Physical and Chemical Characteristic of Mukah Coal – Sarawak in Relation to Gas Content and Composition

by

Lim Woan Chin

A project dissertation submitted to the
Chemical Engineering Programme
in partial fulfillment of
the requirement of the
Bachelor of Engineering (Hons)
Chemical Engineering

JANUARY 2009

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CERTIFICATION OF APPROVAL

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Approved:

AP. Dr. Chow Weng Sum
Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2009

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.

LIM WOAN CHIN

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TABLE OF CONTENTS

CERTIFICATION OF APPROVAL i

CERTIFICATION OF ORIGINALITY ii

ABSTRACT iii

ACKNOWLEDGEMENT iv

CHAPTER 1: INTRODUCTION 1

1.1 Background of Study 1

1.2 Problem Statement 3

1.3 Objectives 5

1.4 Scope of Study 5

CHAPTER 2: LITERATURE REVIEW 6

2.1 Coal 6

2.2 Formation of Coal 6

2.3 Types of Coal 7

2.4 Analysis of Coal. 8

2.5 Experiments Theory. 9

2.5.1 Porosity and Permeability . . . 9

2.5.2 Moisture Content 17

2.5.3 Analysis of Gas Content by GC . . 17

CHAPTER 3: METHODOLOGY 19

3.1 Project Flow Diagram. 19

3.2 Experimental Procedures 20

3.3 Tools Required 22
3.3.1 Core Cutting Machine. . . 22
3.3.2 Core Trimming Machine . . 23
3.3.3 POROPERM 24
3.3.4 XRD Analysis. . . . 25
3.3.5 Sputter Coater. . . . 25
3.3.6 Scanning Electron Microscope (SEM). 26
3.3.7 Energy Dispersive X-ray (EDX) . 27
3.3.8 Drying Oven. 27
3.3.9 XRF Analysis 28
3.3.10 2000kN Flexural and Compression Machine 29
3.3.11 Gas Chromatography . . . 29
3.4 Gantt Chart 31
CHAPTER 4: RESULTS AND DISCUSSION . . . 32
4.1 Porosity and Permeability Test. . . 32
4.2 Pore Size and Structures of Coal. . . 37
4.3 Composition of Coal 38
4.4 Moisture Content and Compressive Strength. 41
4.5 Gas Content of Mukah Coal. . . . 44
CHAPTER 5: CONCLUSION AND RECOMMENDATION. . 46
5.1 Conclusion 46
5.2 Recommendation 47
REFERENCES 48
APPENDICES 49

LIST OF ILLUSTRATIONS

Figure 1.1 East Malaysia map 3

Figure 1.2 World Liquid Fuels Consumption, 2002-2010 4

Figure 1.3 World Crude Oil and Liquid Fuels Consumption, 2008-2010 5

Figure 2.1 Illustration to show the different volumes consist in a system 10

Figure 2.2 Schematic diagram of initial and final conditions of POROPERM 11

Figure 2.3 Schematic diagram of pressure falloff gas permeameter . 14

Figure 3.1 POROPERM application window 21

Figure 3.2 Core cutting machine 22

Figure 3.3 Core trimming machine 23

Figure 3.4 SEM inner view 26

Figure 3.5 Drying Oven 28

Figure 3.6 XRF 28

Figure 3.7 2000kN flexural and compression machine. . . . 29

Figure 3.8 Gas extraction equipment setup. 30

Figure 3.9 GC Equipment 30

Figure 4.1 Picture of core plug from Ulu Sikat 32

Figure 4.2 Picture of core plug from Ulu Penipah . . . 33

Figure 4.3 Front and back view of two coal samples from Ulu Sikat . 35

Figure 4.4 Front and back view of two coal samples from Ulu Penipah . 35

Figure 4.5 Ulu Penipah sample's image using SEM at magnification of 3000x 37

Figure 4.6 Ulu Sikat sample's image using SEM at magnification of 3000x 38

Figure 4.7 EDX spectrums that displays the peaks of elements in Ulu Penipah's sample 39

Figure 4.8 EDX spectrums that displays the peaks of elements in Ulu Sikat's sample 40

Figure 4.9 Mukah coal before compression 42

Figure 4.10 Mukah coal after compression. 42

Figure 4.11 Gas Chromatography results for Ulu Sikat coal. . . 43

Figure 4.12 Gas Chromatography results for Ulu Penipah coal. . . 44

Figure A-1 Map of Mukah Balingian area indicating approximate mining location I

LIST OF TABLES

Table 3.1 Gantt Chart 31

Table 4.1 Results of diameter measurement of core plug in different section. 33

Table 4.2 Results of length measurement of core plug in different section. 33

Table 4.3 Properties of core plug and experiment condition in laboratory. 34

Table 4.4 Average length and diameter, others factor of core plug. . 34

Table 4.5 Results of porosity tested and calculated by using POROPERM. 35

Table 4.6 Results of permeability tested and calculated by using POROPERM. 36

Table 4.7 Composition of Ulu Penipah's coal in weight percent in EDX. 39

Table 4.8 Composition of Ulu Sikat's coal in weight percent in EDX . 40

Table 4.9 Composition of Mukah coal using XRF 41

Table 4.10 Moisture content for Mukah coal 42

Table 4.11 Comparison of moisture content and compressive strength with standard properties for Lignite and Bituminous coal . . 43

Table 4.12 Summary of Gas content composition for Ulu Sikat coal. . 44

Table 4.13 Summary of Gas content composition for Ulu Penipah coal . 45

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Coal is a readily combustible black or brownish-black sedimentary rock composed primarily of carbon and hydrogen along with small quantities of other elements such as sulphur. There is natural gas adsorbed in the solid matrix of the coal which is Coalbed methane (CBM), it has becomes an important energy source in some of the countries. It is found that gas contained in Coalbed methane is mainly methane alone with small quantities of ethane, nitrogen, carbon dioxide and few other gases, it is also called 'sweet gas' because it contains small quantities of hydrogen sulphide only. The methane in coal's solid matrix is in a near-liquid state which lining inside the pores of coal.

In Malaysia, compressed natural gas (CNG) is an alternative energy source for car since it burns more cleanly than petrol and diesel. Petroleum price hiking up continuously in the global market forces Malaysians started consider using alternative energy source other than petroleum such as compressed natural gas to save money; therefore the usage of compressed natural gas will be increase with the petroleum selling price.

Study area is bounded by Batang Balingian on the east and Batang Mukah on the west and is situated on the low-lying coastal plains of Mukah district, Mukah division of Sarawak. Ulu Penipah's samples are collected in Sarawak Coal Mine's Ulu Penipah

Area (SE1) located in longitude 112°20°E and latitude 2°50°N which is 26.4km to the south east of Mukah town and Ulu Sikat's samples are collected in Sarawak Coal Mine's Bergrih Area (BC1) located in longitude 112°20°E and latitude 2°40°N which is 36km to the south east of Mukah town.

Lignite coal for shallow and deep seams occurs in the coastal lowlands between Batang Mukah and Batang Balingian. Most of the lignitic coal seams up to 15m thick were penetrated within the Miocene Balingian and Pliocene Liang Formation, usually below a Plio-Pleistocene cover of varying thickness. The prospecting results indicate that although quite a large reserve of coal, estimated at about 270 million tonnes exist in the Mukah-Balingian coal field, the coal is apparently of a hydrous, low rank nature. The use of this coal could only be as thermal or steam coal and the most suitable use would be as a fuel for an on-site or near-site power-generating station to provide electricity to Bintulu or Sibu. Nowadays, new usage of coal is found and Coalbed Methane is collects from lignite coal seam.

Mukah area has been folded into a pair of east-west trending anticline and syncline that plunge to the West which is Bedengan Syncline at the North and Teres-Bakau Anticline at the South. However, the post-depositional deformation is not intense, resulting in folds of low amplitude and large wavelength. Dip angles of bedding planes range from 3° to 36° but generally they are of 8° to 12°. Although there is no big scale faulting but localized faults with minor displacement are common. [8]

