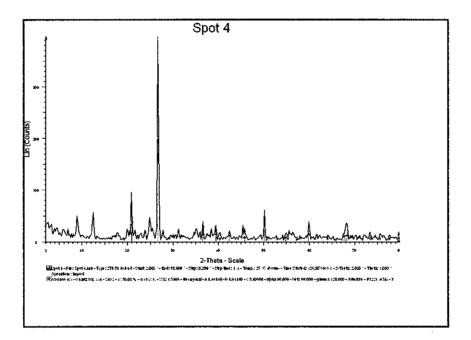


Figure 47: XRD for BG 3 Spot 4

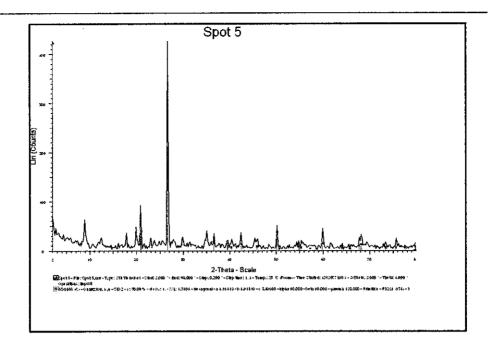


Figure 48: XRD for BG 3 Spot 5

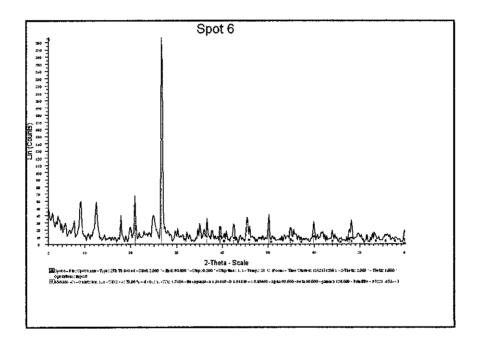


Figure 49: XRD for BG 3 Spot 6

#### **APPENDIX F: SEM Results**

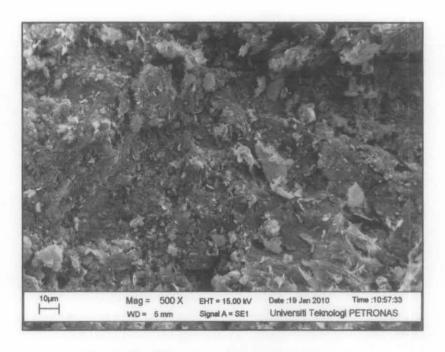


Figure 50: SEM photo for BG 2 fresh sample.

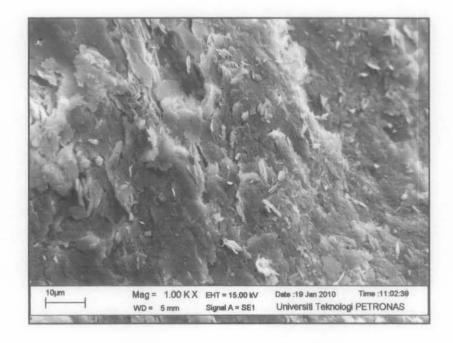


Figure 51: SEM photo for BG 3 fresh sample.

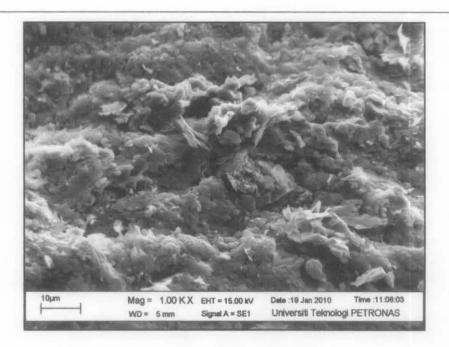


Figure 52: SEM photo for BG 2 half weathered sample.

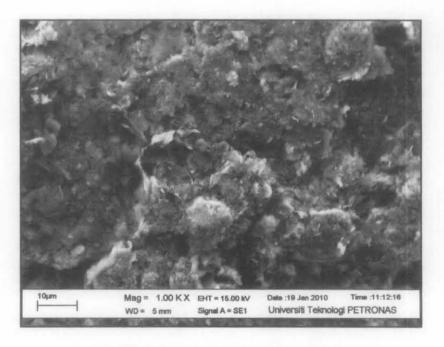


Figure 53: SEM photo for BG 3 half weathered sample.

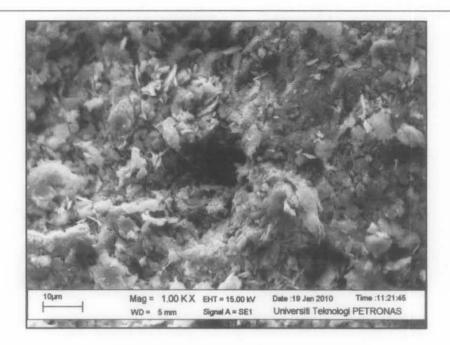


Figure 54: SEM photo for BG 2 weathered sample.

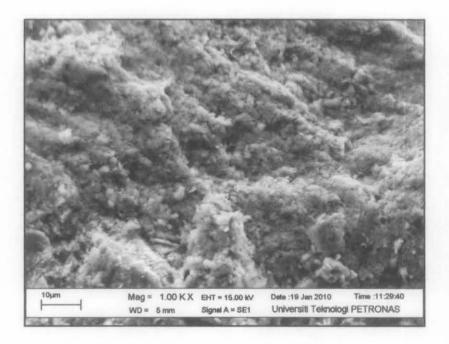


Figure 55: SEM photo for BG 3 weathered sample.

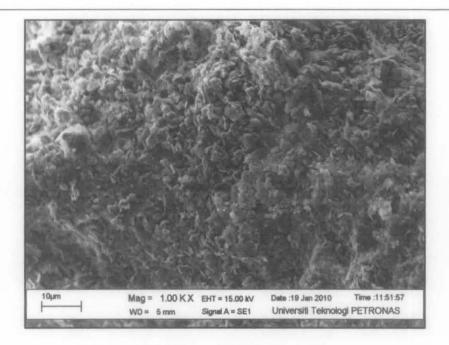


Figure 56: SEM photo for BG 2 top soil sample.

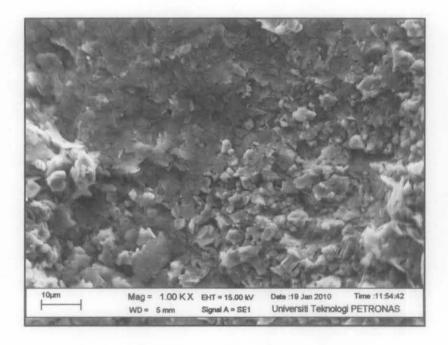


Figure 57: SEM photo for BG 3 top soil sample.