A Study on the Effect of Acid Injection with Surfactants on Malaysian coals for CBM Study

By

Muhammad Haidir Nizam Bin Baharuddin
12736

A project dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Petroleum Engineering)

MAY 2013

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

A STUDY ON THE EFFECT OF ACID INJECTION WITH SURFACTANTS ON MALAYSIAN COALS FOR CBM STUDY

By

Muhammad Haidir Nizam Bin Baharuddin
12736
Petroleum Engineering

A project dissertation submitted
In partial fulfillment of the requirement for the
Bachelor of Engineering (Hons)
(Petroleum Engineering)

MAY 2013

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

Approved by,

…………………….

(Mr. Saleem Qadir Tunio)
Project Supervisor

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.

Regards,

……………………………
(Muhammad Haidir Nizam Bin Baharuddin, 12736)

ABSTRACT

Acid injection is one of the well stimulation technique that usually been imposed on sandstone, but rarely been implemented for CBM recovery. This research will focus on the study consisting the effect of acid injection with surfactant on the CBM recovery which is also known as Enhanced Coal Bed Methane (ECBM). This research will attempt to resolve the formation damage problem by introducing surfactants that could probably avoid the formation damage, thus will assist acid injection to increase the permeability of the CBM. The surfactant is employed in an aqueous acid solution, preferably having pH equal to or less than 3.0. The method is carried out by injecting the aqueous acid solution containing alcohol and also SLS (Sodium Lauryl Sulfate) that act as surfactant into a coal sample, preferably at injection rates lower than that would fracture for formation or at matrix injection rates. Tests have shown that the acid solution containing surfactant at certain concentration permit stimulation of the formation without damaging the formation itself. The acid solution is believed to act for dissolving calcareous materials in the cleats and the surfactant modifies the wettability of the coal surface which will lead to the improvement of permeability either water or methane or both. Actually, the acid stimulation will improve the formation permeability for the water in the dewatering stage. When sufficient water has been produced from the formation through the interconnected cleats, the resultant reduced pressure allows the methane to desorb from the coalbed formation and flow into and through the interconnected cleats into the wellbore. In this study, ECLIPSE 300 software is used as the initial result on how the surfactant injection will impact the water and methane production from the Coalbed Methane, before proceed to the laboratory tests. Laboratory tests include the utilization of Gas Adsorption Column Unit (GACU) to measure the adsorption capacity, an experiment to measure the porosity and moisture content of the coal and lastly the usage of Mercury Porosimeter equipment to measure the permeability impact of acid and surfactant injection into Malaysia coal sample for CBM study. The permeability for both, water and methane are expected to be increased after injecting acid solution with surfactants of various concentrations.

ACKNOWLEDGEMENT

First and foremost, thank you to Allah the Almighty for the opportunity to live and opportunity to explore His priceless knowledge. My heartiest gratitude goes to my beloved family especially my mother, Zaiton Binti Hussein for being very supportive and always motivate me in finishing this project. I also would like to express my deepest gratitude to my project supervisor, Mr. Saleem Qadir Tunio, for his endlessly support, guidance and invaluable help in ensuring the success of this project. My grateful thanks also go to Dr Iqbal, lecturer from Chemical Engineering Department, UTP, and Mr. Shahafizan, Lab Technician from Chemical Engineering Department, UTP for the valuable technical support and willingness to share their knowledge. My sincere thanks to all my friends whose had been assisting me directly or indirectly throughout this project. Lastly, I also would like to thank you Universiti Teknologi PETRONAS for providing the facilities and also a memorable journey during my study.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

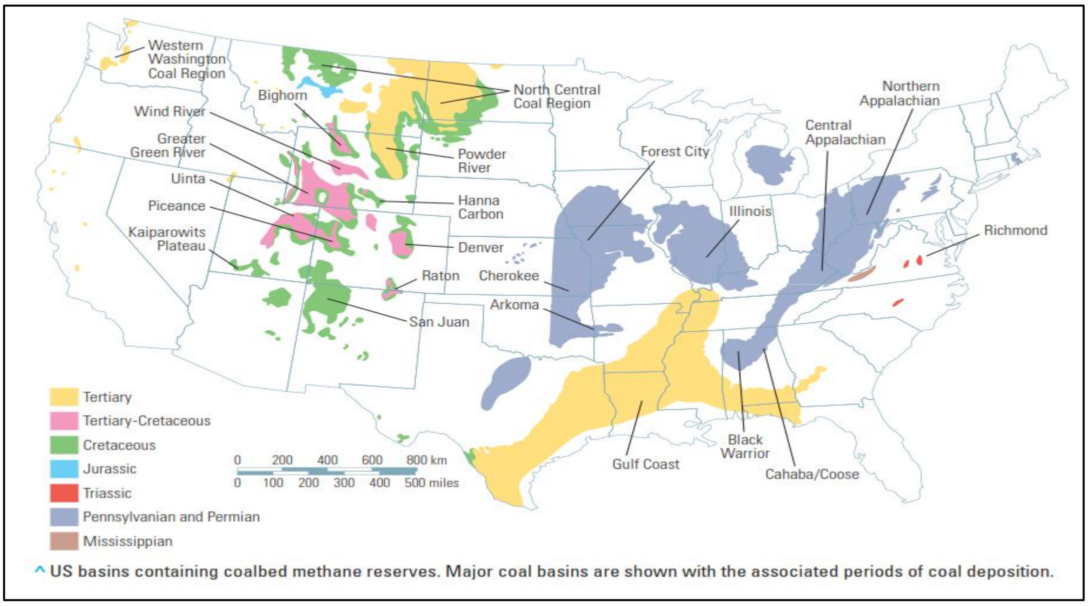
NOMENCLATURE & ABBREVIATION

CBMA	- Coal Bed Methane Additives
GACU	- Gas Adsorption Column Unit
FPR	- Field Pressure Rate
FWPR	- Field Water Pressure Rate
FGPT	- Field Gas Production Total

CHAPTER 1 INTRODUCTION

1.1 Background Study.

In the coming decades, the world will confront two critical energy issues; a need for more electricity and a need for more liquid energy to power combustion engines (Oilfield Review, Schlumberger Magazine, 2003). These increased needs arise from an expected growth in world population and an expanding demand for energy in developing countries. Natural gas plays an important role in meeting those needs, both in generating electricity and in supplying more fuel for automobiles, airplanes, trucks, buses, trains and boats. As production from conventional oil peaks and begin to decline, the world will turn to natural gas and heavy oil to meet the growing demand for liquid fuel. Natural gas supplies are sufficient to meet future demand for the next five decades. According to BP statistics (from website www.bp.com), the world currently has about 5500 trillion cubic feet (Tcf) [156 trillion m^3] of proven gas reserves. Europe and Eurasia together have approximately 40% of the total gas reserves, and the Middle East has 36 %. In 2002, the world consumption of natural gas was about 88 Tcf [2.5 trillion m^3], of which 28 Tcf [0.8 trillion m^3] (31%) was used in North America and 36 Tcf [1.0 trillion m^3] (41 %) was used in Europe and Eurasia (Retrieved from www.bp.com). At the present rate of consumption, known reserves of natural gas will last about 50 years. The oil and gas industry has implemented much less exploring for natural gas than for oil. The industry is just starting to look for natural gas in tight-gas sands, coalbed methane (CBM) and gas hydrates. Focusing more on coal bed methane, there are a lot of improved methods are being introduced for the coal bed methane recovery. To state the current condition of coal bed methane, below are two figures which notate the reserves and also the current trending of producing well.



^ US basins containing coalbed methane reserves. Major coal basins are shown with the associated periods of coal deposition.

Figure 1. Map of US containing coalbed methane reserves. Major coal basins are shown with the associated periods of coal deposition.

Source: From Autumn 2003, Oilfield Review, Schlumberger Magazine, page 10

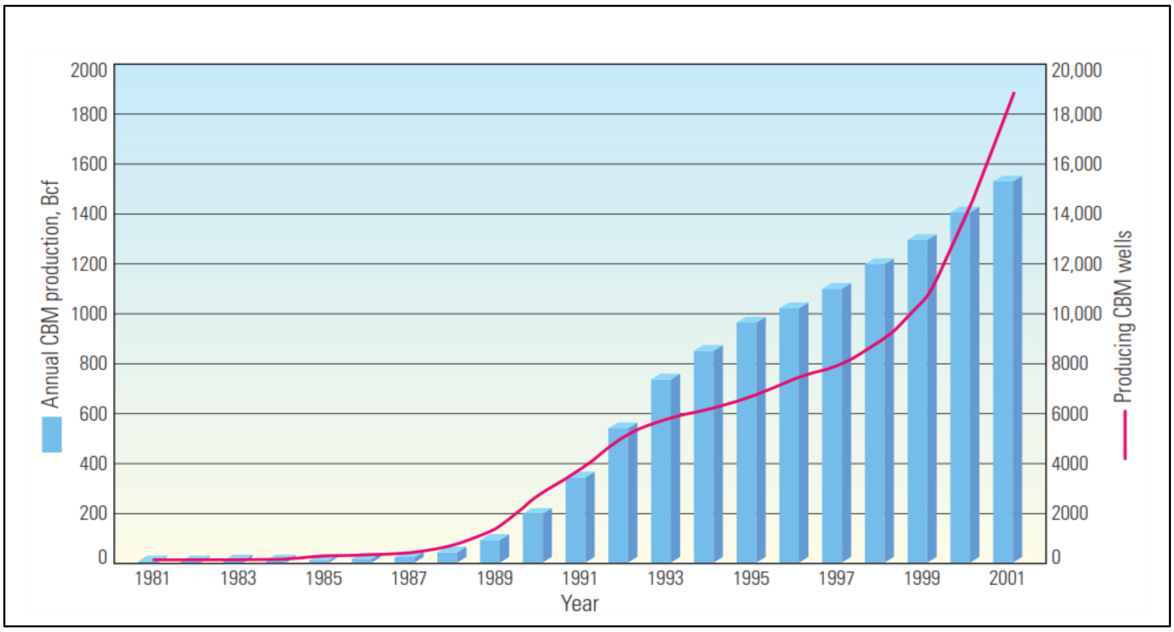


Figure 2. US CBM Production (Blue) and number of producing wells (Red).

Source: From Autumn 2003, Oilfield Review, Schlumberger Magazine, page 9

1.2 Problem Statement.

How to stimulate the coalbed methane formation without damaging the formation?

CBM is one of alternative sources for Natural Gas. It contributes merely 7% to the energy, specifically in U.S.. Most CBM reservoirs initially produce only water because the cleats are filled with water. Typically, water must be produced continuously from coal seams to reduce reservoir pressure and release the gas. The cost to treat and dispose the produced water can be a critical factor in the economics of a coalbed methane project. Once the pressure in the cleat system is lowered by water production to the "critical desorption pressure," gas will desorb from the matrix. Critical desorption pressure, is the pressure on the sorption isotherm that corresponds to the initial gas content. As the desorption process continues, a free methane gas saturation builds up within the cleat system. Once the gas saturation exceeds the critical gas saturation, the desorbed gas will flow along with water through the cleat system to the production well.

The methods used for CBM production vary across basins and one basin from another, depending on the geology and reservoir properties. To select optimal engineering applications to maximize well performance, it is crucial to determine the influence of these geologic parameters on the success of specific drilling, completion, or stimulation practices/methods. The methods used to improve the coalbed methane recovery is knows as Enhanced Coal Bed Methane recovery (ECBM). Acid Injection is one of the mechanisms to stimulate the reservoir to produce natural gas or methane. Recovery of natural gas from coalbed methane is different with the conventional one. In coalbed methane production, the methane is desorbed from the coalbed, whereas in the recovery of hydrocarbons form sandstone and limestone formations, the hydrocarbon is produced by tapping into the formation and reducing the formation pressure which causes the pressurized gas gas or oil to flow into the wellbore. As stated earlier, to desorb methane from the coalbed methane, it needs dewatering the cleats to reduce the pressure to a level which allows the methane to detach from the coalbed and flow into the the cleats and the wellbore. Due to the cleats generally contain calcite or carbonate deposits or coal fines which restrain the flow through the cleats, it is the general practice to treat coalbed formations by acidizing to dissolve these calcareous materials. However, there is issue while practicing this method, because acid treatments frequently resulted in little or no stimulation and sometimes even damages the formation. Thus, to overcome the problem, lately there is an introduction of using amphoteric surfactant to alter the wettability of the coal from strongly oil wet to a neutral or slightly oil wet state. These amphoteric surfactants act as wetting agent to change the wettability. In this project, acetone and methanol are the two surfactants which will be investigated either they can act the role as good surfactant or not in order to overcome the above problem statement.