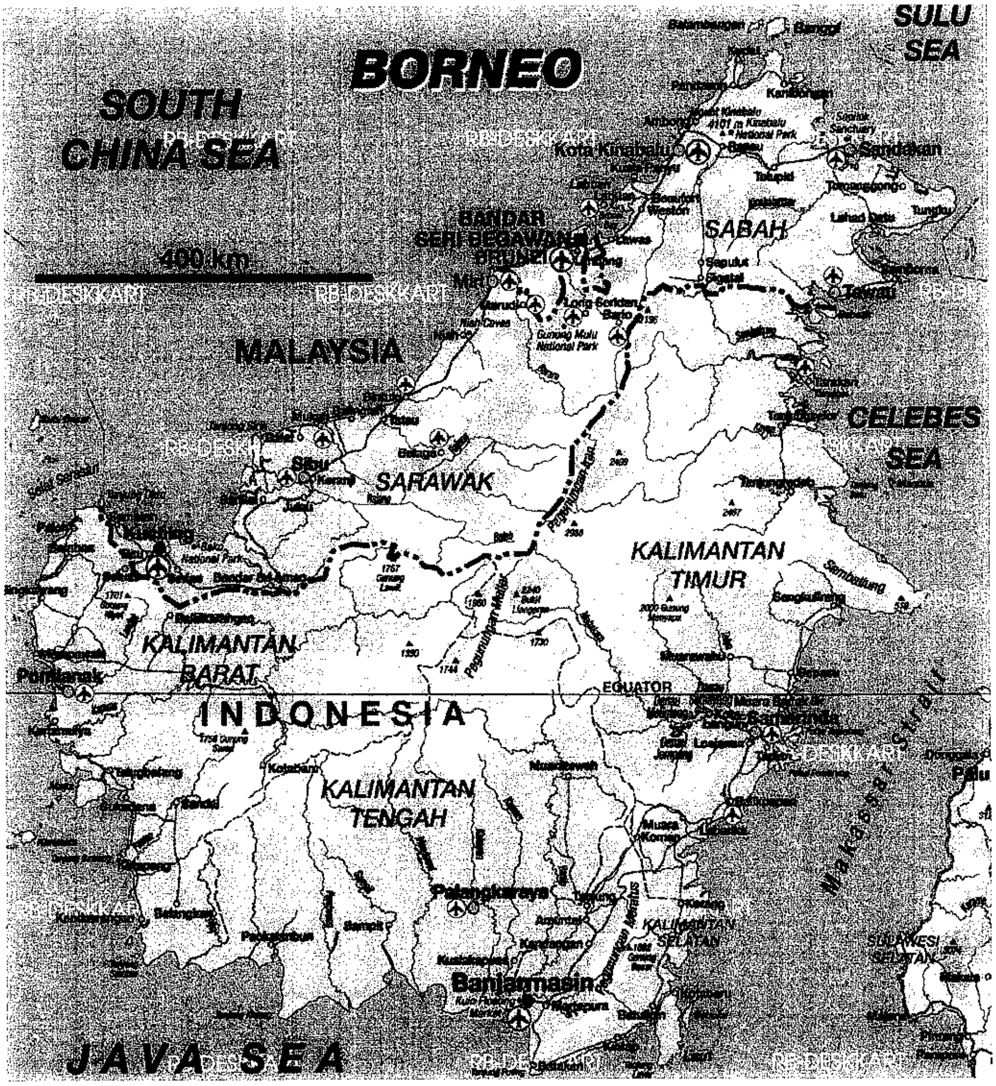


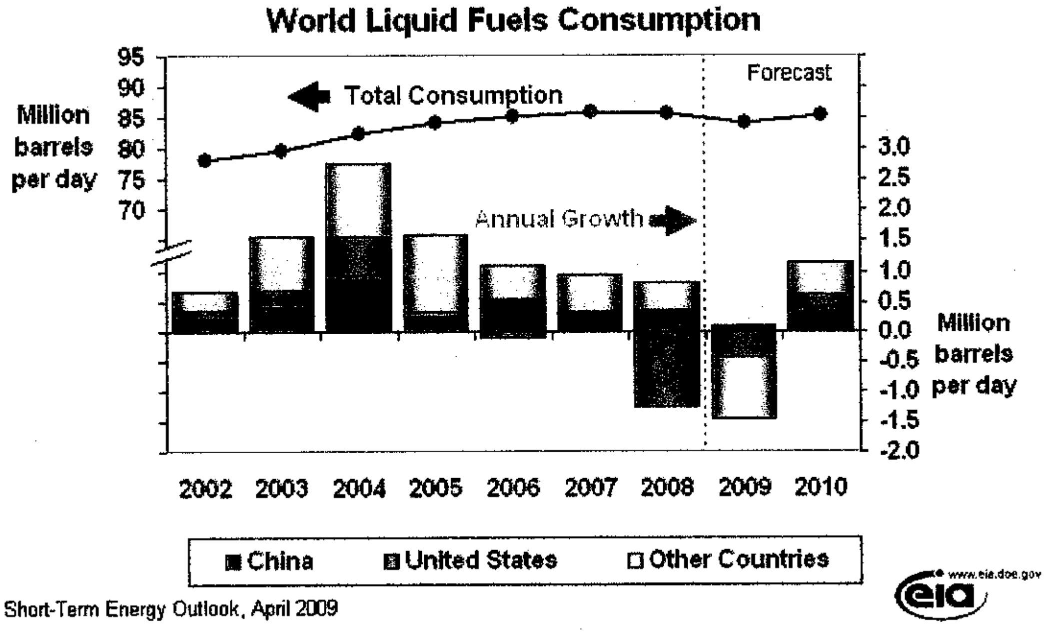
Figure 1.1: East Malaysia map

1.2 Problem Statement

As shown in Figure 1.2 the annual growth of world oil consumption is expected to drop by 1.35 million barrels per day in 2009 compared with previous year due to economic recession but expected to grow by 1.1 million barrels per day in 2010. The total consumption in year 2008 is 85 million barrels per day and is expected to drop to 83 million barrels per day in year 2009 but the consumption is assumed to be increase in 2010.

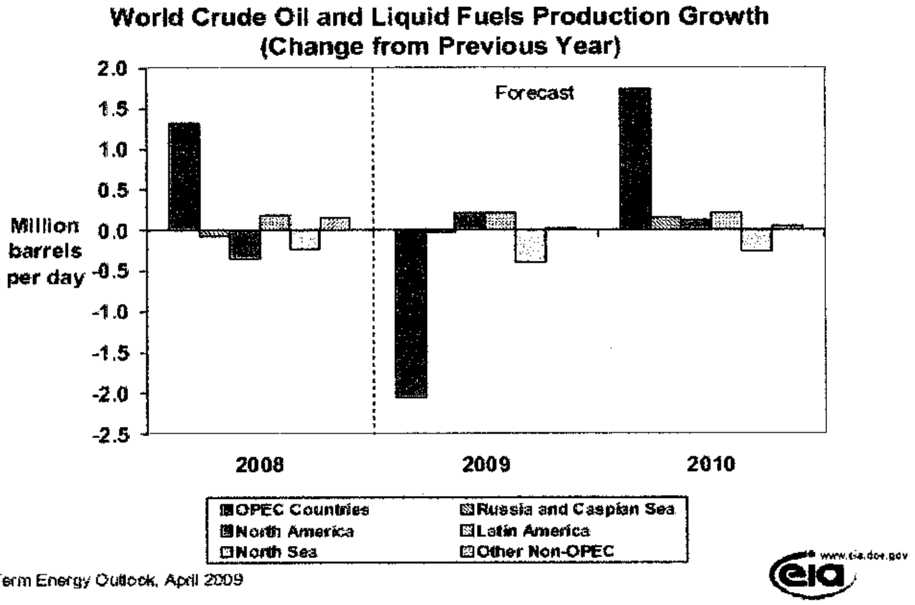
In Figure 1.3 the production from Organization of the Petroleum Exporting Countries (OPEC) is estimated to fell 2.1 million barrels per day in the first quarter of 2009 to 28.5 millions barrels per day and rise to 29.8 millions barrels per day in 2010. Non-OPEC supplies in 2009 are expected be close to 2008 but is expected to increase by a modest 260,000 barrels per day in 2010. Organization for Economic Co-operation and Development (OECD) commercial inventories at year 2008 stood at 2.58 billion barrels which will continue remain above average.

If the petroleum production is reduces, consumption of petroleum will be more than production and therefore the petroleum will be exhausted. Therefore, alternative energy sources must be found to replace the impending worldwide petroleum exhaustion. Coalbed methane is one of the alternatives to extract the natural gas from the coal solid matrix. The physical and chemical properties of coal are analysed to indicate its quality which leads to its possible uses. Correlation is made between the chemical properties and gas content in coal as methane in coal beds may be an alternative energy source.



Short-Term Energy Outlook, April 2009

Figure 1.2: World Liquid Fuels Consumption, 2002-2010



-Term Energy Outlook, April 2009

Figure 1.3: World Crude Oil and Liquid Fuels Production Growth, 2008-2010

1.3 Objectives

The objectives of this project are as follows:

- To analyse physical and chemical properties of Mukah coal.
- To determine gas content in Mukah coal.
- To correlate physical and chemical properties of Mukah coal with gas content.

1.4 Scope of Study

In this research, cope of studies includes understanding the properties of Lignite coal and Bituminous coal and compares the experimental results of Mukah coal with the standard. Literature review must be conduct in order to have better understanding for coal properties and experiments methodology. Experiments are conducted according to standard procedure such as American Society for testing of Materials (ASTM) to find out the properties of Mukah coal. The physical and chemical properties of coal are then correlates with the gas content of Mukah coal.

CHAPTER 2
LITERATURE REVIEW

2.1 Coal

Coal is an organic sedimentary rock that contains varying amounts of carbon, hydrogen, nitrogen, oxygen, and sulphur as well as trace amounts of other elements including mineral matter. Coal is a solid, brittle, combustible, carbonaceous rock formed by the decomposition and alteration of vegetation by compaction, temperature, and pressure over geologic time as discussed below. Coal consists of more than 50% by weight and more than 70% by volume of carbonaceous material. [1]

2.2 Formation of Coal

A geosyncline is a zone of inward movement in the earth's crust. Such an inward movement can only be explained by assuming strong lateral compressive forces acting on the crust. When the lateral compressive stress in such a crustal section exceeds the elasticity limit, deformations are due to occur. This results a thickening of the affected area, which is followed by bulging out at top and root. [3]

Assumed that the geosynclines did not sink at a constant rate causes slow subsidence rate and exceeded by sedimentation, as a result, the lagoons become shallow which favours the growth of aquatic plants and extensive swamps can form. The water in the swamps is nearly stagnant and plant debris continues to accumulate. In a subsequent period, trees grow to a height of tens metres develop on this weak boggy soil. Roots of Carboniferous trees are found in nearly all coal seams which is the clearest proof that the coal has generally been formed in situ (autochtonous formation). After some time, the swamp is submerged and covered by sedimentary deposits and a new future coal seam has formed. [3]

2.3 Types of Coal

Coal classified into various types which included anthracite, bituminous, subbituminous and lignite coal. Anthracite is the highest rank of coal which contains high percentage of fixed carbon and a low percentage of volatile matter. The moisture content of fresh-mined anthracite is generally less than 15% and the heat content is ranges from 22 to 28 million Btu/ton on a moist, mineral-matter free-basis. Anthracite coal is hard, brittle and black lustrous coal.

Bituminous coal has higher quality than lignite coal but poorer quality than anthracite coal. It is a soft coal that black or dark brown in colour which contains of bitumen. Bituminous coal is formed by diagenetic and sub-metamorphic compression to produce macerals, vitrinite and exinite. The carbon content of bituminous coal is around 60% to 80% and moisture content is usually less than 20%. The heat content of bituminous coal ranges from 21 to 30 Btu/ton on a moist, mineral-matter free basis. Bulk density typically around 833 kg/m^3.

Subbituminous coal is a coal that dull, dark brown to blank, and soft and crumbly at the lower end of the range, to bright, black, hard, and relatively strong at the upper end. The inherent moisture by weight is ranges from 20% to 30% and the heat content ranges from 17 to 24 million Btu/ton.

Lignite which also called brown coal is a soft brown fuel with characteristic between coal and peat. Lignite is the lowest rank of coal which is used as a fuel for steam-electric power generation. Lignite has a heat content of 9 to 17 million Btu/ton on a moist, mineral-matter-free basis. Lignite has around 60% of carbon content and high inherent moisture content as high as 66% which will create problems in transportation and storage. The low energy density of brown coal is not worth it if traded to far location, therefore it is often burned in power stations builds very near to the mines.

2.4 Analysis of Coal

Analyses of coal may be reported on different bases with regard to moisture content. Indeed, results that are as-determined refer to the moisture condition of the sample during analyses in the laboratory. Loss of weight during air drying is determined to enable calculation on as as-received basis (the moisture condition when the sample arrived in the laboratory). This is equivalent to the as-sampled basis if no gain or loss of moisture occurs during transportation to the laboratory from the sampling site. Dry basis are calculated on the basis that there is no moisture associated with the sample. Analytical data that are reported on a dry, ash-free basis are calculated on the assumption that there is no moisture or mineral matter associated with the sample.

2.5 Experiments Theory

2.5.1 Porosity and Permeability

Porosity is the fraction of the volume of coal that is occupied by pores and can be calculated from the equilibrium moisture content. Porosity dictates the rate at which methane can diffuse out of the coal. Calculation of porosity is derived from the determination of the true specific gravity and is derived from the relationship:

Porosity =100-100(Apparent Specific Gravity/ True Specific Gravity)

Or

$\text{Porosity}=100 \times \text{density}_{Hg} (1/\text{density}_{Hg} - 1/\text{density}_{He})$

Where:

density_{Hg} : mercury density;

density_{He} : Helium density which displace into the coal in order to measure the porosity.

The porosity and permeability are measured using a POROPERM instrument. POROPERM can determine the properties of coal sample at ambient confining pressure. The gas permeability determination is based on unsteady stated method (pressure falloff) whereas the pore volume is determined using the Boyle's Law technique. Length and diameter of the sample are measured with digital calliper and subsequently bulk volume is determined automatically.

There are four types of volumes definite in porosity measurements which included:

i. The bulk volume or BV: the apparent volume of the sample;

ii. The grain volume or GV: the volume of the whole grains that composed the plug;

iii. The total pore volume or PV: the volume of the whole voids (linked and unlinked) in the plug;

iv. The effective pore volume or PVe: the volume of the linked voids in the plug.

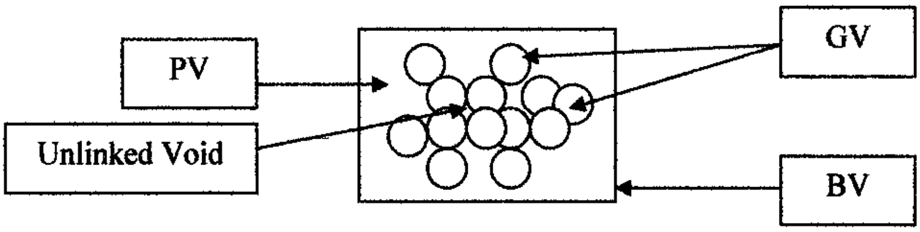


Figure 2.1: Illustration to show the different volumes consist in a system

A common definition of the porosity is Bulk volume (BV) minus pore volume (PV) but there are two types of porosity can be defined using four volumes stated above:

i. The total porosity: ratio between the total pore volume and bulk volume:

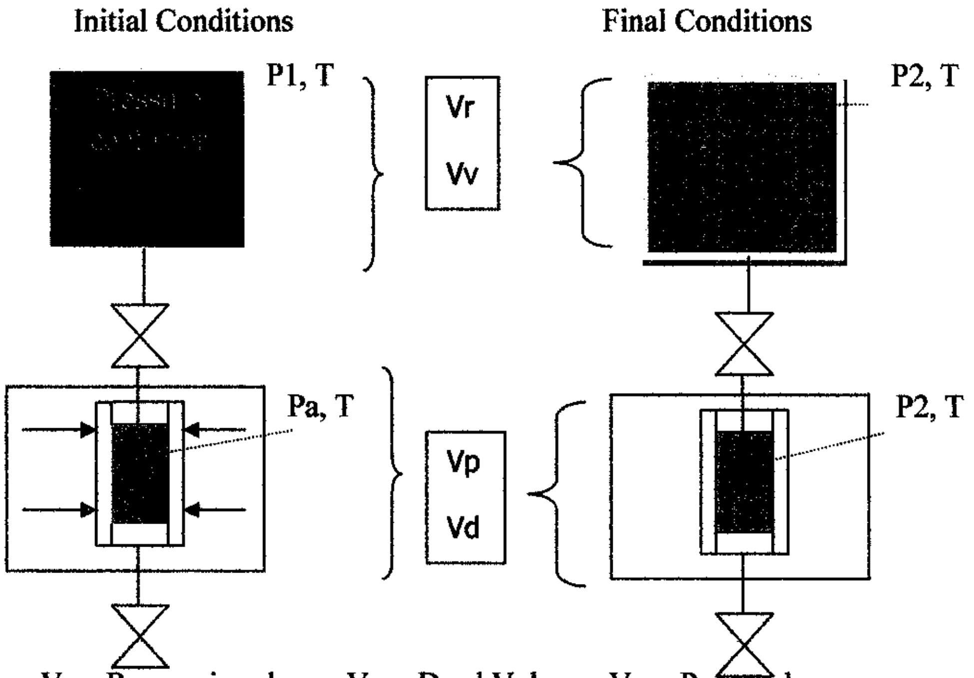
$$\text{Total Porosity} = PV/BV$$

ii. The effective porosity: ratio between the effective pore volume and the bulk volume:

$$\text{Effective Porosity} = PVe/BV$$

The method used to calculate porosity is "Boyle's law Single Cell Method for direct void volume measurement" (API recommended practice 40, February 1998, p 5-12 / 5-18). Pore volume is determined using Helium gas vented into the sample's pore volume. The sample is held in a core holder which utilizes an elastomer sleeve and end plugs. A low confining pressure of 300 psi is applied to the sleeve to hold the coal. The experiment is isothermal.

The initial and final conditions of sample are illustrated in Figure 2.2:



Vr = Reservoir volume; Vv = Dead Volume; Vp = Pore Volume; Vd = Dead Volume

Legend:

- ■ Helium
- ▩ Air
- ▨ Helium and Air mixed

Figure 2.2: Schematic diagram of initial and final conditions of POROPERM

From the real gas law: PV=Nrt

At the initial conditions, the inlet pressure is fixed at 100 psi and the total number of gaseous moles: $n_{(total)} = n_{(helium)} + n_{(air)}$

$$n_{total} = \frac{P_1(Vr + Vv)}{Z_{He1}RT} + \frac{Pa(Vd + Vp)}{Z_a RT}$$

At the final conditions, total number of gaseous moles:

$$n_{(total)} = n_{(helium\ \&\ air\ mixed)}$$

$$n_{total} = \frac{nHe}{ntotal} \bullet \frac{P_2(Vr + Vv + Vd + Vp)}{Z_{He2}RT} + \frac{nair}{ntotal} \bullet \frac{Pa(Vr + Vv + Vd + Vp)}{Z_{a2}RT}$$

Assumption can be made where $P_1 >> P_a$, and then $n_{He} >> n_{air}$.

Thus it comes: $\frac{nHe}{ntotal} \cong 1$ and $\frac{nair}{ntotal} \cong 0$

Hence, the final material balance that gives after simplifications:

$$ntotal = \frac{P_2(Vr + Vv + Vd + Vp)}{Z_{He2}RT}$$

The pore volume can be easily deduced from this relationship:

$$Vp = \frac{Vr(\frac{P_1 x Z_{He2}}{P_2 x Z_{He1}} - 1) - Vv}{1 - \frac{P_a x Z_{He2}}{P_2 x Z_a}} - Vd$$

And the porosity is given by:

Porosity (%) = 100Vp / BV

With apparent volume of the sample BV = $(\pi D^2 / 4) * L$ [8]

Permeability is a property of a porous medium and is a measure of its ability to transmit fluids. The reciprocal of permeability represents the viscous resistivity that the porous medium offers to fluid flow when low flow rates prevail. The effective permeability of a porous medium is a measure of its fluid conductivity to a particular phase of a multi-phase fluid system residing within the medium, where the saturation of each phase is specified. Relative permeability is the ratio of the effective permeability of a particular fluid phase to some arbitrary reference permeability. [8]

Permeability of one meter squared will permit a flow of 1 m^3/s of fluid of 1 Pa.s viscosity through an area of 1 m^2 under a pressure gradient of 1 Pa/m. Therefore, one Darcy equals 0.986923 x 10^{-12} m^2. The common unit is μm^2. One Darcy equals to 0.986923 $\mu m^{2.}$

Transient pressure technique for gases: Pressure-Falloff, Axial Gas Flow measurements (API, Recommended practise 40, February 98, section 6.4). Transient measurements employ fixed-volume reservoirs for gas. These may located upstream of the sample from which the gas flows into the sample being measured. The pressure falloff apparatus employs an upstream gas manifold that is attached to a sample holder capable of applying hydrostatic stresses to a cylindrical plug. An upstream gas reservoir of calibrated volume can be connected to the calibrated manifold volume by using a valve.

The downstream end of the sample is vented to atmospheric pressure and an accurate pressure transducer is connected to the manifold immediately upstream of the sample holder. Therefore the reservoir, manifold and sample are filled with gas. After a few seconds for thermal equilibrium, the pressure transient is initiated by opened the outlet valve. Pressure and times are collected when the upstream pressure has decayed about 85% of the fill pressure. This technique has a useful permeability range of 0.001 to 20,000 milliDarcys.

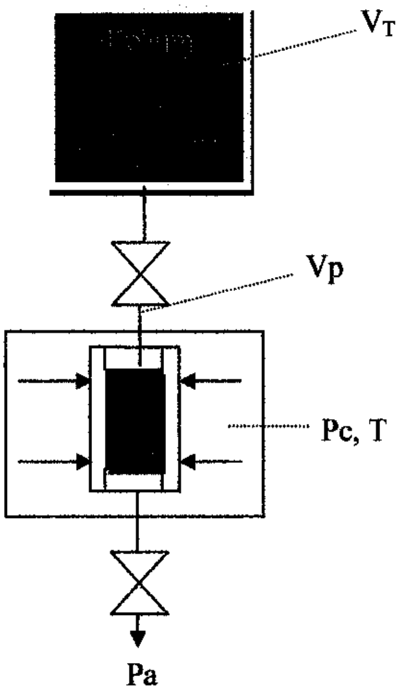


Figure 2.3: Schematic of pressure falloff gas permeameter

Darcy's law established a relationship between the permeability of a porous media and the potential gradient observed during the flow of a fluid through it but this equation is only restricted to low volumetric fluxes.

$$-dP/dL = \mu V/k$$

Where:

μ is the dynamic viscosity of the fluid

V is the volumetric flow of the fluid

K is the permeability

-dP/dL is the potential gradient

At higher fluxes, Forchheimer observed that the potential gradient required for a given volumetric flux is greater than predicted by Darcy's law by an amount proportional to the product of fluid's density, ρ and the square of volumetric flux V. the coefficient of proportionality, β (ft^{-1}) is the inertial resistivity of the porous medium which is due to accelerations a fluid undergoes as it travels tortuous paths through a porous media.

$$-dP/dL = \mu V/k + \beta\rho V^2$$

The permeability of a porous medium to gas is dependent upon the mean free path of the flowing gas due to a phenomenon known as slip, as a consequence the apparent permeability of a porous medium varies with pressure and give a relationships that when pressure increase, the permeability increase. Klinkenberg permeability, k_∞ is equal to the permeability obtained using non-reactive liquid which is invented to avoid the problem of obtaining pore-pressure-dependent gas permeability. [8]

$$k(x, t) = k_{\infty}(1 + b / [P(x, t) + Pa])$$

Where: b (psi) is the slip factor, b>0.

The gas permeability is uncorrected for gas slippage and it is obtain from the following relationship:

$$k_g = k_{\infty}\left(1 + \frac{\frac{b_m \mu_c}{\mu_m}\sqrt{\frac{T_c M_m}{T_m M_c}}}{1/2P_g + P_a}\right)$$

Where:

Subscript m = measured, Subscript c = theoretical

b = slip factor (psi)

μ = viscosity of the gas (cp)

M = molecular weight of the gas (g/mol)

T = temperature (oF)

Pg = geometrical mean pressure (psia)

Pa = atmospheric pressure (psi)

kg = gas permeability (md)

2.5.2 Moisture Content

Calculation the percent moisture in the analysis sample is as follows:

$$\text{Moisture in sample, \%} = [(A-B)/A] \times 100$$

Where:

A = Grams of sample used, and
B = Grams of sample after heating

Repeatability, reproducibility and bias should be used for judging the acceptability of results. For repeatability and reproducibility criteria of the results, the duplicate results by the same laboratory should be less than 0.2% for coals having less than 5% moisture and 0.3% for coals having more than 5% moisture.

2.5.3 Analysis of Gas Content by GC

The number of significant digits retained for the quantitative value for each component shall be such that accuracy is neither sacrificed nor exaggerated.