1.3 Objectives.

- To study the effect of surfactant injection into coal bed formation.
- To study the adsorption rate of methane gas from coal bed methane.
- To study the porosity of the Malaysian sub bituminous coal..
- **MAIN OBJECTIVES**: **To study the acid with surfactant injection into coal bed methane formation in terms of porosity and permeability.**

1.4 Scope of Study.

In this research, the nature of Hydrochloric Acid, acetone, methanol, and Sodium Lauryl Sulfate need to be studied. With their characteristics, their reactivity could be acknowledge and predict their potential to play as the role in Enhanced Coal Bed Methane (ECBM) recovery. Acid injection actually has been investigated before towards the recovery of methane from CBM, but it has stopped due to formation damage caused by acid injection. The information regarding the surfactant need to be obtained to overcome the problem, and directly will improve the permeability of the coal.

ECLIPSE 300 simulation is also one of the tool that need to be utilized in this project to produce the result of surfactant injection into coalbed methane.

CHAPTER 2 LITERATURE REVIEW

2.1 Coalbed Methane (CBM).

Definition of Coalbed Methane.

Coal Bed Methane, is often referred to as CBM. It is the natural gas contained within coal. The gas storage mechanism is unlike what is found in a conventional reservoir. In a typical gas reservoir, gas is compressed by the pressure in the formation (Retrieved from www.fekete.com/software/cbm/media/webhelp/c-te-concepts.htm). Expansion of the gas provides the means for the gas to be produced. In a coal reservoir, the gas is stored within the coal matrix by a process known as adsorption*. Apart from that, gas also stored as free gas. In adsorption, the gas molecules adhere to the surface of the coal. As the reservoir pressure is reduced, gas is released from the coal surface, diffuses through the coal matrix, flows through the fracture system of the coal, and then on to be produced. Gas stored by adsorption can, under certain conditions exceed gas stored by compression. The release of gas is commonly described by a pressure relationship called the Langmuir Isotherm.

Coal Structure.

For the purpose of CBM production a coal reservoir is considered to be a system that consists of fracture and matrix.

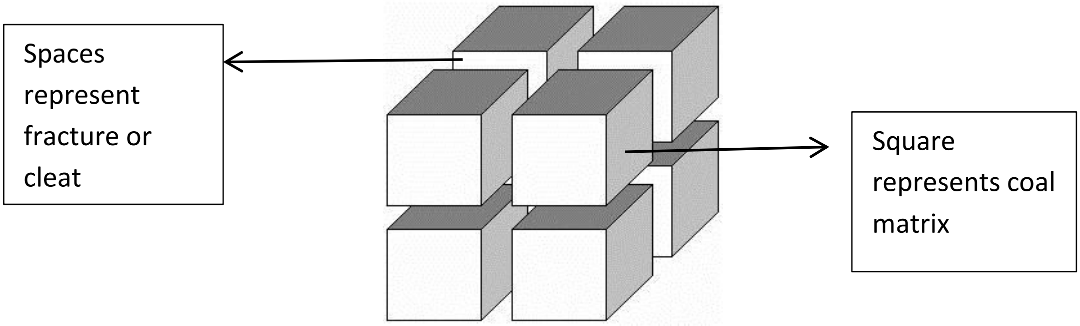


Figure 3. Coal Structure

Source: From website http://www.fekete.com/software/cbm/media/webhelp/c-te-concepts.htm

Coal is naturally fractured, that are collectively known as cleats. There are two main cleat types: face cleats and butt cleats. Face cleats act as the main channels for flow in coal. Butt cleats typically terminate perpendicular to a face cleat.

*Note that this process is a**d**sorption, and not a**b**sorption

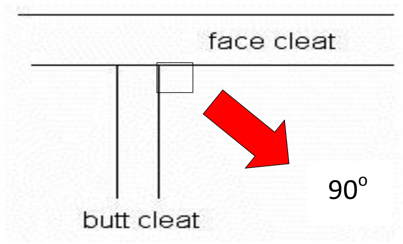


Figure 4. Categorization of cleats in coal

Source: From website http://www.fekete.com/software/cbm/media/webhelp/c-te-concepts.htm

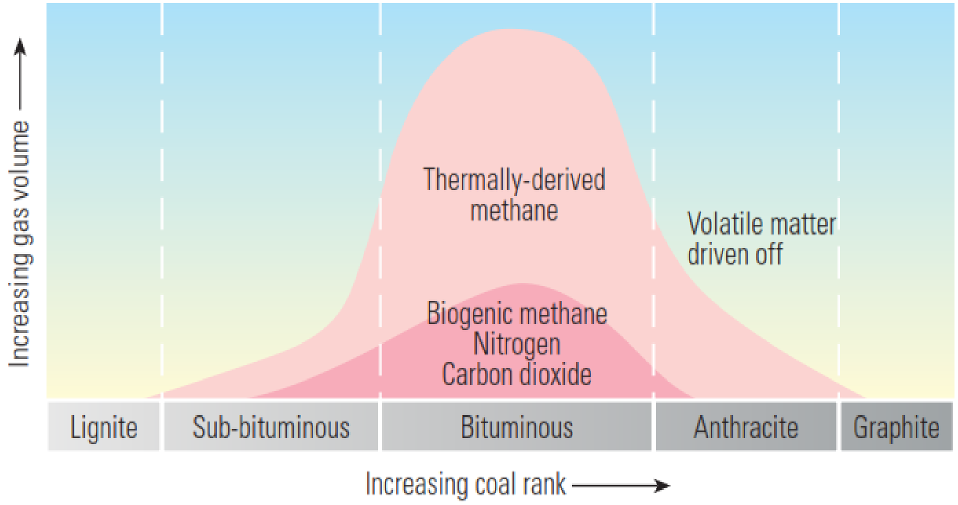


Figure 5. Gas Generation as a function of Coal Rank

Source: From Autumn 2003, Oilfield Review, Schlumberger Magazine, page 12

Figure 5 above shows the gas generation in coal. As can be observed from the graph, as temperature and pressure increase, coal rank changes along with its ability to generate and store methane. Through time, dewatering and devolatization occur, causing shrinkage of the coal matrix and creation of cleats.

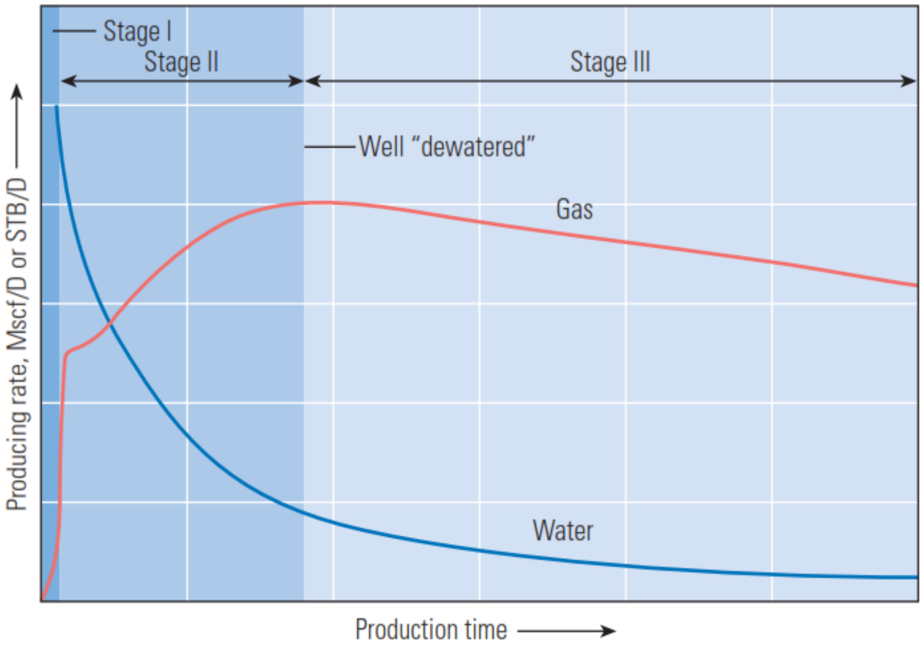


Figure 6. Coalbed Production Characteristics

Referring to the figure above, during stage I, production is dominated by water. Gas production increases during stage II, as water in the coal is produced and the relative permeability to gas increases. During stage III, both water and gas production decline.

2.2 Stimulation of Coalbed Methane Production.

Coal seams contain a system of vertical fractures or known as cleats that are typically water saturated. The gases contained in the coalbeds is adsorbed on the internal surface of the coalbed matrix. The production of methane gas from the formation generally involves producing formation water (in dewatering phase) from the coal seams thereby reducing the reservoir pressure. After reduction of the reservoir pressure, methane gas will desorb and flows through the cleat network to reach the wellbore. Based on the coalbed internal structure, it is different to recover methane from coalbed compared with the recovery of hydrocarbon from sandsrtone or limestone type formations. They are different in terms of lithology and minerology. And, the methane hydrocarbon is deposited in coalbed by adherence to the matrix of the coal or to the surfaces of the cleat, while in the sandstone and limestone formations, the hydrocarbon occupies the interstices of the formation. As stated earlier, to desorb methane, it requires dewatering the cleats to reduce the pressure to a certain limit which will allow the methane to detach from the coalbed and flow into the cleats and the wellbore. There is a study shows that cleats generally contain calcite or carbonate deposits or coal fines which restrict the flow through the cleats, it is the general practice to dissolve those calcareous materials by matrix acidizing. Efforts to improve the dewatering stage in methane production have involved stimulation treatment using surfactants in the well treating fluid. For instance, SPE Paper 23455 describes the tendency of coalbed reservoirs to become damaged as a result of stimulation or workover treatments with certain surfactants. The paper suggests the use of an additive identified as CBMA of Dowell.

2.3 Design and Evaluation of Stimulation and Workover Treatments in Coal Seam Reservoirs.- SPE 23455

This paper unveil the results of field trials conducted primarily to evaluate the efficacy of a new additives developed for use in coal seams. This paper also presents results from laboratory tests which show the new additive may also be useful in workover treatments. Coal seam shows some special characteristics which include desorption controlled production and the tendency of coal reservoirs to produce fines and undergo wettability changes. Laboratory and fields tests have proved that the new products and designs for use in coal seam reservoirs. The field tests which have been implemented in the San Juan Basin, Black Warrior and Appalachian Basins shows that there was a remarkable improvements in dewatering rates and methane production with fewer problems associated with coal fines. To be specific, this SPE 23455 paper describes the results obtained during field trials using a new surfactant system which will be referred as CBMA additive. The additive has potential of improving the coal's permeability to water by lowering the surface tension of the water, maintaining or conditioning the coal surface so that it has a low ionic surface charge and is preferentially oil wet and lastly, minimizing dispersion and migration of the fines which can bridge cracks or pores and lower fracture conductivity. From this paper, it concludes several points which are; first, the design and execution of stimulation and workover treatments in coal seam reservoirs must consider the reactivity of the coal to produce a significant production improvement result,secondly is the proper use of additives can lead to remarkable enhancement in methane and water production in coalbed methane. Improve the dewatering generally lead to improvement in the methane production. The next point is effective workover treatment must be based on accurate problem diagnosis. They must reflect the tendency of many coals to undergo adverse reactions when exposed to commonly-used oilfiled materials.

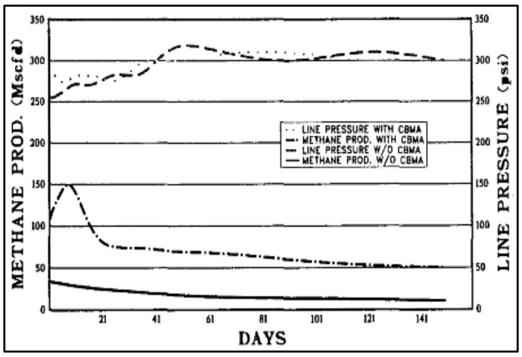


Figure 7 Methane production from a well-treated with CBMA surfactant vs untreated- San Juan Basin

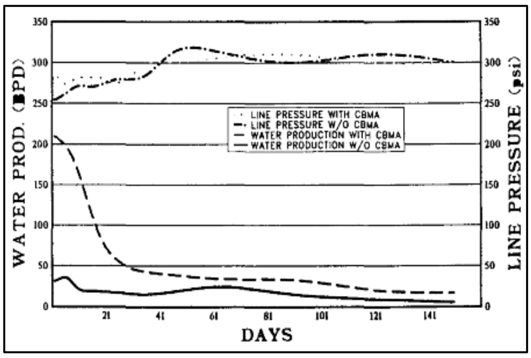


Figure 8 Water production from a well-treated with CBMA surfactant vs untreated- San Juan Basin

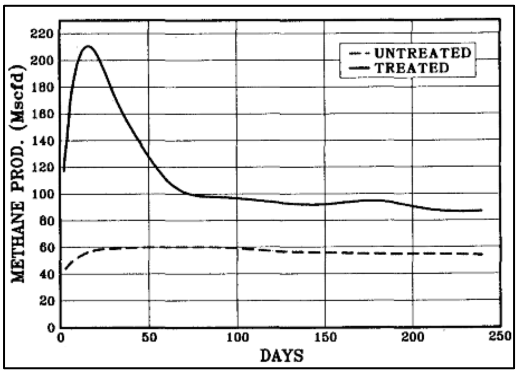


Figure 9 Methane production from a well-treated with CBMA surfactant vs untreated well- Appalachian Ba

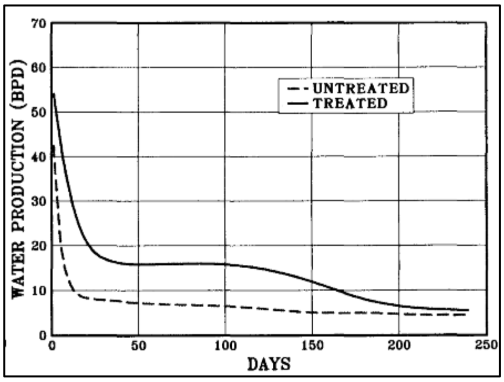


Figure 10 Water production from a well-treated with CBMA surfactant vs untreated well- Appalachian Basin

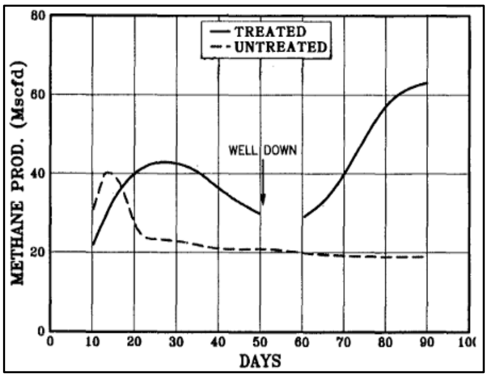


Figure 11 Methane production from a well-treated with CBMA surfactant vs untreated well- Black Warrior Basin

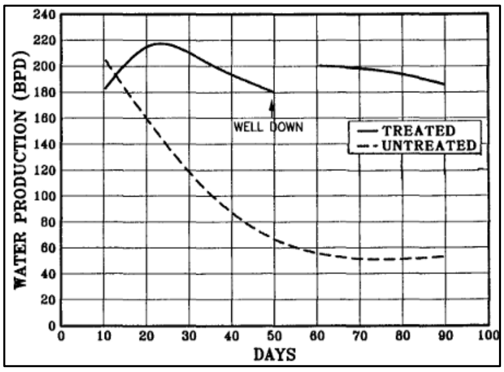


Figure 12 Water production from a well-treated with CBMA surfactant vs untreated well- Black Warrior Basin

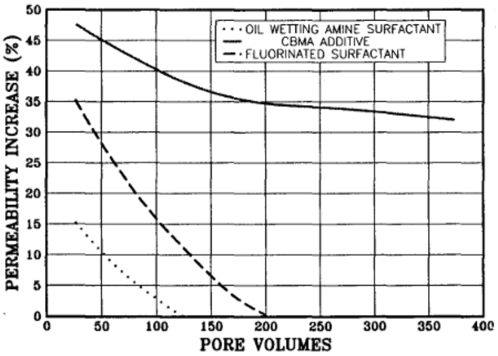
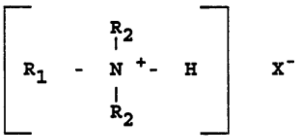


Figure 13 A comparison of the CBMA additive with "conventional" surfactants; baseline permeability = 3.0 darcies

2.4 Usage of Amphoteric Surfactants in Acid Injection.

The new development of matrix acidizing involves the treatment of a methane containing coalbed formation with an aqueous treating liquid containing an amphoteric surfactant. The surfactant is an organic amphoteric tertiary ammonium compound (e.g. salt) having from 1 to 3 tertiary ammonium group, wherein each group is bonded to at least one C_3 to C_4 unsaturated carboxylic acid group and wherein the compound has a terminal C -Cg hydrocarbon group when the terminal group is bonded directly to N of the tertiary ammonium group and a terminal Cg to C-^g alkyl hydrocarbon group when connected to a tertiary ammonium group through an imidazoline linkage. The preferred amphoteric surfactants have general formulas I and II as shown below:



$$\left[R_1 - \overset{R_2}{\underset{R_2}{N^+}} - H \right] X^-$$

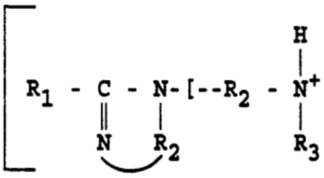
(I)

where:

R-l is a benzyl or a C_4 to Cg alkyl group;

R_2 is independently a propanoic group (C_3 H_4OOH) or a 2-methyl propanoic group (2-CH_3- C_3H_4OOH) ;

and X" is independently $Cl^{''}$, $HCOO^{''}$, NH_2S0_3 $^{''}$, or CH_3COO



where:

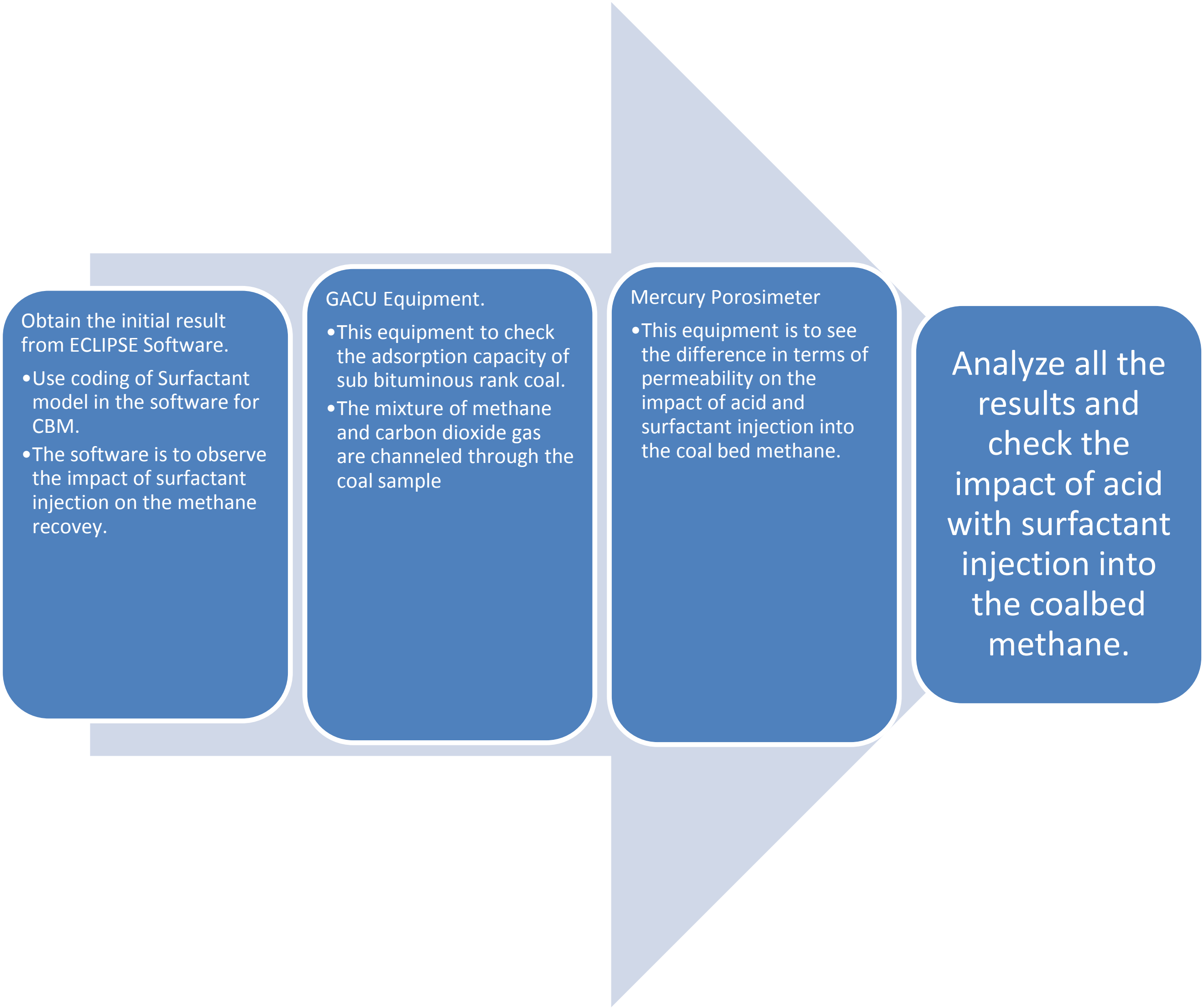
n is an integer from 1 to 3; R-$_{j\wedge}$ is an alkyl Cg to C^g hydrocarbon;

R_2 is a C_2 H_4 or C H_g group;

R_3 is independently a propanoic group (CH_3H_4OOH) or a 2-methyl propanoic group (2- $CH_3C_3H_4OOH$) ; $X^{"}$ is as described above for Formula I.

The amphoteric surfactant is applied in an acid solution, which have a pH equal to or less than 3.0, usually Hydrochloric or Formic Acid. The method in carried out by injecting the acid solution containing amphoteric surfactant into a coalbed formation, with the injection pressure lower than the fracture pressure or at matrix injection rates. Tests have shown that the acid solution containing the amphoteric surfactant allows stimulation of the formation without damaging the formation. The role of acid here is to dissolve the calcareous material in the cleats and the amphoteric surfactant alters the wettability of the coal surface resulting in improved permeability to water and/or methane. Laboratory tests have proved that the treatments in accordance with the current development do in fact stimulate dewatering and methane production.

CHAPTER 3 METHODOLOGY



Obtain the initial result from ECLIPSE Software.

- Use coding of Surfactant model in the software for CBM.
- The software is to observe the impact of surfactant injection on the methane recovey.

GACU Equipment.