For Pentanes and Lighter components, the heights of each component peak is measured and convert to the same attenuation for corresponding components in the sample and reference standard, and calculate the concentration of each component in the sample as follows:

$$C = S \times (A/B)$$

Where:

C = Component concentration in the sample, mol%,

A = Peak height of component in the sample, mm,

B = Peak height of component in the standard, mm, and

S = Component concentration in the reference standard, mol%

If air has been run at reduced pressure for oxygen or nitrogen calibration, or both, correct the equation for pressure as follows:

$$C = S \times (A/B) \times (Pa/Pb)$$

Where:

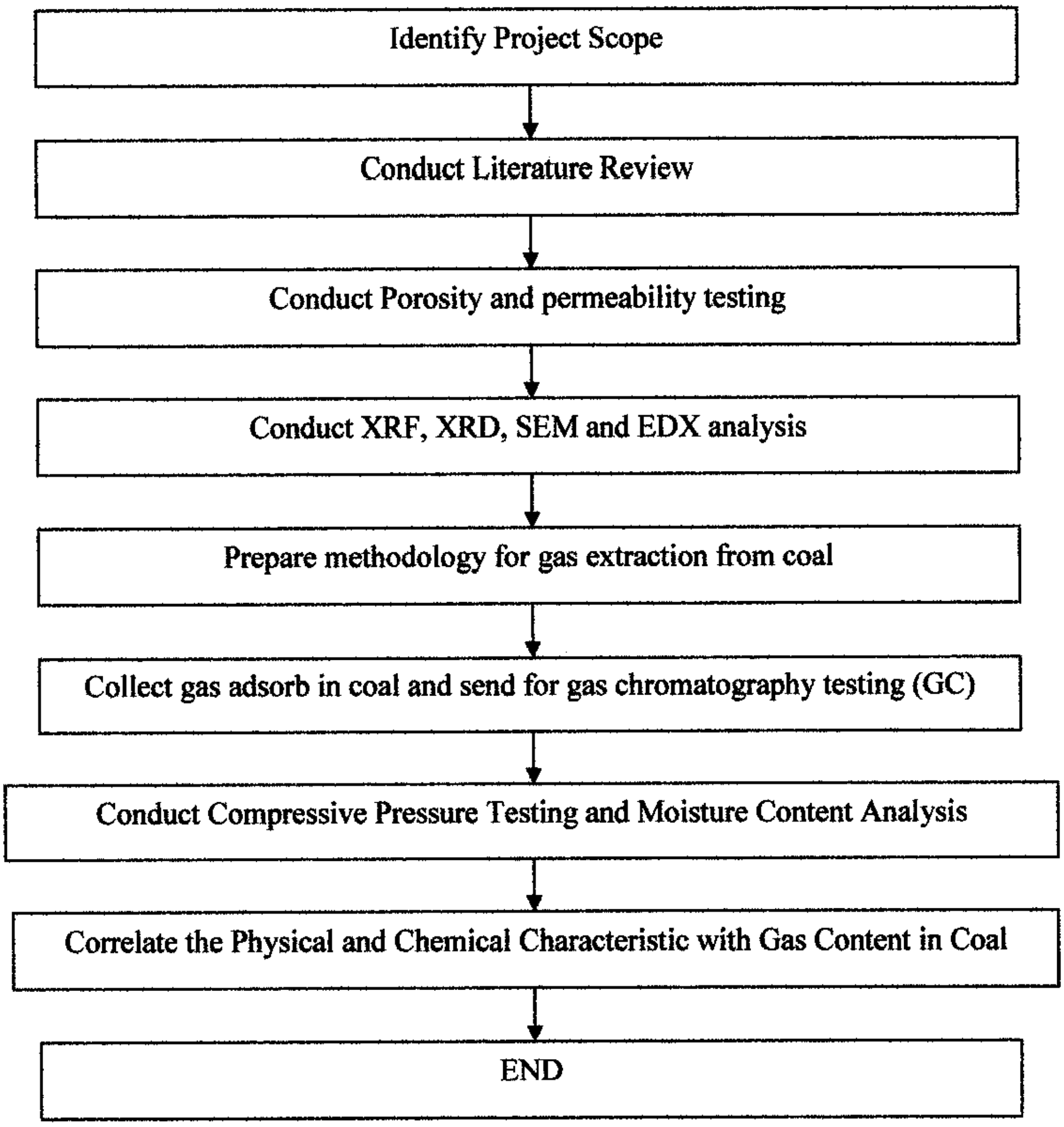
Pa = Pressure at which air is run, and

Pb = True barometric pressure during the run, with both pressures being expressed in the same units.

CHAPTER 3

METHODOLOGY

3.1 PROJECT FLOW DIAGRAM



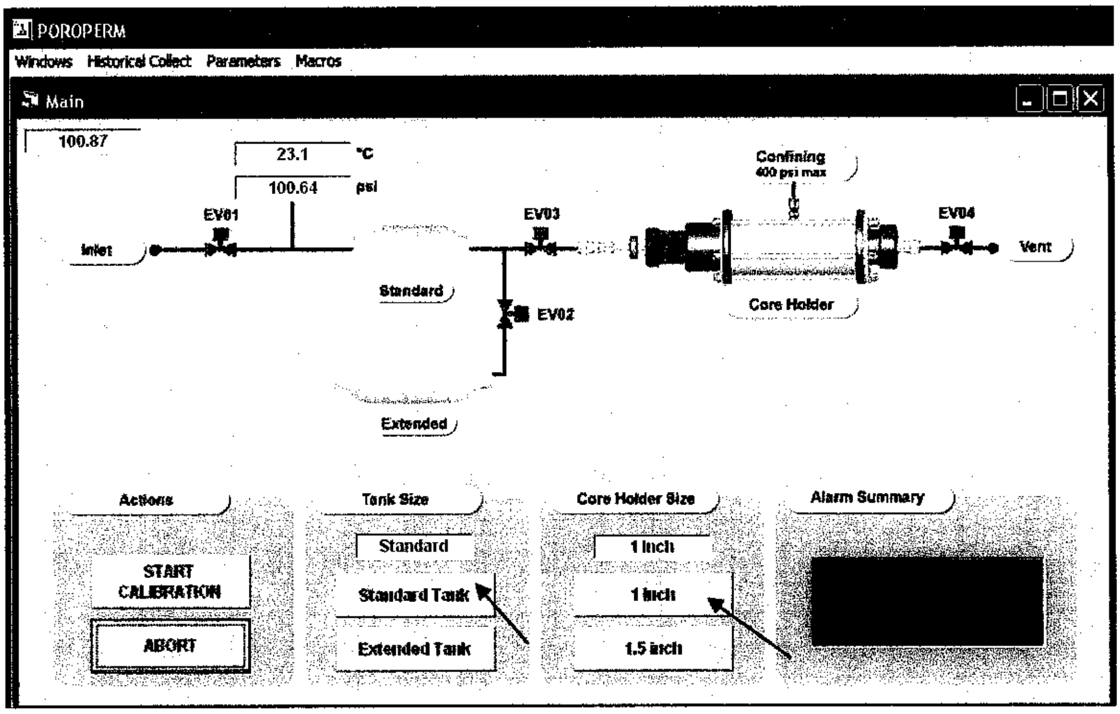
3.2 EXPERIMENTAL PROCEDURES

Firstly, any particular coal that needs to be tested must be ensure that the sample is a true representative of the bulk material and does not undergo any chemical or physical changes after completion of the sampling procedure and during storage prior to analysis. [1]

Before laboratory experiments conducted, a gross sample (a sample that represents a lot of coal which neither reduction nor division has been performed) is prepared. Once a gross sample has been taken, it is reduced in both particle size and quantity to yield a laboratory sample. Mechanical sample divider is used for division of the sample and mechanical crushing equipment for reduction of the sample.

There are two types of chemical analysis used to classify coals which is the proximate analysis and the ultimate analysis. Proximate analysis classifies coals by giving the relative amounts of moisture, ash, volatile matter and fixed carbon by difference with caloric value is reckoned. Ultimate analysis gives the amounts of the principal chemical elements in coal such as carbon, hydrogen, oxygen, nitrogen and sulphur. [1]

First properties of coal to be measured are porosity and permeability; it is measured using a POROPERM instrument. The experiment started by changing the core holder from 1.5 inch core holder to 1 inch core holder. Firstly, the Teflon line and stainless steel line of confining from the core holder are disconnected. Take out the core holder and replaced it using new core holder. Connect the Teflon line and stainless steel line of confining to the new core holder. Software application which is used to control the POROPERM is started up. Select the "Standard Tank" for the tank size and "1 inch" core holder size in the window shown in Figure 1.



100.87

23.1 °C

100.64 psi

Tank Size: Standard

Core Holder Size: 1 inch

Figure 3.1: POROPERM application window

Before starting a new series of measurement, create a new file template using file directory C:\APPLILAB\PROJECT\POROPERM\EXCEL FILE\Template.xls and rename it. Fill in the "Info" sheet with sample ID, diameter, length, weight and atmospheric pressure for each sample.

Next, choose the measurement that need to be tested for the samples which is porosity and permeability. The experiments is continues by loading the sample to measure into the core holder and start the measure sequence by clicking on the button "Start measure". The name of the measure file must be stated before the operation stated to enable the readings generated in the POROPERM can be recorded in excel.

The next experiment conducted is to measure the moisture content. Thermal drying method is most commonly used to identify total moisture in coal. For this testing, moisture determination by measuring weight loss of a coal sample (about 1 g) of heating in a flow of dry, oxygen-free nitrogen to temperature 107 ± 3°C for 1 hour. Most of the low-rank coals with high moisture content are susceptible to oxidation; therefore the heating should be done in an inert atmosphere.

The analysis continues with determine the mineral composition of coal, mineral composition can be determined by using X-Ray diffraction (XRD) or X-Ray fluorescence (XRF). Gas content in the coal can also be identified using gas chromatography method.

3.3 Tools Required

3.3.1 Core Cutting Machine

Core cutting machine is used to cut the coal gross sample into laboratory sample size. For POROPERM, the diameter of core plug can be 1 inch or 1.5 inch and the length of the core plug must not longer than 4 inch. Core cutting machine is uses to cut the coal into core plug with 1 inch or 1.5 inch diameter. The cooling water need to be used in order to cool down the core when drilling the coal.

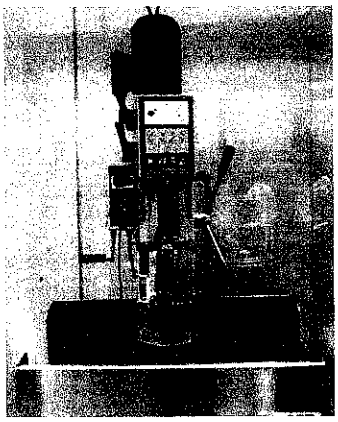


Figure 3.2: Core cutting machine

3.3.2 Core Trimming Machine

Core trimming machine is used to trim out the unsmooth surface of core plug and trim the core plug into desired length. For the laboratory sample used in POROPERM, the length of sample used is 1 inch. The cutter for this machine is very hard since it has diamond on the cutting edge which able to cut very hard material. The cooling water need to be used in order to cool down the core when trimming.