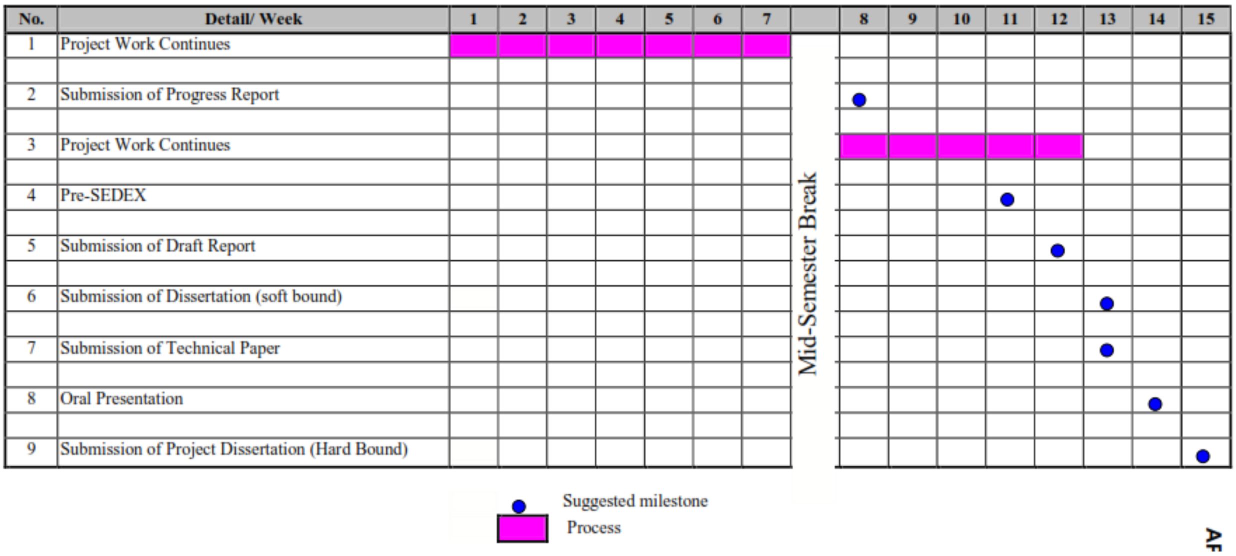
- This equipment to check the adsorption capacity of sub bituminous rank coal.
- The mixture of methane and carbon dioxide gas are channeled through the coal sample

Mercury Porosimeter

- This equipment is to see the difference in terms of permeability on the impact of acid and surfactant injection into the coal bed methane.

Analyze all the results and check the impact of acid with surfactant injection into the coalbed methane.

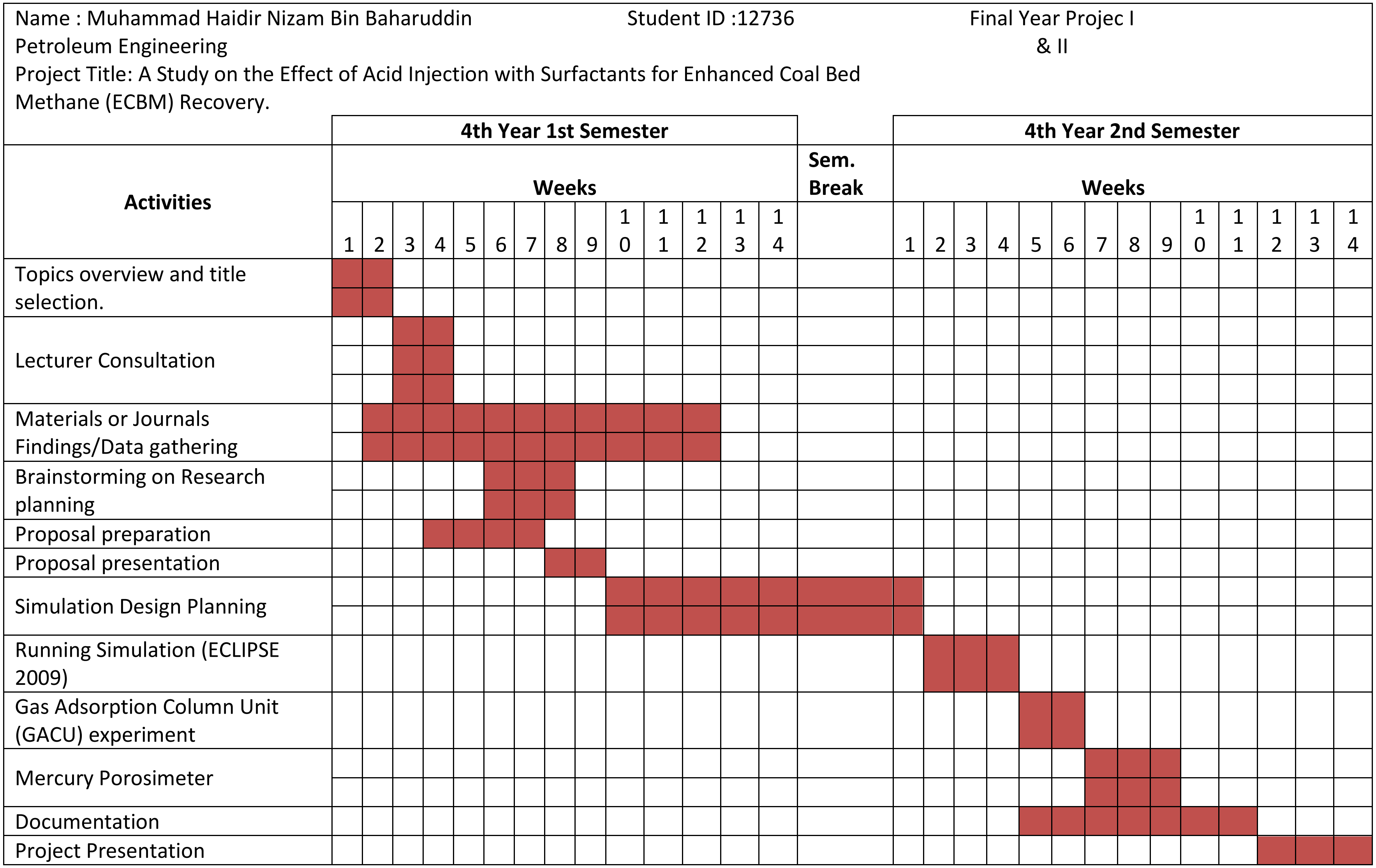
Figure 14 Key Milestone for FYP (FYP I to FYP II)



No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Project Work Continues	🟪	🟪	🟪	🟪	🟪	🟪	🟪	Mid-Semester Break								
2	Submission of Progress Report									●							
3	Project Work Continues									🟪	🟪	🟪	🟪	🟪			
4	Pre-SEDEX												●				
5	Submission of Draft Report													●			
6	Submission of Dissertation (soft bound)														●		
7	Submission of Technical Paper														●		
8	Oral Presentation															●	
9	Submission of Project Dissertation (Hard Bound)																●

● Suggested milestone
🟪 Process

Figure 15 Gantt Chart for FYP 2



Name : Muhammad Haidir Nizam Bin Baharuddin Student ID :12736 Final Year Projec I & II
Petroleum Engineering
Project Title: A Study on the Effect of Acid Injection with Surfactants for Enhanced Coal Bed Methane (ECBM) Recovery.

Activities	4th Year 1st Semester														Sem. Break	4th Year 2nd Semester													
	Weeks															Weeks													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14		1	2	3	4	5	6	7	8	9	10	11	12	13	14
Topics overview and title selection.	🟥	🟥																											
Lecturer Consultation			🟥	🟥																									
Materials or Journals Findings/Data gathering		🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥																	
Brainstorming on Research planning						🟥	🟥	🟥																					
Proposal preparation				🟥	🟥	🟥	🟥																						
Proposal presentation								🟥	🟥																				
Simulation Design Planning										🟥	🟥	🟥	🟥	🟥	🟥	🟥													
Running Simulation (ECLIPSE 2009)																	🟥	🟥	🟥										
Gas Adsorption Column Unit (GACU) experiment																				🟥	🟥								
Mercury Porosimeter																						🟥	🟥	🟥					
Documentation																				🟥	🟥	🟥	🟥	🟥	🟥	🟥			
Project Presentation																											🟥	🟥	🟥

Figure 16 Gantt Chart of Overall Process of FYP

3.1 Correlation between Methods and Objectives

Table 1. Objectives and Methodology

Objectives	Methodology
To study the impact of surfactant injection into coal bed formation	ECLIPSE 100 Software
To study the adsorption and desorption rate of methane gas from coal bed methane	Gas Adsorption Column Unit equipment
To study the porosity of the sub bituminous coal with equation, with different in additives (Acid and surfactants)	Porosity measurement experiment
To study the acid with surfactant injection into coal bed methane formation in terms of porosity and permeability	Mercury Porosimeter equipment

3.2 Apparatus.

1. Hammer.
2. Crucible and Lid
3. GACU Machine (Gas Adsorption Control Unit).
4. Mercury Porosimetry.
5. Weighing Scale.
6. Syringe.
7. Oven.
8. Crucible tongs.
9. Beakers.

3.3 Materials.

1. Coal Sample (Sub Bituminous Rank)
2. Hydrochloric Acid Solution.
3. Surfactant:
    a. Methanol
    b. Acetone
    c. Sodium Lauryl Sulfate (SLS)

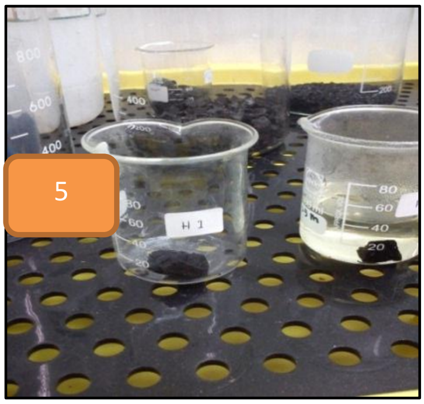
3.4 Samples Collection and Preparation

Coal sample are taken from Ulu Sikat field, Sarawak

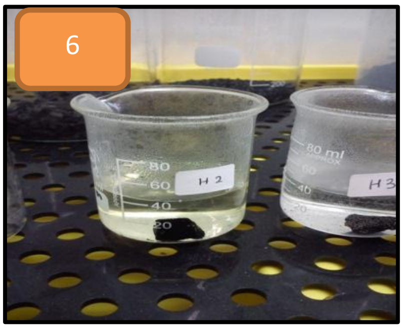
Sample is crashed into finer sizes using hammer

Fined size coal sample

After the crashed coal being sorted into 5 samples, their weight are measured and recorded, before put in oven to heat at 100°C



After heating, every sample is weighed and records their weight. Figure above is sample 1. The description of each sample is tabulated in Table 1.



Sample 2

Sample 3

Sample 4

Sample 5

Every sample is dipped in their respective solution (Refer to Table 2) in 6 hours. After being left 6 hours, they are taken out from the beaker, wipe with filter paper and then record their weights. From this, Porosity of every sample can be calculated, which will discuss further in the next section.

3.5 Experimental Procedure/ Project Activity.

3.5.1 GACU Procedure (Gas Adsorption Capacity).

Step 1: Prepare the coal samples.

Step 2: Prepare Hydrochloric Acid solution.

Step 3: Prepare Surfactants:

1. Methanol
2. Acetone
3. Sodium Lauryl Sulfate (SLS)

Step 4: Mix the surfactant with the aqueous acid solution for sample 3,4,5,6,7 and 8. Prepare two samples for each type of surfactant with different volume mixed with acid solution.

Table 2. Samples Preparation

Surfactant	Sample No.	Volume of Surfactant (ml)	Volume of 37% Hydrochloric Acid (ml)
-	1	-	-
-	2	-	40
Methanol	3	10	30
	4	20	20
Acetone	5	10	30
	6	20	20
Sodium Lauryl Sulfate (SLS)	7	10	30
	8	20	20

Step 5 : Weight the coal sample (Without injecting acid).

Step 6 : Insert the coal sample into GACU machine (Gas Adsorption Column Unit). Channel the mixture of methane and carbon dioxide gases through the sample. Observe the compositional percentage of both gases after go through the sample at the Gas Chromatograph via the computer interface.

Step 7 : Remove the sample from the GACU machine.

Step 8 : Repeat step 5 to 7 for sample 2 until sample 8.

Step 9 : Record and tabulate the measurement data.