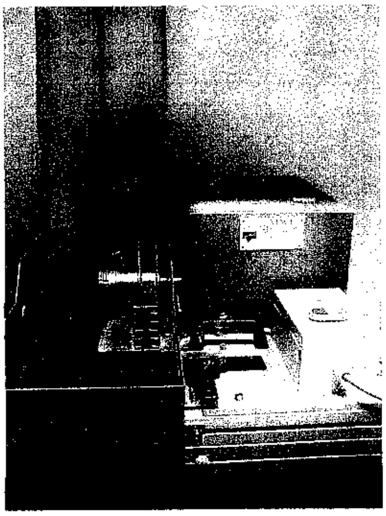


Figure 3.3: Core trimming machine

3.3.3 POROPERM

The POROPERM instrument is a permeameter and porosimeter used to determine properties of plug sized core samples at ambient confining pressure. The instrument offers reporting and calculation facilities other than the direct properties measurement. The gas permeability is calculated based on the unsteady stated method while the porosity is determined using Boyle's Law equation. The direct measurements using POROPERM are:

i. gas permeability, pore volume

ii. core length and diameter

The calculated parameters are included:

i. Klinkenberg slip factor "b"

ii. Klinkenberg corrected permeability

iii. Inertial coefficients

iv. Sample bulk volume and porosity

v. Grain volume and grain density.

3.3.4 XRD Analysis

X-Ray Diffraction (XRD) analysis is the most direct and accurate analytical method for determining the presence and absolute amounts of mineral species in a sample. X-Rays of a known wavelength are passed through a sample to be identified the crystal structure, the X-Ray wave will diffracted by the lattice of the crystal to give a unique pattern of peaks of reflection at different angles and intensity.

When a beam of monochromatic X radiation is directed at a crystalline material, the reflection or diffraction of the X-rays at various angles with respect to the primary beam is observed.

3.3.5 Sputter Coater

Sputter coater is uses to allow deposition of a suitable coating such as gold to increase the electrical conductivity of a specimen before put into Scanning Electron Microscope (SEM). Sputtering means the erosion of the target material when bombarded with fast heavy particles and the sputtered atoms will condense on the surface of the specimen to be coated. The above process taking place in the conditions of a gaseous glow discharge between an anode and cathode is termed sputtering.

Under gaseous glow discharge condition, ion bombardment of cathode will occur and results in the erosion of the cathode material, the subsequent omni-directional deposition of the sputtered atoms forming coatings of the original cathode material. The most common arrangement is put the target material to be sputtered which is Gold as negative cathode and the specimens to be coated on anode which is usually earthed to the system.

3.3.6 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) is a microscope that uses electrons to form an image. SEM produces images of high resolution and closely spaced features can be examined at a high magnification. The specimens that tested using SEM must be electrically conductive, if the specimen is non conductive, a conductive thin film is coated to reduce thermal damage and improve secondary electron emission using sputter coater.

There are few requirements when preparing the samples which included removes all water, solvents or other materials that could vaporize while in the vacuum, non-metallic specimens must be coated and finally is firmly mount all the specimens.

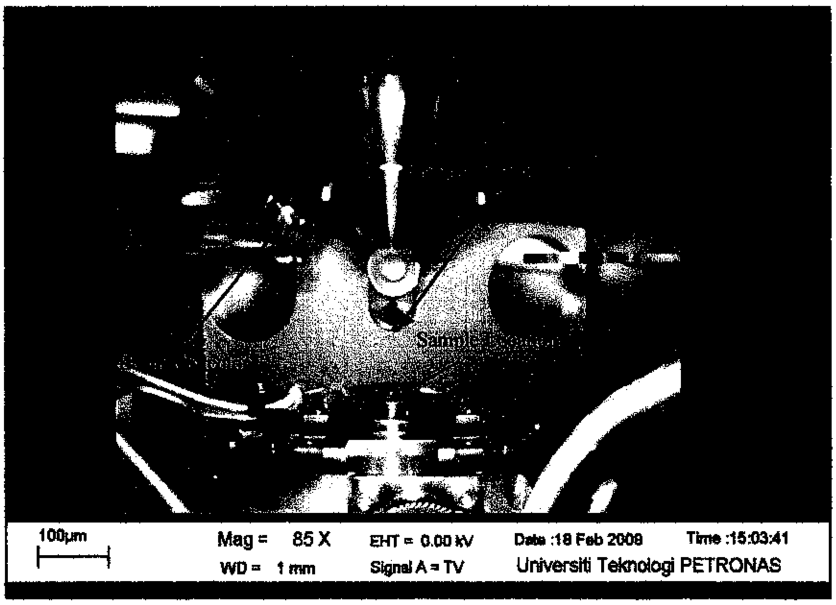


Figure 3.4: SEM inner view

3.3.7 Energy Dispersive X-Ray (EDX)

This EDX analysis system works as an integrated feature of a scanning electron microscope (SEM). EDX is used to identify the elemental composition of the specimen at an area of interested or a single point.

The specimen must be conductive to enable the electron beam bombarded through the specimen. The bombarded electrons collide with the specimen atoms' own electrons and knocking some of them off in the process. An inner shell electron is ejected and the vacated position is occupied by a higher-energy electron from an outer shell. The transferring of the outer electron will give up some of the energy by emitting an X-ray. The atom of every different element release X-rays with different amounts of energy and the identity of the atom will be identified by measuring the amounts of energy present.

Output of an EDX analysis is an EDX spectrum which normally displays peaks corresponding to the energy levels for the X-ray received. If the element is concentrated in the specimen, the peak of spectrum will be higher.

3.3.8 Drying Oven

Drying oven is typically used for the removal of moisture from water-based coatings and adhesives. Drying Oven is also used for removing water from the surface or interior of certain products or substrates.

Figure 3.5: Drying Oven

3.3.9 X-Ray Fluorescence (XRF)

XRF is the emission of fluorescent X-rays from a material that has been bombarded with high-energy gamma rays.

Figure 3.6: X-ray Fluorescence (XRF)

3.3.10 2000kN Flexural and Compression Machine

This machine can be used to test either cubes of cylinders, the information that can be obtain using this machine included the maximum load, stress and pace rate that applied to the sample.

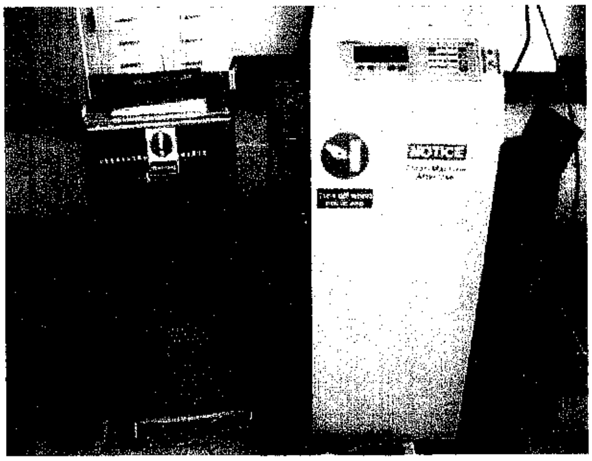


Figure 3.7: 2000kN flexural and compression machine

3.3.11 Gas Chromatography (GC)

The purpose of conduct this experiment is identify the gas adsorb inside the coal matrix, the gas used to force the gas adsorb is Helium gas since the Gas Chromatography equipment unable to detect Helium gas and therefore the composition of the gas content will not be affected by the Helium gas. First of all, the gas content should be extracted out from coal matrix using the equipment setup shown in Figure 3.8. After that, the gas collected is inserted into Gas Chromatography equipment as shown in Figure 3.9 and the composition of gas content will be shown in different spectrum peak.

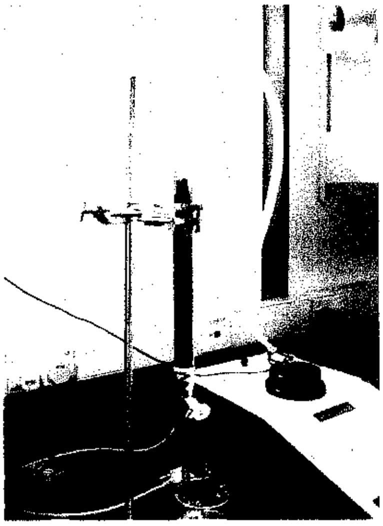


Figure 3.8: Gas Extraction Equipment setup

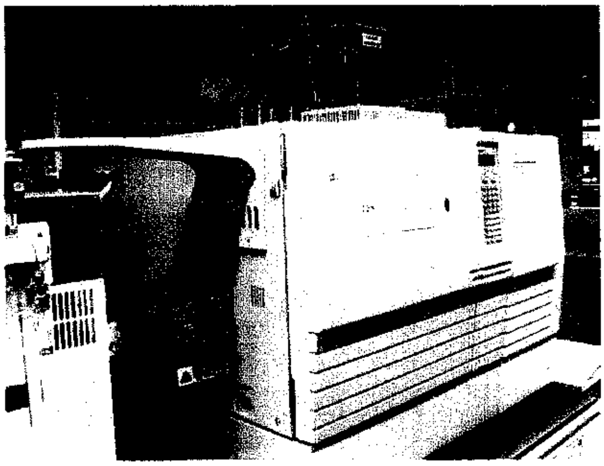


Figure 3.9: Gas Chromatography Equipment

3.4 GANTT CHART

Table 3.1: Gantt chart

No	ACTIVITIES / WEEK	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15-17	18	19	20
1	Briefing and update on progress								MID SEM BREAK											
2	Study and confirmed ASTM , Conduct Experiment such as XRD, XRF, SEM	▨	▨	▨																
3	Submission of Progress Report 1				**13/2**															
4	Conduct Experiment such as GC				▨	▨	▨	▨												
5	Submission of Progress Report 2									**4/4**										
6	Seminar										▨	▨	▨							
7	Conduct Experiment for compressive strength and moisture content									▨	▨	▨	▨							
8	Poster Exhibition, Pre-EDX, Progress Reporting											**8&9/4**								
9	Submission of Dissertation													**30/4**						
10	Final Oral Presentation																	▨	▨	
11	Submission of Project Dissertation																			**12/6**

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Porosity and Permeability Test

The samples used for analysis are taken from two different coal basins in Mukah area in Sarawak which is Ulu Sikat and Ulu Penipah. From the observation of physical outlook of core plug, coal collected from Ulu Penipah basin is brownish black in colour, soft and easily fractured, coal in Ulu Sikat basin is black in colour, hard, brittle and glossy as shown in picture below.