Table 3. Example of Table Result (GACU Experiment)

Sample No.	Time Period (min)	Methane, CH_4 (%)	Carbon Dioxide, CO_2 (%)	Impurities (%)	Outlet Flow rate (L/min)

Step 10: Analyze the result data.

3.5.2 Experiment (Porosity and Moisture Content measurement)

Step 1 : Prepare small size of coal sample.

Step 2 : Take eight small size of coal sample and weighing them.

Step 3 : Heat all the eight samples in the oven at the temperature of 100°C for two hours.

Step 4 : After two hours, take out the samples and directly weighing the weight to obtain the accurate dry weight.

Step 5 : Prepare eight beaker with different solution (Refer to Table 1).

Step 6 : Dip all the samples into their respective solution (Refer to Table 1) for 6 hours.

Step 7 : After 6 hours, take out all the samples from the beaker using tong and wipe it with filter paper.

Step 8 : Weigh the sample and record the value. Tabulate all the measurement data.

Table 4. Example of Table Result (Porosity Measurement Experiment)

Sample No.	Weight before heating at 100°C (g)	Dry Weight/ Weight after heating at 100°C(g)	Volume of HCL (ml)	Type of Surfactant	Volume of Surfactant (ml)	Wet Weight (g)

Step 9 : Analyze the result.

3.5.3 Mercury Porosimetry Procedure. (Permeability measurement).

Step 1 : Take all the samples from the previous experiment.

Step 2 : Dry them for one day.

Step 3 : Take sample 1 and put in the equipment.

Step 4 : Handle the flow of mercury into the sample with care as it is dangerous.

Step 5 : After about 3 hours in the equipment, the sample is taken out and the result is obtained from the computer interface.

Step 6 : Repeat step 3 to step 5 for the other samples.

Step 7 : Record and tabulate all the measurement data.

Step 8 : Analyze the result from the equipment.

CHAPTER 4 RESULTS AND ANALYSIS

(A) Simulation Results:

See Appendix 1.

Using Eclipse 100 (2009 version), the result of surfactant injection into the well is obtained. This result is used as the initial result to observe either it notates the difference between surfactant injection into the wellbore and without the injection. There are three graphs that show the performance of the production. Based on graph (a), the **Field Pressure Rate** is decreasing as the **Time/Days** increase, but to compare between the two cases, the surfactant injection shows the higher **Field Pressure Rate**, but until for certain period, 84 days, compared with the well with no surfactant injection. After the 84th days, it shows that without surfactant injection, it increases back the **Field Pressure Rate** which is higher compared with the surfactant injection. For graph (b), the **Field Water Pressure Rate** is declining as the **Time/ Days** increase, for both cases. It shows no big difference between the two cases, although at the initial stage, it observes that without surfactant injection, it is bit higher compared with surfactant injection, before the two curves meet at around 86th days. For graph (c), as the **Time/ Day** increases, the **Field Gas Production Total** also increase but it shows that with surfactant injection, it is remarkably higher compared with no surfactant injection. Hence, as a conclusion from the simulation, it indeed shows the impact of surfactant injection towards the recovery of methane, but to make it more efficient, it needs to be study further on applying it with other well stimulation techniques, for example Hydraulic Fracturing and Matrix Acidising. For this paper, it focuses on the Matrix Acidising and to improvise the present invention, surfactant will be introduced into the Acid Injection.

(B) Methane Gas Adsorption (Gas Adsorption Column Unit):

See Appendix 2 for GACU figure.

For GACU experiment, its purpose is to measure the adsorption capacity of the coal. Generally, it is known that for methane to be recovered, it must be detached from the coal matrix surface, desorb and flow through the cleats before flowing into the well. Thus, to improve the recovery, the coal must be easily desorbing the methane, but to desorb, it must have something to displace it. So, if the coal adsorbs more, it is assumed that it will also desorb more methane gas then in sequence, it will improve the recovery. Hence, the higher the adsorption capacity, the higher the recovery. In GACU experiment, coal will act as adsorbate and mixture of methane and carbon dioxide gases will act as adsorbant. To indicate the adsorption capacity, the mixture of gases is allowed to flow though the coal sample in the column to permit the adsorption to occur. Then, the composition percentage of carbon dioxide and methane will be measured by the Gas Chromatograph. Based on the result, it shows that at certain period, the composition percentage and outlet flowrate are constant. It actually display that at that particular time, the coal sample has become saturated with the gases. Thus, it actually results in the adsorption capacity of the coal.

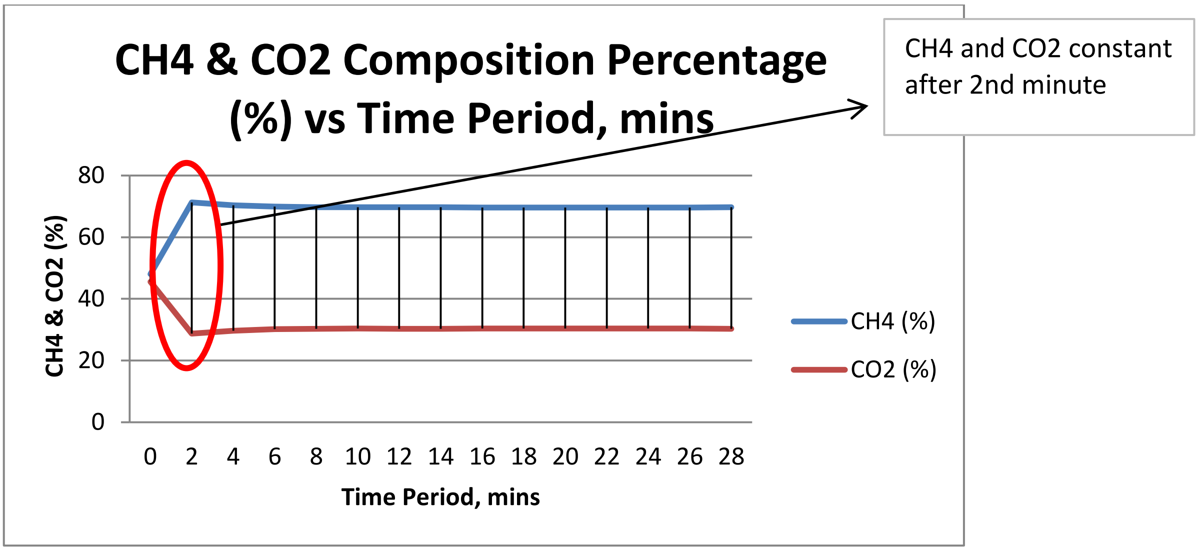
Percentage of CO_2 and CH_4 start to stagnant

Total outlet flow rate start to become constant

#First Trial (Lab Coal/ Commercialised coal)

Table 5. Result of Commercialised coal without acid and surfactant

Time Period (minutes)	Methane, CH_4 (%)	Carbon Dioxide, CO_2 (%)	Impurities (%)	Outlet Flow Rate (L/min)
0	48.0541	45.5590	6.3869	2.00
2	71.2772	28.7228	0	2.30
4	70.3339	29.6661	0	2.40
6	69.8675	30.1325	0	2.60
8	69.6983	30.3017	0	2.60
10	69.6577	30.3423	0	2.61
12	69.6701	30.3299	0	2.61
14	69.6626	30.3374	0	2.61
16	69.6533	30.3467	0	2.60
18	69.6349	30.3651	0	2.60
20	69.5875	30.4125	0	2.60
22	69.5925	30.4075	0	2.60
24	69.5754	30.4246	0	2.60
26	69.6430	30.3570	0	2.60
28	69.6825	30.3174	0	2.61



CH4 and CO2 constant after 2nd minute

Figure 17. Graphical Result of Commercialised coal without acid and surfactant

- In Trial 1, the coal from lab is used to observe the trending of the composition percentage of methane and carbon dioxide gas.

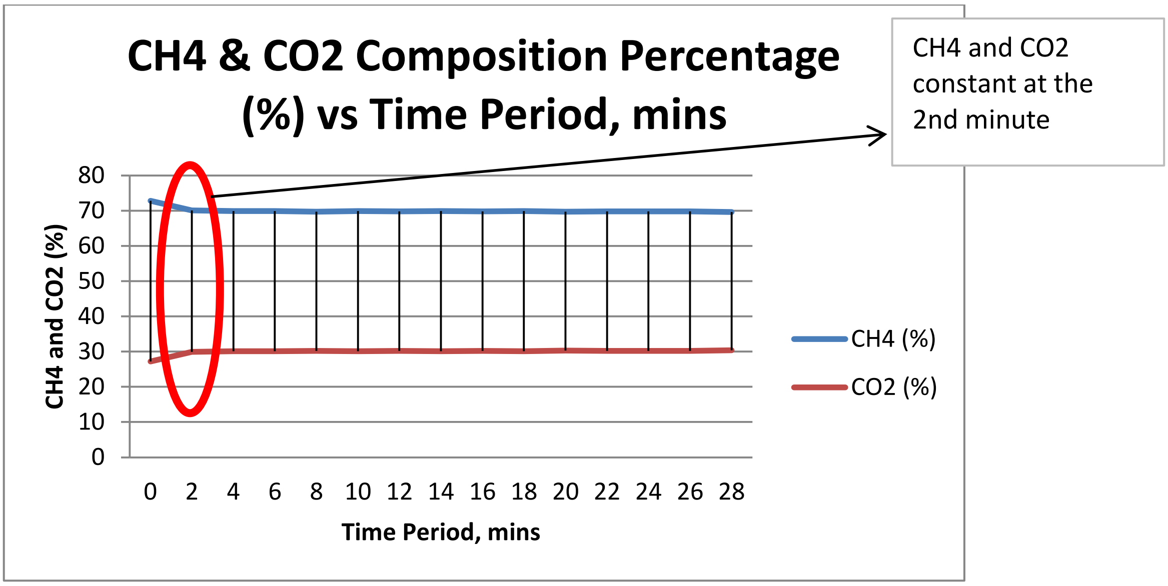
Percentage of CO_2 and CH_4 start to constant

Total outlet flow rate start to become constant

#Second Trial (Lab Coal/ Commercialised coal with Acid)

Table 6. Result of Commercialised coal with acid

Time Period (minutes)	Methane, CH_4 (%)	Carbon Dioxide, CO_2 (%)	Impurities (%)	Outlet Flow Rate (L/min)
0	72.7724	27.2276	0	2.37
2	70.0929	29.9071	0	2.65
4	69.8969	30.1031	0	2.66
6	69.9176	30.0824	0	2.66
8	69.7572	30.2428	0	2.66
10	69.8960	30.1040	0	2.66
12	69.7739	30.2261	0	2.66
14	69.8838	30.1152	0	2.66
16	69.7840	30.2160	0	2.66
18	69.8822	30.1178	0	2.66
20	69.7417	30.2583	0	2.66
22	69.7763	30.2237	0	2.66
24	69.7934	30.2066	0	2.66
26	69.8071	30.1929	0	2.66
28	69.6010	30.3990	0	2.66



CH4 and CO2 constant at the 2nd minute

Figure 18. Graphical Result of Commercialised coal with acid

- In Trial 2, the coal from the lab is injected with the acid, and let for dry for about one day to see the difference impact between acid injection and without acid injection. These two trials are crucial because it will show how the equipment respond, and how the result will support further on this paper as this paper will try to observe the impact of surfactant injection with acid towards the recovery of CBM.

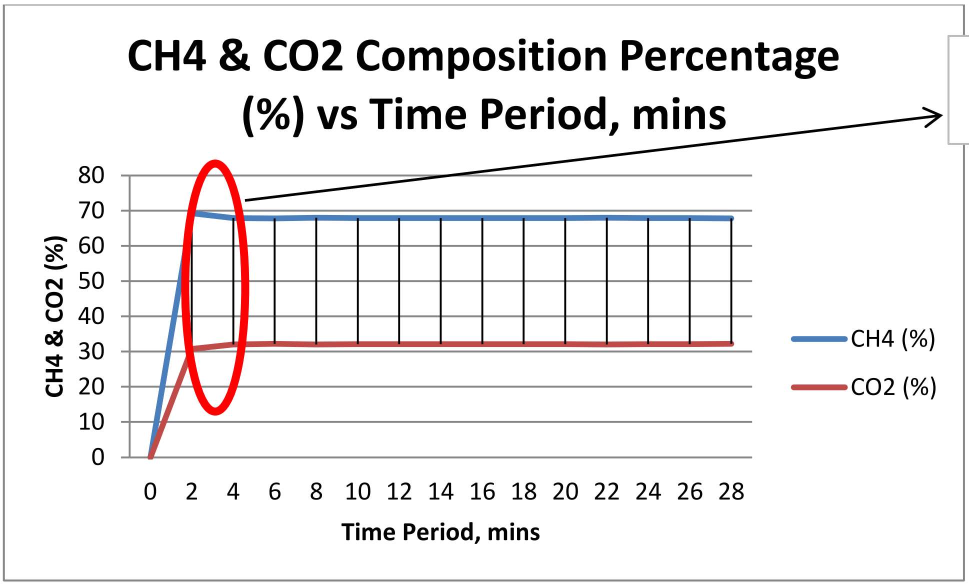
Percentage of CO_2 and CH_4 start to constant

Total outlet flow rate start to become constant

Original Mined Coal Sample from Ulu Sikat Field, Sarawak

Table 7. Result of mined coal sample (Sub Bituminous Rank)

Time Period (minutes)	Methane, CH_4 (%)	Carbon Dioxide, CO_2 (%)	Impurities (%)	Outlet Flow Rate (L/min)
0	0	0	0	0.90
2	68.2390	31.7610	0	1.49
4	67.9408	32.0592	0	2.09
6	67.8287	32.1713	0	2.09
8	67.9703	32.0297	0	2.09
10	67.8738	32.1252	0	2.09
12	67.9323	32.0677	0	2.09
14	67.9010	32.0990	0	2.08
16	67.8999	32.1001	0	2.08
18	67.9330	32.0670	0	2.08
20	67.8700	32.1300	0	2.09
22	67.9444	32.0556	0	2.08
24	67.8969	32.1031	0	2.09
26	67.9123	32.0877	0	2.09
28	67.8355	32.1645	0	2.09



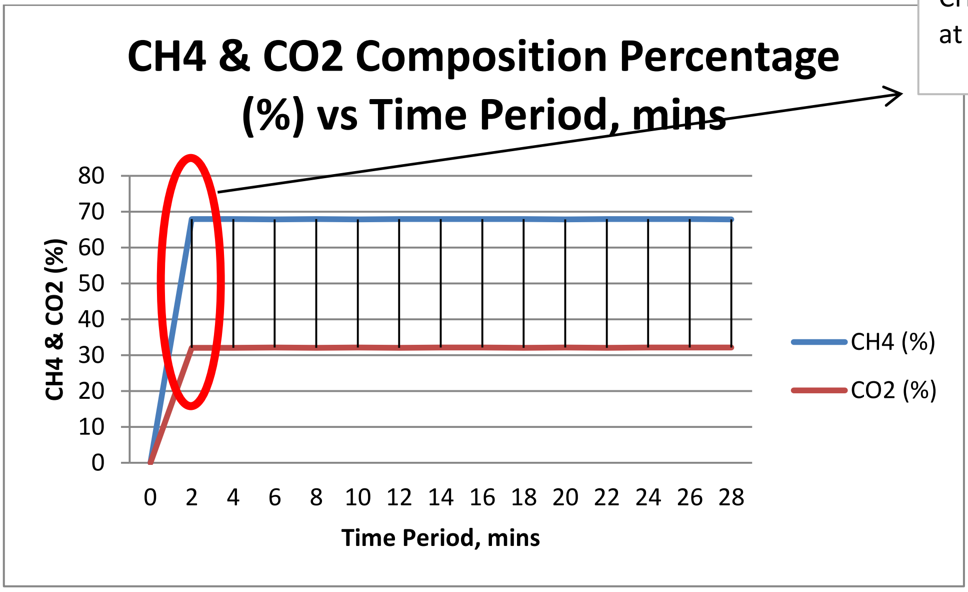
CH4 and CO2 constant after the 2nd minute

Figure 19. Graphical Result of Mined Coal Sample without acid and surfactant

- In the next test, the coal from Ulu Sikat field, Sarawak is used to check its adsorption capacity and to use it as the real sample for the experiment. By measuring the adsorption capacity of the coal, the potential of the Malaysian coal could be predicted to produce methane as the natural gas source.

In Trial 1, it shows the longer period for the coal to become saturated compared with the Trial 2. It indicates that acid interrupt the adsorption capacity of the coal. To confirm it further, the result for the next test, which use the coal from Sarawak is analysed. The result displays the same period for the coal to become saturated with the Trial 2. To investigate it further, the coal form Sarawak need to be injected with acid, but unfortunately, the equipment is malfunction as the thermocouple rod could not resist with acid and thus corroded.

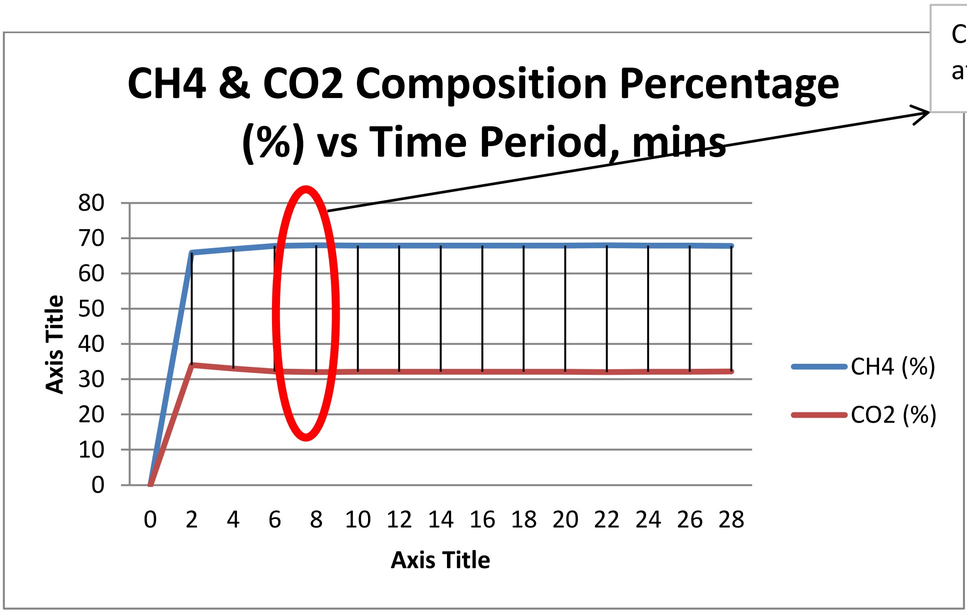
<u>Predicted Trend for mined coal sample with acid.</u>



CH4 and CO2 constant at the 2nd minute

Figure 20. Predicted Trend for mined coal with acid

Predicted Trend for Mined Coal sample with acid and surfactant.



CH4 and CO2 constant after the 2nd minute

Figure 21. Predicted Trend for mined coal with acid and surfactant

(C) Porosity Calculations:

Table 8. Result of Porosity Measurement Experiment

Sample	Weight before heating at 100°C (g)	Dry Weight/ Weight after heating at 100°C(g)	Volume of HCL (ml)	Type of Surfactant	Volume of Surfactant (ml)	Wet Weight (g)	Porosity (%)
1	2.36	1.95	-	-	-	1.99	2.109
2	1.22	0.98	40	-	-	1.07	8.798
3	1.22	0.98	30	Methanol	10	1.05	6.979
4	1.17	0.93	20	Methanol	20	1.00	7.327
5	1.20	0.96	30	Acetone	10	1.02	6.161
6	1.15	0.90	20	Acetone	20	0.96	3.211
7	1.19	0.95	20	Sodium Lauryl Sulfate (SLS)	20	1.02	7.184
8	1.24	1.00	20	Sodium Lauryl Sulfate (SLS)	20	1.10	9.506

$$Porosity\ (\%) = \frac{\rho_{M_{dry}}\left(M_{wet} - M_{dry}\right)}{\rho_{M_{dry}}\left(M_{wet} - M_{dry}\right) + \rho_w \times M_{dry}} \times 100$$

Where;

$M_{dry} - Dry\ weight\ (g) of\ coal$

$M_{wet} - Wet\ weight\ (g) of\ coal$

$\rho_{M_{dry}} - Dry\ density\ of\ the\ coal\ (\frac{gm}{cm^{-3}})$ = 1.25

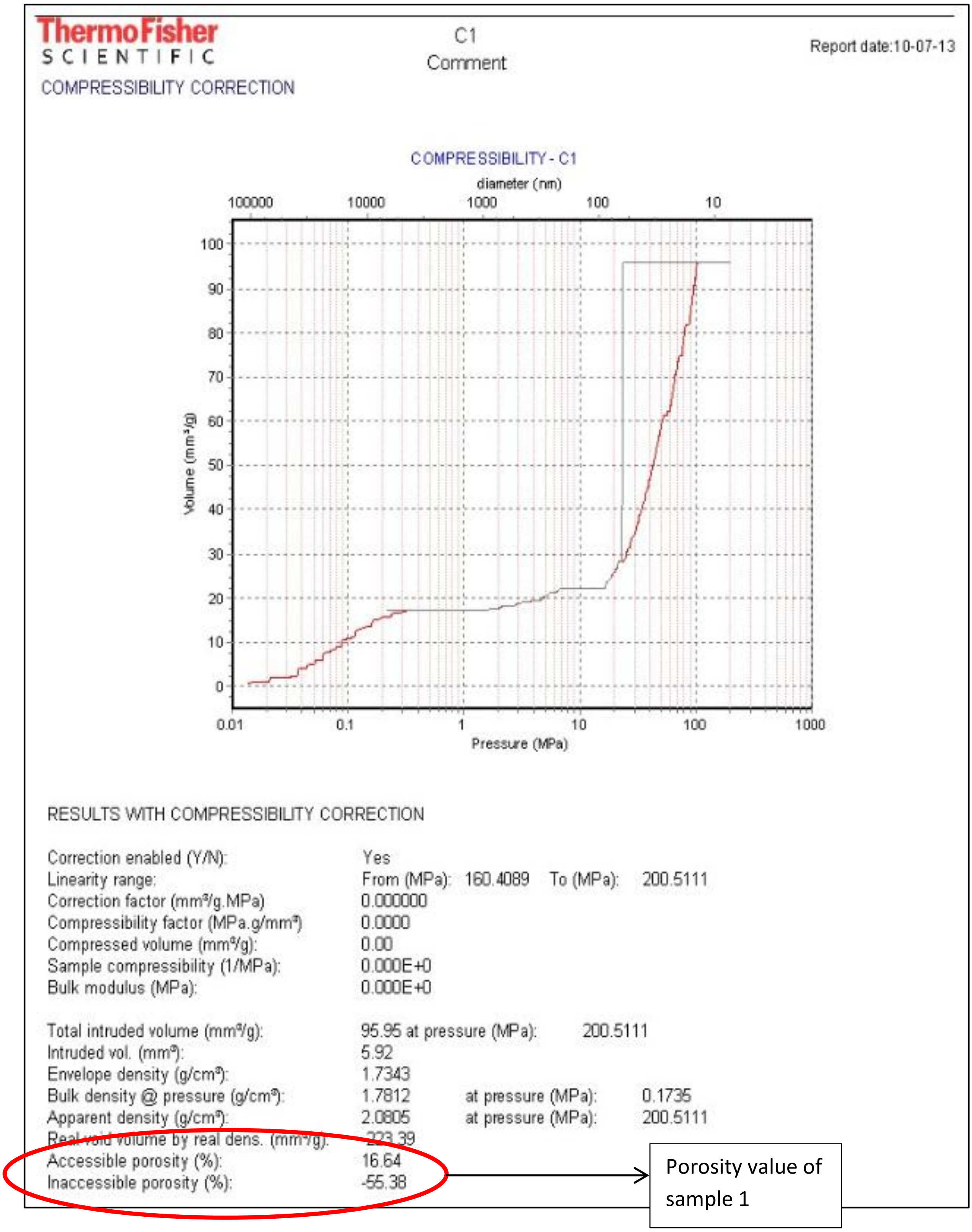
$\rho_{M_{wet}} - Wet\ Density\ of\ the\ coal\ (\frac{gm}{cm^{-3}})$

$\rho_w \quad - \ Density\ of\ wetting\ agent\ (acid)$ = 1.19

For Porosity calculation, the experiment is conducted to get the parameters which are dry weight and wet weight. These data are required in the Porosity equation, refer to previous section. From the result, it shows that acid injection with surfactant alter the porosity of the coal. But, as the volume of surfactant increases, the porosity decreases. It represents that the surfactant concentration does not affect the porosity changes, maybe due to the suitability of the surfactant on the acid. The result trends are same for moisture measurement and wettability measurement. To investigate more on the effect of acid injection with surfactant towards the recovery of methane, other equipment will be used to measure the permeability of the coal.