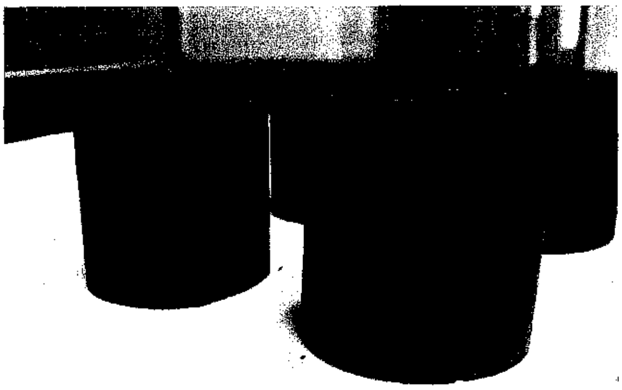


Figure 4.1: Picture of core plug from Ulu Sikat

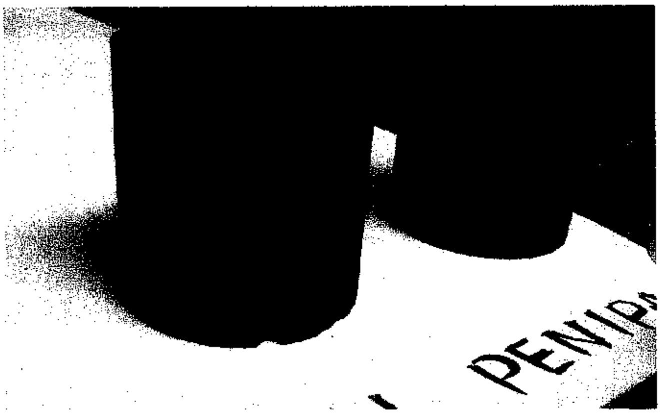


Figure 4.2: Picture of core plug from Ulu Penipah

Before conduct the experiments, measure the diameter and length of the core plug using electronic vernier scale which has 0.01 mm precision, measure the weight using electronic weighing scale which has 0.001 g precision. In order to get an accurate reading, at least 3 to 5 reading of diameter and length in different section of core plug are measure and average of diameter and length are used as final core data.

Table 4.1: Results of diameter measurement of core plug in different section

Sample N°	**Sample ID**	**Core Diam 1 (mm)**	**Core Diam 2 (mm)**	**Core Diam 3 (mm)**	**Core Diam 4 (mm)**	**Core Diam 5 (mm)**	**Core Diam average (mm)**
1	Ulu Penipah	25.300	25.260	25.290	25.310	25.240	25.28
2	Ulu Sikat	25.510	25.260	25.240	25.120	25.170	25.26

Table 4.2: Results of length measurement of core plug in different section

Sample N°	**Sample ID**	**Core Length 1 (mm)**	**Core Length 2 (mm)**	**Core Length 3 (mm)**	**Core Length 4 (mm)**	**Core Length 5 (mm)**	**Core Length average (mm)**
1	Ulu Penipah	25.530	25.540	25.540	25.520	25.570	25.54
2	Ulu Sikat	25.510	25.750	25.340	25.300	25.650	25.51

The pressure use for core plug measurement is atmospheric pressure, 14.7 psi and the temperature is range from 23°C to 24°C. The effective flow area, bulk volume, radial shape factor and axial shape factor are calculated in the POROPERM excel sheet which the formula is fixed such as:

i. Effective Flow Area $= \pi r^2 = \pi \times (24.82/2)^2 = 483.83$ mm ^{2}
ii. Bulk Volume $= \pi r^2 L = \pi \times (24.82/2)^2 \times 24.99 = 12.09$ cc

Table 4.3: Properties of core plug and experiment condition in laboratory

Sample No	**Sample ID**	**Core dia (mm)**	**Core Length (mm)**	**Bulk Vol (cc)**	**Weight (g)**	**Room temp. (°C)**	**Atmosph. Pressure (psi)**
1	Ulu Penipah	25.28	25.54	12.82	16.078	23.20	14.70
2	Ulu Sikat	25.26	25.51	12.78	15.566	23.80	14.70

Table 4.4: Average length and diameter, others factor of core plug

Sample N°	**Sample ID**	**Core Diam average (mm)**	**Core Length average (mm)**	**Effective Flow Area (mm^2)**	**Bulk Volume (cc)**	**Radial Shape Factor**	**Axial Shape Factor**
1	Ulu Penipah	25.28	25.54	501.93	12.82	1.00	1.00
2	Ulu Sikat	25.26	25.51	501.14	12.78	1.00	0.99

The Helium gas is used to pass through the core plug to analysis porosity and permeability in POROPERM. By using POROPERM, the pore volume and grain volume are identified while the effective core porosity are calculated using the value identified. If a low isostatic confining pressure of less than 500 psi is applied to the sleeve by means of a confining liquid circuitry, the equation used to calculate porosity is stated below.

$$\begin{aligned} \text{Porosity (\%)} &= 100\ V_p / BV \\ &= 100 \times (9.55 / 12.09) \\ &= 78.99\% \end{aligned}$$

Where:

Vp: Pore Volume (cc)

BV: Bulk Volume (cc)

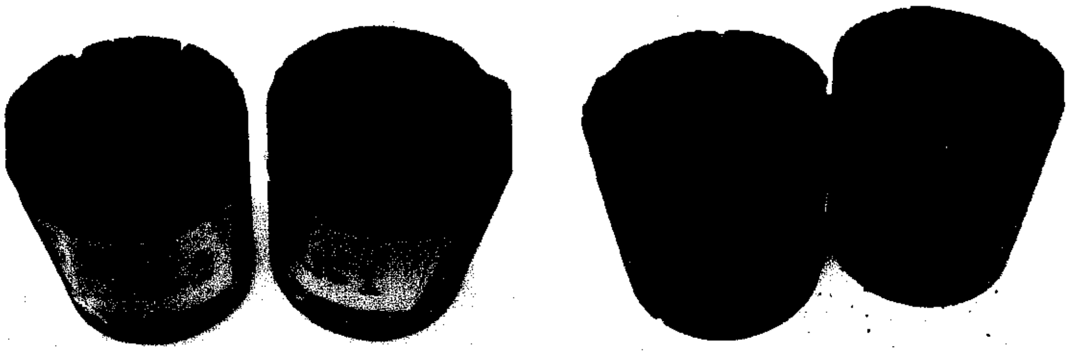


Figure 4.3: Front and back view of two coal samples from Ulu Sikat

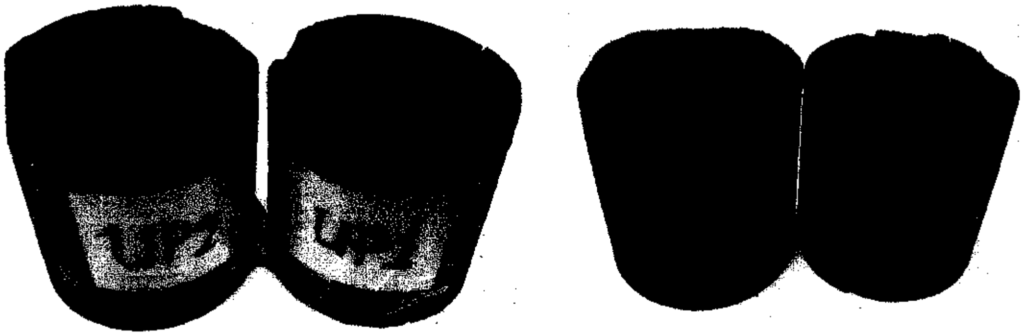


Figure 4.4: Front and back view of two coal samples from Ulu Penipah

Table 4.5: Results of porosity tested and calculated by using POROPERM

Sample No	**Sample ID**	**V Grain (cc)**	**Pore Vol selected (cc)**	**Pore Vol calculated (cc)**	**Grain density (g/cc)**	**Effective Core Porosity (%)**
1	Ulu Penipah	0.92	11.90	11.90	17.48	92.82%
2	Ulu Sikat	3.02	9.76	9.76	5.15	76.37%

From the results in table 4.5, in general the porosity of coal sample collected from Ulu Penipah and Ulu Sikat are very high which is more than 75%. The experiments have some errors exist since the fractured in core plug due to the brittle properties of Mukah coal, the fractured coal which is as shown in Figure 4.3 and Figure 4.4 is calculated as pores volume which might not be fully accurate.

From the readings, it shows that coal sample from Ulu Penipah has the higher porosity than Ulu Sikat's coal sample. Further analysis should be conduct to identify the methane gas percentage in coal.

Table 4.6: Results of permeability tested and calculated by using POROPERM

Sample No	**Sample ID**	**Permeability K_{air} (mD)**	**Permeability $K_{Klikenberg}$ (mD)**	**Factor Alpha**	**Factor Beta**
1	Ulu Penipah	0.1	0.096	2.75E+03	7.82E+13
2	Ulu Sikat	33.552	33.487	1.02E+01	9.50E+08

In Table 4.6, it shows that permeability Klinkenberg calculated is smaller than permeability of coal to gas is because of the inertia effects is properly accounted. Permeability of coal sample from Ulu Penipah is very low compare to coal sample from Ulu Sikat. Ulu Penipah's coal has a porosity of 92.82% but has a very low permeability which is 0.1mD, this happens due to there is a lots of dead volume but not effective pores volume, dead volume is the pores volumes that are not connected to each other and therefore it is not able to transmit fluids through it. Ulu Sikat's coal has higher permeability of 33.552 mD which means that there are more pores volumes that are connected in the coal sample compare to Ulu Penipah's coal.

4.2 Pore Size and Structures of Coal

Scanning Electron Microscope (SEM) can magnify an object up to 30,000 times than its original size. There are some information that stated in the image of SEM which included walking distance, it is the distance between the electron beam gun and the sample. Another information is Energy High Tension (EHT) which is the energy used for the electron to penetrate the sample. Figure 4.5 shows the image of Ulu Penipah's coal sample taken by using SEM at magnification of 3000 times to original size at walking distance of 11mm and EHT of 15kV which is able to penetrate the sample until certain thickness. From the scale stated in the image, the average pores size is found to be 5µm. The sample is very brittle since there are many fractures on the coal surface.

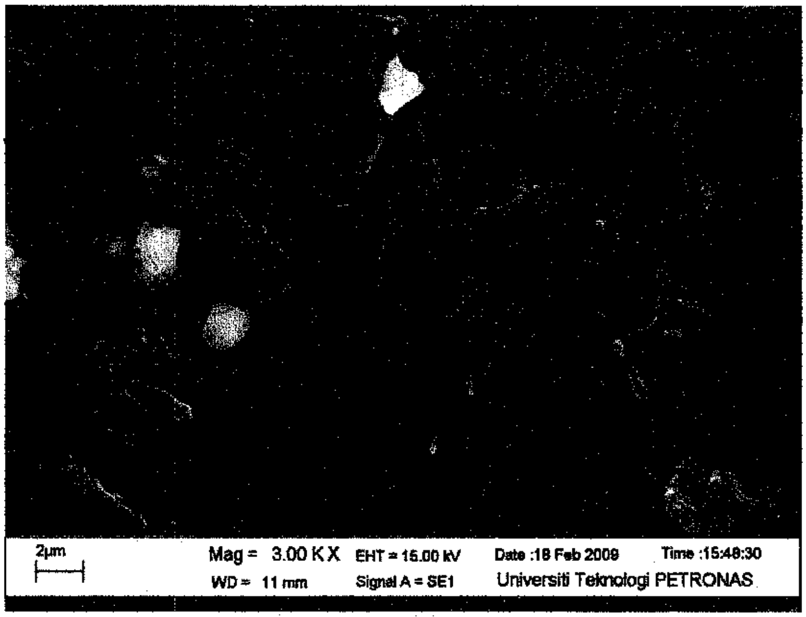


Figure 4.5: Ulu Penipah sample's image using SEM at magnification of 3000x

Figure 4.6 shows the image of Ulu Sikat's coal sample taken by using SEM at magnification of 3000 times to original size at walking distance of 10mm and EHT of 15kV. From the scale stated in the image, the pores size for this specimen is found to be 1µm. The sample is very brittle since there are many fractures on the coal surface but if compare with Ulu Penipah's sample, the fracture in Ulu Sikat's sample is more obvious.