(D) Mercury Porosimeter results:

Result of Sample 1 from the equipment (Coal without acid and surfactant):



ThermoFisher SCIENTIFIC

C1

Comment

Report date:10-07-13

COMPRESSIBILITY CORRECTION

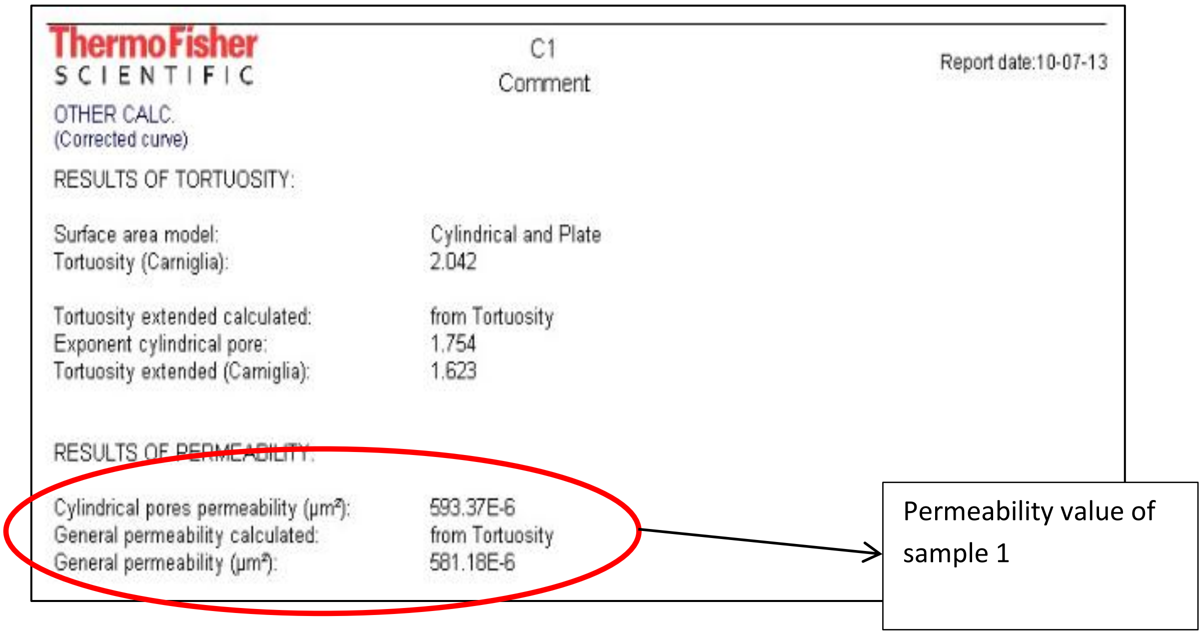
RESULTS WITH COMPRESSIBILITY CORRECTION

Correction enabled (Y/N): Yes
Linearity range: From (MPa): 160.4089 To (MPa): 200.5111
Correction factor (mm³/g.MPa) 0.000000
Compressibility factor (MPa.g/mm³) 0.0000
Compressed volume (mm³/g): 0.00
Sample compressibility (1/MPa): 0.000E+0
Bulk modulus (MPa): 0.000E+0

Total intruded volume (mm³/g): 95.95 at pressure (MPa): 200.5111
Intruded vol. (mm³): 5.92
Envelope density (g/cm³): 1.7343
Bulk density @ pressure (g/cm³): 1.7812 at pressure (MPa): 0.1735
Apparent density (g/cm³): 2.0805 at pressure (MPa): 200.5111
Real void volume by real dens. (mm³/g): 223.39
Accessible porosity (%): 16.64
Inaccessible porosity (%): -55.38

Porosity value of sample 1

Figure 22. Porosity value of Sample 1 from Mercury Porosimeter



ThermoFisher SCIENTIFIC

C1

Comment

Report date:10-07-13

OTHER CALC.
(Corrected curve)

RESULTS OF TORTUOSITY:

Surface area model: Cylindrical and Plate
Tortuosity (Carniglia): 2.042

Tortuosity extended calculated: from Tortuosity
Exponent cylindrical pore: 1.754
Tortuosity extended (Carniglia): 1.623

RESULTS OF PERMEABILITY:

Cylindrical pores permeability (μm²): 593.37E-6
General permeability calculated: from Tortuosity
General permeability (μm²): 581.18E-6

Permeability value of sample 1

Figure 23. Permeability Value of Sample 1 from Mercury Porosimeter

$$1m^2 = 1.013249966 \times 10^{12} D$$

$$1\mu m^2 = 10^{-12} m^2 = 1.013249966 D$$

$$518.18 \times 10^{-6} \mu m^2 = 518.18 \times 10^{-18} m^2 = 0.525 mD$$

Where;

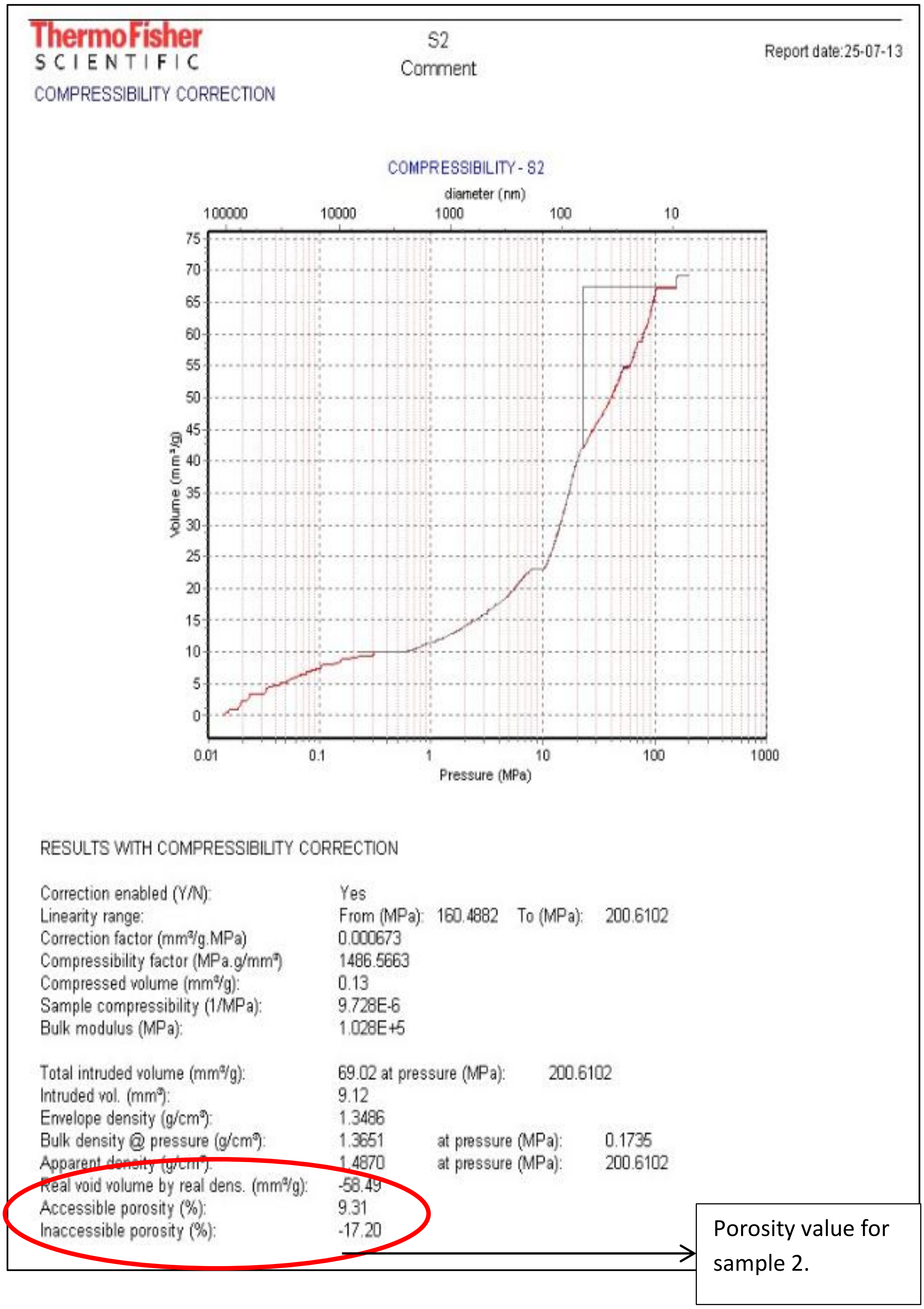
m^2 – meter squared

$\mu\mathrm{m}^2$ – micro meter squared

D – darcy

mD – milli darcy

Result of Sample 2 (Acid without surfactant):



ThermoFisher SCIENTIFIC

S2

Comment

Report date:25-07-13

COMPRESSIBILITY CORRECTION

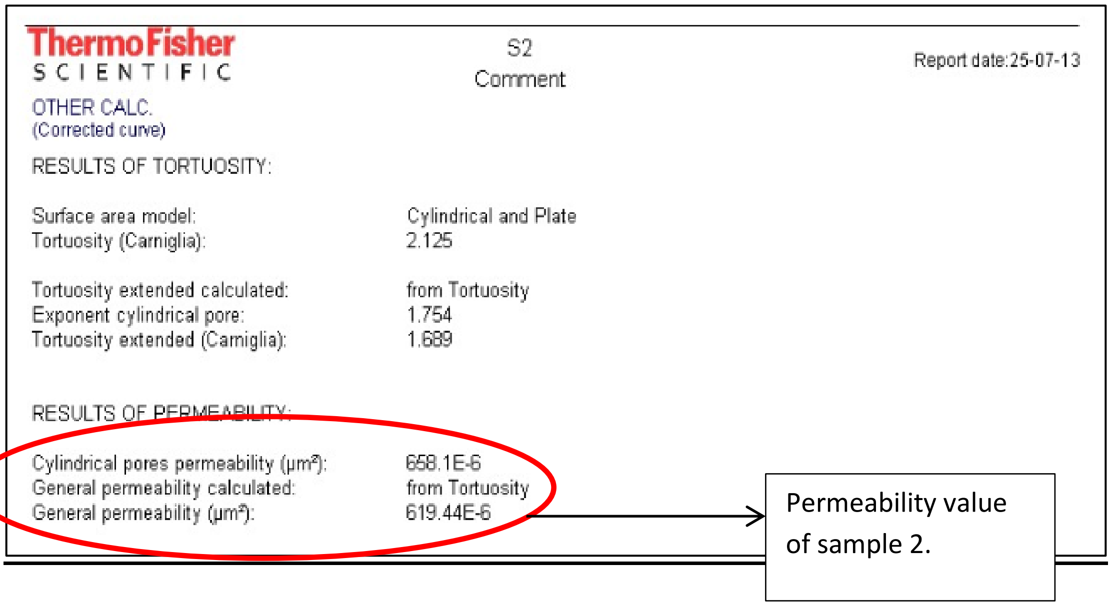
RESULTS WITH COMPRESSIBILITY CORRECTION

Correction enabled (Y/N):	Yes			
Linearity range:	From (MPa): 160.4882		To (MPa):	200.6102
Correction factor (mm^3/g.MPa)	0.000673			
Compressibility factor (MPa.g/mm^3)	1486.5663			
Compressed volume (mm^3/g):	0.13			
Sample compressibility (1/MPa):	9.728E-6			
Bulk modulus (MPa):	1.028E+5			

Total intruded volume (mm^3/g):	69.02 at pressure (MPa):	200.6102	
Intruded vol. (mm^3):	9.12		
Envelope density (g/cm^3):	1.3486		
Bulk density @ pressure (g/cm^3):	1.3651	at pressure (MPa):	0.1735
Apparent density (g/cm^3):	1.4870	at pressure (MPa):	200.6102
Real void volume by real dens. (mm^3/g):	-58.49		
Accessible porosity (%):	9.31		
Inaccessible porosity (%):	-17.20		

Porosity value for sample 2.