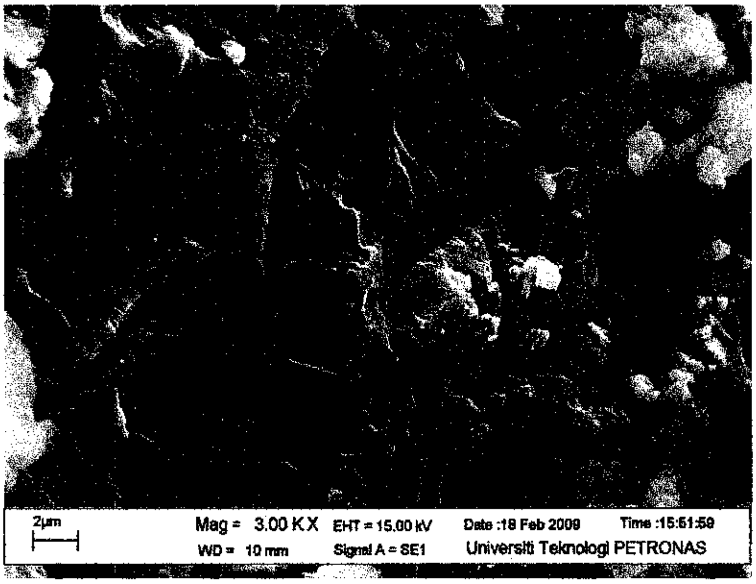


Figure 4.6: Ulu Sikat sample's image using SEM at magnification of 3000x

The pore size for Ulu Penipah's coal is larger than Ulu Sikat's coal. This results show that the Ulu Penipah's coal has a high porosity might because of the large pore size in the coal but the pores are not connected and therefore gives a very low permeability. Ulu Penipah's coal is higher rank coal compare with Ulu Sikat's coal, theoretically it should contain lower porosity but the results is not accurate, this might because of the fracture of the internal of coal that cannot be observed and immature of Ulu Penipah's coal.

4.3 Composition of Coal

The composition of Ulu Penipah's coal sample is analyzing using Energy Dispersive X-ray (EDX) and the results are shows in Figure 4.7 and Table 4.7. From Table 4.7, it is found that there are total of seven elements which included Carbon, Oxygen, Aluminium, Silicon, Phosphorus, Potassium and Calcium but the major component is Carbon which is 60.55wt%. This result shows that there are many impurities in this sample but detail XRD and XRF testing will be conducted to confirm the composition of the coal.

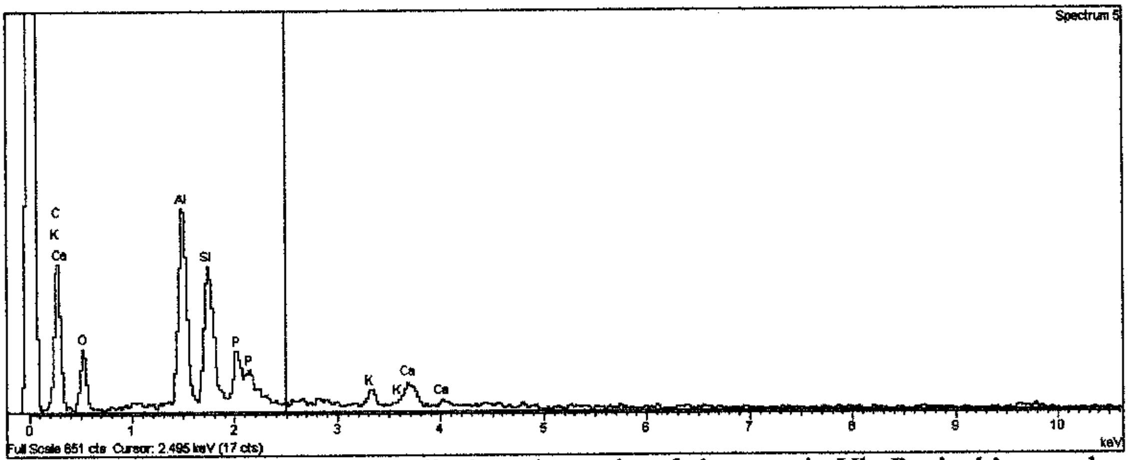


Figure 4.7: EDX spectrums that displays the peaks of elements in Ulu Penipah's sample

Table 4.7: Composition of Ulu Penipah's coal sample in weight percent using EDX

Element	Weight %	Atomic %
Carbon, C	60.55	70.82
Oxygen, O	25.49	22.38
Aluminium, Al	6.31	3.28
silicon, Si	4.49	2.25
Phosphorus, P	1.49	0.68
Potassium, K	0.68	0.25
Calcium, Ca	0.99	0.35
Totals	100	100

From Table 4.8, it is found that there are only two major elements which is Carbon and Oxygen in Ulu Sikat's coal sample, the major component is Carbon which is 72.94wt%. This result shows a higher composition of Carbon compare to Ulu Penipah's coal sample, this might because of the existence of impurities in Ulu Penipah's coal sample, theoretically, Ulu Penipah's coal sample should have higher Carbon content then Ulu Sikat's coal sample since Ulu Penipah's sample is bituminous coal which has higher rank and Carbon content than Ulu Sikat's sample which is Lignite coal. The standard range for carbon content in Lignite coal is around 60% and Bituminous coal is 60% to 80%, both the experimental results are close to the standard range.

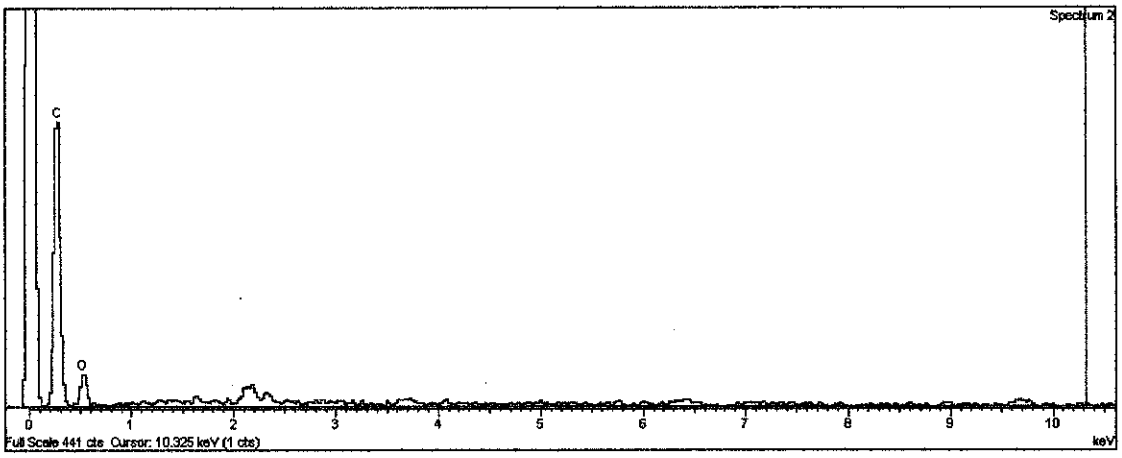


Figure 4.8: EDX spectrums that displays the peaks of elements in Ulu Sikat's sample

Table 4.8: Composition of Ulu Sikat's coal sample in weight percent using EDX

Element	**Weight %**	**Atomic %**
Carbon, C	72.94	78.21
Oxygen, O	27.06	21.79
Totals	100	100

Another experiment is conducted which is X-Ray Fluorescence to determine the composition of the coal. The result is show in Table 4.9. The results obtained did not contain Carbon due to low molecular weight since gamma ray unable to detect components with low molecular weight. From the results, it shows that Ulu Sikat's coal having a high percentage of Ferum which causes the coal to be brownish in colour after coring while Ulu Penipah's coal shows a high percentage of Silicone in the composition.

Table 4.9: Composition of Mukah coal using XRF

Component	% in Ulu Sikat Coal	% in Ulu Penipah Coal
Al	1.06%	9.02%
Ca	16.20%	6.28%
Compton	0.07%	0.11%
Cu	0.41%	0.18%
Fe	32.10%	6.14%
K	0.51%	3.01%
Mg	0.72%	0.00%
Mn	0.55%	0.00%
Mo	0.36%	0.13%
O	35.60%	44.50%
Pd	1.43%	3.31%
Pd	0.57%	1.34%
Rayleigh	0.52%	0.61%
S	5.58%	4.74%
Si	2.19%	14.90%
Sr	0.76%	3.08%
Ti	0.26%	1.81%
Zn	0.70%	0.50%

4.4 Moisture Content and Compressive Strength of Coal

Moisture is determined by establishing the loss in weight of the sample when heated under rigidly controlled conditions of temperature, time and atmosphere, sample weight, and equipment specifications. [10]

Calculation of the percent moisture in the analysis sample is as follows:

$$\text{Moisture in analysis sample, \%} = [(A-B)/A] \times 100$$

where:

A = grams of samples used, and

B = grams of sample after heating

Table 4.10: Moisture content for Mukah Coal

Sample	Weight before Heating (g)	Weight after Heating (g)	Moisture Content, %
Ulu Sikat Coal	41.3437	35.8878	13.20
Ulu Penipah Coal	133.0834	126.1839	5.18

Ulu Sikat's coal has a higher porosity and permeability than Ulu Penipah's coal which lead to a higher moisture content of the coal. Theoretically Bituminous coal has lower moisture content in Ulu Sikat's coal which is accurate in this experiment. The high porosity but low moisture content of Ulu Penipah's coal might because of water lost during transportation of coal.

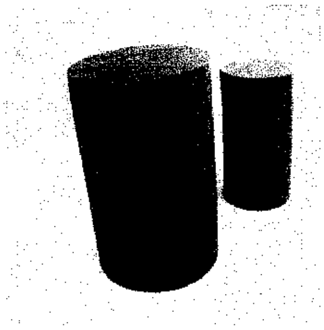


Figure 4.9: Mukah Coal before Compression

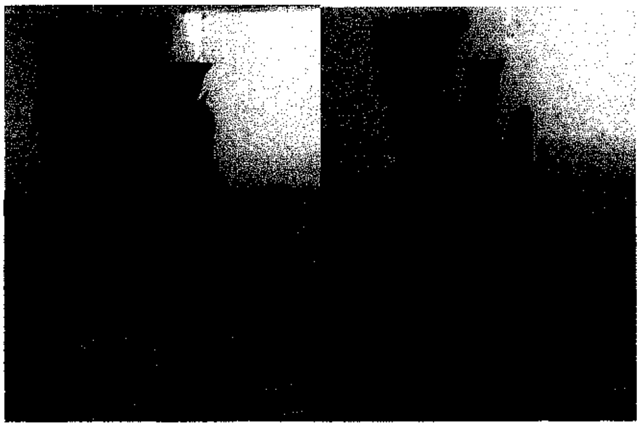


Figure 4.10: Mukah Coal after compression, LHS is Ulu sikat coal, RHS is Ulu Penipah coal

Table 4.11: Comparison of moisture content and compressive strength with standard properties for Lignite and Bituminous Coal

Parameter	**Lignite Coal**		**Bituminous Coal**	
	Ulu Sikat Coal	**Std Lignite Coal**	**Ulu Penipah Coal**	**Std Bituminous Coal**
Moisture Content (%)	13.22%	25-45%	5.18%	2-15%
Compressive Strength (psi)	3276.40	2190-6560	1575.11	310-2490

The moisture content of Ulu Sikat's coal is lower than the existing range of moisture content for Lignite coal, this might because of water loss during transportation. Ulu Penipah's coal moisture content is within the existing moisture content range but it is consider quite a low value in the range. The compressive strength for Ulu Sikat's coal and Ulu Penipah's coal are both within the standard coal range which shows the experiments value is accurate.

4.5 Gas Content of Mukah Coal

The results of GC testing are shown in Figure 4.11 and 4.12. From the Figure 4.11, it is shown that Ulu Sikat's coal having higher composition of carbon dioxide adsorb in the coal matrix while Ulu Penipah's coal having lower carbon dioxide composition as shown in Figure 4.12 which having a lower height for the spectrum peak.

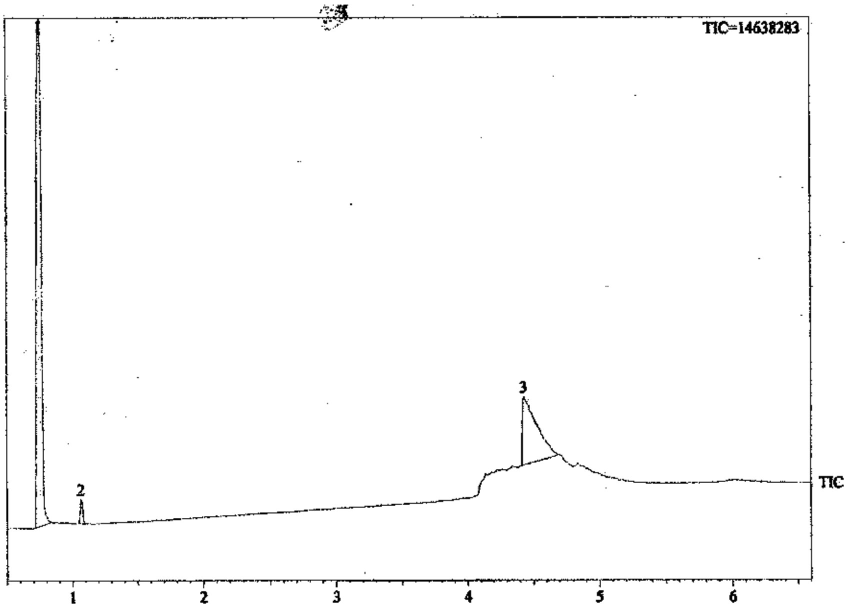


Figure 4.11: Gas Chromatography results for Ulu Sikat coal

Table 4.12: Summary of Gas Content Composition for Ulu Sikat Coal.

Peak No.	Component	Total %
1	Air	70.74
2	CO2	1.64
3	H2O	27.62

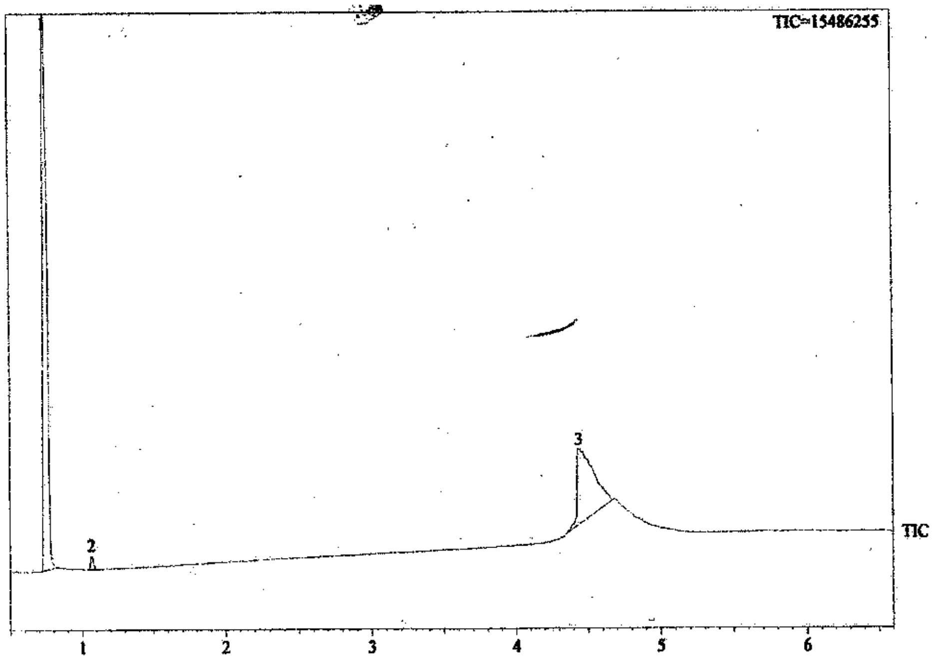


Figure 4.12: Gas Chromatography results for Ulu Penipah coal

Table 4.13: Summary of Gas Content Composition for Ulu Penipah Coal.

Peak No.	Component	Total %
1	Air	65.69
2	CO2	0.95
3	H2O	33.37

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The physical and chemical properties of coal sample are analysed, the properties that have been identified are porosity, permeability, moisture content, composition and compressive strength.

Generally the porosity of Mukah coal is high but the permeability is low because the pore volumes are not connected. The porosity will eventually affects the moisture content and gas content of the coal sample. The higher the porosity of coal, the higher the moisture content of the coal, at the meanwhile the gas content adsorb will be higher. Theoretically the higher rank of coal having higher carbon content which eventually reduce the pores volume of the coal, this has been identified that Bituminous coal (Ulu Penipah coal) having lower porosity than Lignite coal (Ulu Sikat coal).

Ulu Sikat coal is classified as Lignite coal which is not as mature as Ulu Penipah coal which is Bituminous coal. Experimental reading of compressive strength is within the range for the reference standard given and the experimental compressive strength of the Ulu Sikat's coal is higher than Ulu Penipah's coal which proven that the Lignite coal having higher strength than Bituminous coal.

The objectives of the project are achieved which the physical and chemical properties are identified, the gas content and composition of Mukah coal is identified and finally correlate the maturity of coal with the porosity, moisture content, carbon content and the gas content of the coal.

5.2 Recommendation

Further analysis should be conducted to identify potential of Mukah coal as Coalbed Methane (CBM) which included the identify relative permeability of methane gas with respect to oil, water and inert gas, this experiment should be conducted with a coal plug that consist of porosity more than 20% in order to obtain accurate experimental results.

The recover factor of methane gas using oil, water and inert gas will be identified by conducting the experiment with the volume of gas recovered in the experiments. Lastly is conducts an experiment to identify the adsorption and desorption of the Mukah coal.

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APPENDICES

Figure A-1: Map of Mukah Balingian area indicating approximate mining location

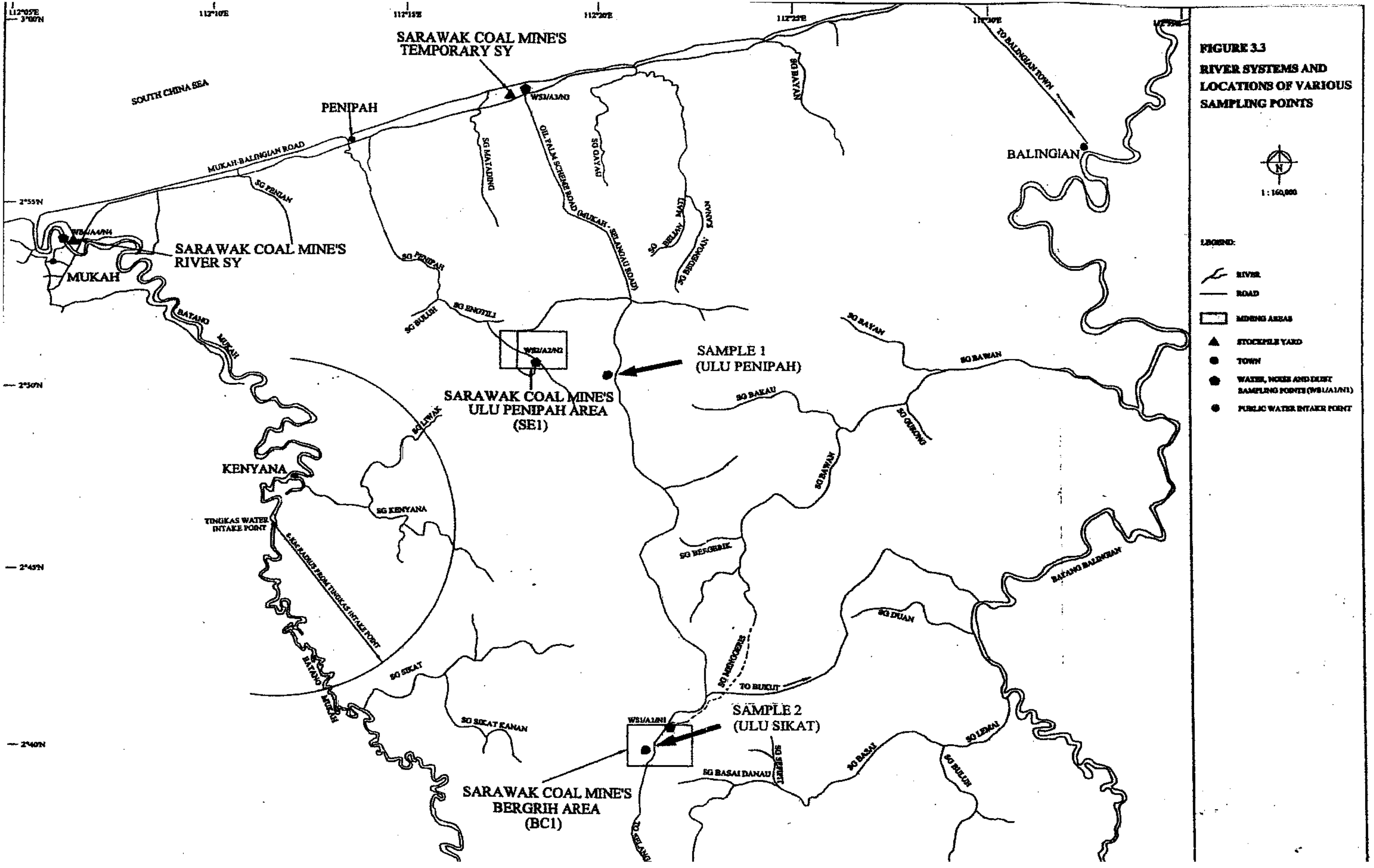
FIGURE 3.3
RIVER SYSTEMS AND LOCATIONS OF VARIOUS SAMPLING POINTS