Figure 24. Porosity Value of Sample 2 from Mercury Porosimeter



ThermoFisher SCIENTIFIC

S2

Comment

Report date:25-07-13

OTHER CALC.
(Corrected curve)

RESULTS OF TORTUOSITY:

Surface area model:	Cylindrical and Plate
Tortuosity (Carniglia):	2.125
Tortuosity extended calculated:	from Tortuosity
Exponent cylindrical pore:	1.754
Tortuosity extended (Carniglia):	1.689

RESULTS OF PERMEABILITY:

Cylindrical pores permeability (μm²):	658.1E-6
General permeability calculated:	from Tortuosity
General permeability (μm²):	619.44E-6

Permeability value of sample 2.

Figure 25. Permeability Value of Sample 2 from Mercury Porosimeter

$$1m^2 = 1.013249966 \times 10^{12} D$$

$$1\mu m^2 = 10^{-12} m^2 = 1.013249966D$$

$$619.44 \times 10^{-6} \mu m^2 = 619.44 \times 10^{-18} m^2 = 0.628mD$$

Where;

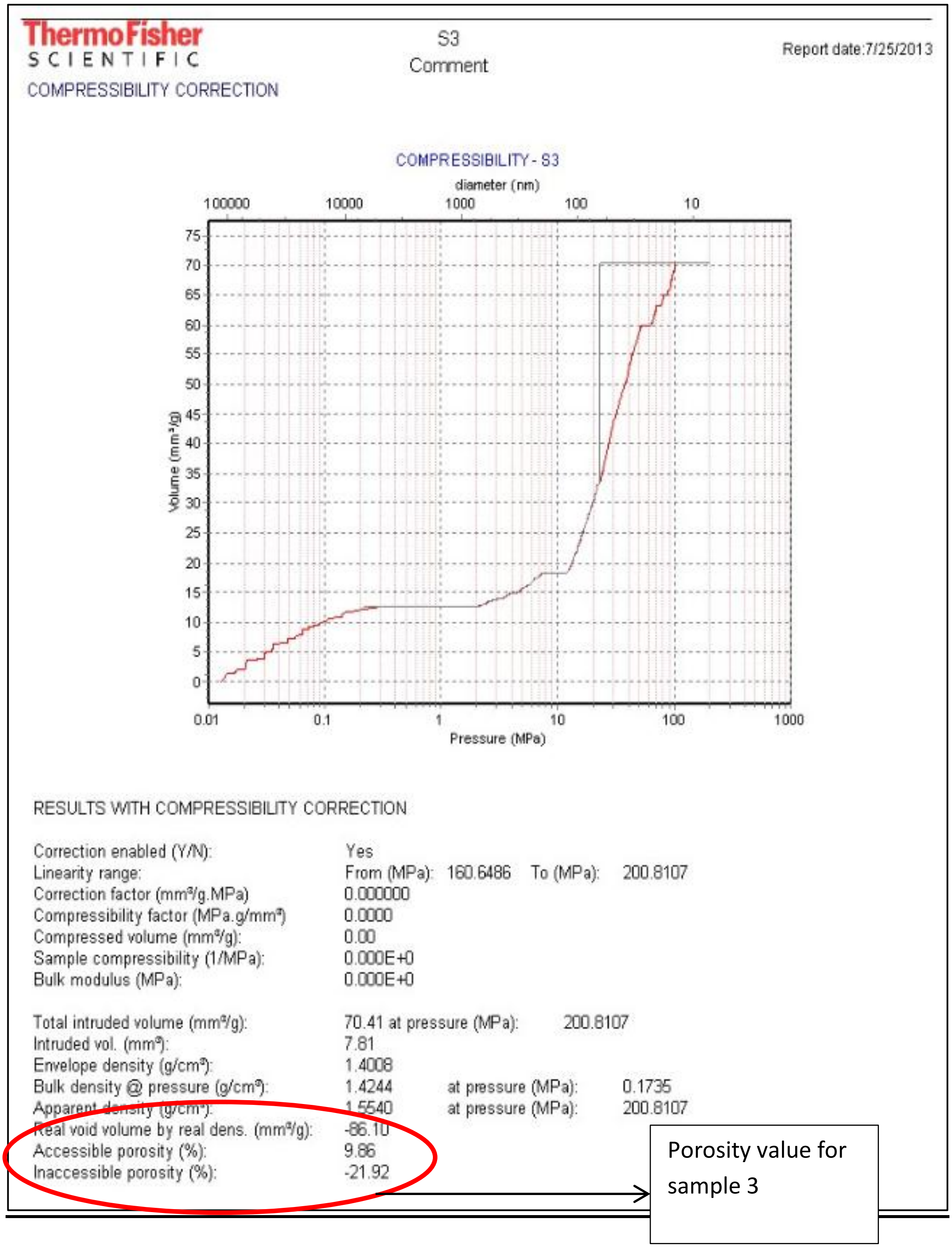
m^2 – meter squared

$\mu\mathrm{m}^2$ – micro meter squared

D – darcy

mD – milli darcy

Result of Sample 3 (Acid with surfactants-Methanol):



ThermoFisher SCIENTIFIC

COMPRESSIBILITY CORRECTION

S3
Comment

Report date:7/25/2013

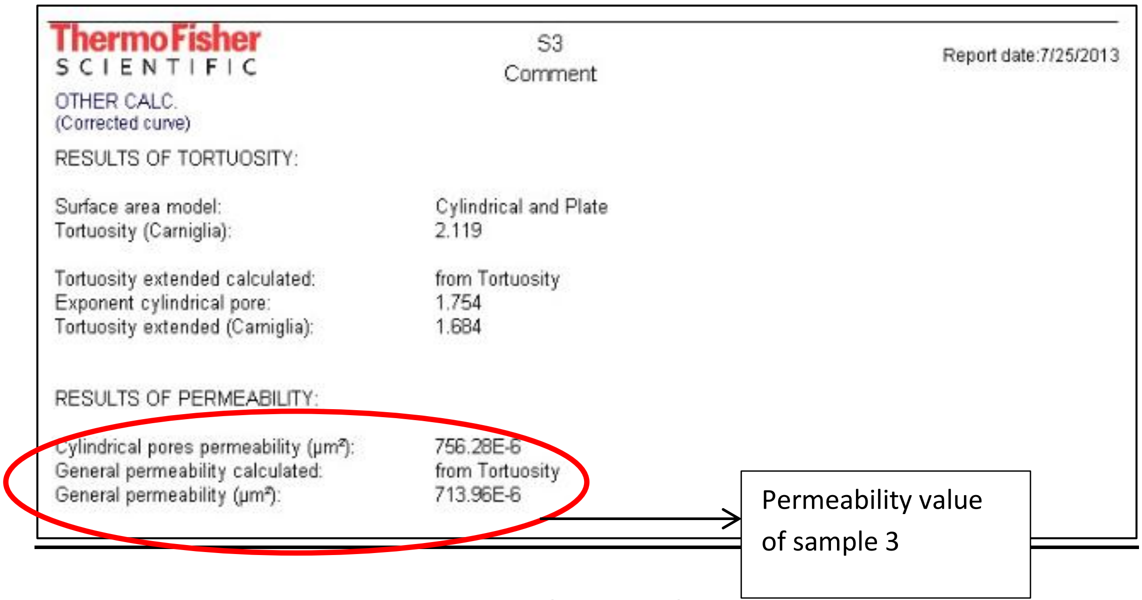
```
RESULTS WITH COMPRESSIBILITY CORRECTION

Correction enabled (Y/N):                    Yes
Linearity range:                             From (MPa):  160.6486    To (MPa):     200.8107
Correction factor (mm³/g.MPa)                0.000000
Compressibility factor (MPa.g/mm³)           0.0000
Compressed volume (mm³/g):                   0.00
Sample compressibility (1/MPa):              0.000E+0
Bulk modulus (MPa):                          0.000E+0

Total intruded volume (mm³/g):               70.41 at pressure (MPa):      200.8107
Intruded vol. (mm³):                         7.81
Envelope density (g/cm³):                    1.4008
Bulk density @ pressure (g/cm³):             1.4244        at pressure (MPa):      0.1735
Apparent density (g/cm³):                    1.5540        at pressure (MPa):      200.8107
Real void volume by real dens. (mm³/g):      -86.10
Accessible porosity (%):                     9.86
Inaccessible porosity (%):                   -21.92
```

Porosity value for sample 3

Figure 26. Porosity Value of Sample 3 from Mercury Porosimeter



ThermoFisher SCIENTIFIC

S3

Comment

Report date:7/25/2013

OTHER CALC.

(Corrected curve)

RESULTS OF TORTUOSITY:

Surface area model:	Cylindrical and Plate
Tortuosity (Carniglia):	2.119
Tortuosity extended calculated:	from Tortuosity
Exponent cylindrical pore:	1.754
Tortuosity extended (Carniglia):	1.684

RESULTS OF PERMEABILITY:

Cylindrical pores permeability (µm²):	756.28E-6
General permeability calculated:	from Tortuosity
General permeability (µm²):	713.96E-6

Permeability value of sample 3

Figure 27. Permeability Value of Sample 3 from Mercury Porosimeter

$$1m^2 = 1.013249966 \times 10^{12}D$$

$$1\mu m^2 = 10^{-12}m^2 = 1.013249966D$$

$$713.96 \times 10^{-6}\mu m^2 = 713.96 \times 10^{-18}m^2 = 0.723mD$$

Where;

m^2 – meter squared

$\mu\mathrm{m}^2$ – micro meter squared

D – darcy

mD – milli darcy

The next experiment is by using the Mercury Porosimeter to see the changes in permeability of the coal after being injected with acid and surfactant. Based on the three samples results at the Result Section (Previous section), it shows the increment in permeability after the coal been injected with acid, which is 0.628mD, higher than 0.525mD for coal without acid. Furthermore, the surfactant also shows best result in terms of permeability value compared with the other two samples, which gives the value of 0.723Md. Thus, this experiment shows that surfactant injection has its impact in order to avoid the formation damaged problem which is occurred because of acid injection.

CONCLUSION AND RECOMMENDATIONS

Based on the 4 research activities doing in this project, which are, ECLIPSE Simulation Software, the Gas Adsorption Column Unit experiment, the porosity calculation experiment and lastly the Mercury Porosimeter experiment, they indeed show the impact of acid injection with surfactant on the CBM recovery. They show the impact in terms of the production rate (ECLIPSE), gas adsorption capacity, porosity and permeability. Sodium Lauryl Sulfate (SLS) is a good surfactant, besides amphoteric surfactant, that has the potential to be used in the acid injection process to prevent the formation damage. However, the acid injection with surfactants need to be study and investigated further and deeper as this actually has a good prospect to increase the recovery of the CBM.

For the follow up process, the experiment which investigates the effect of surfactant only, without acid should be implemented as the surfactants have shown a good response towards the recovery of methane gas from CBM, based on this project. As acetone and methanol are widely available compared with another surfactants, thus these two surfactants, together with SLS which also show a very good effect on methane recovery, need to be investigated further , and focus on the effect of reservoir temperature on these three types of surfactants.

REFERENCES

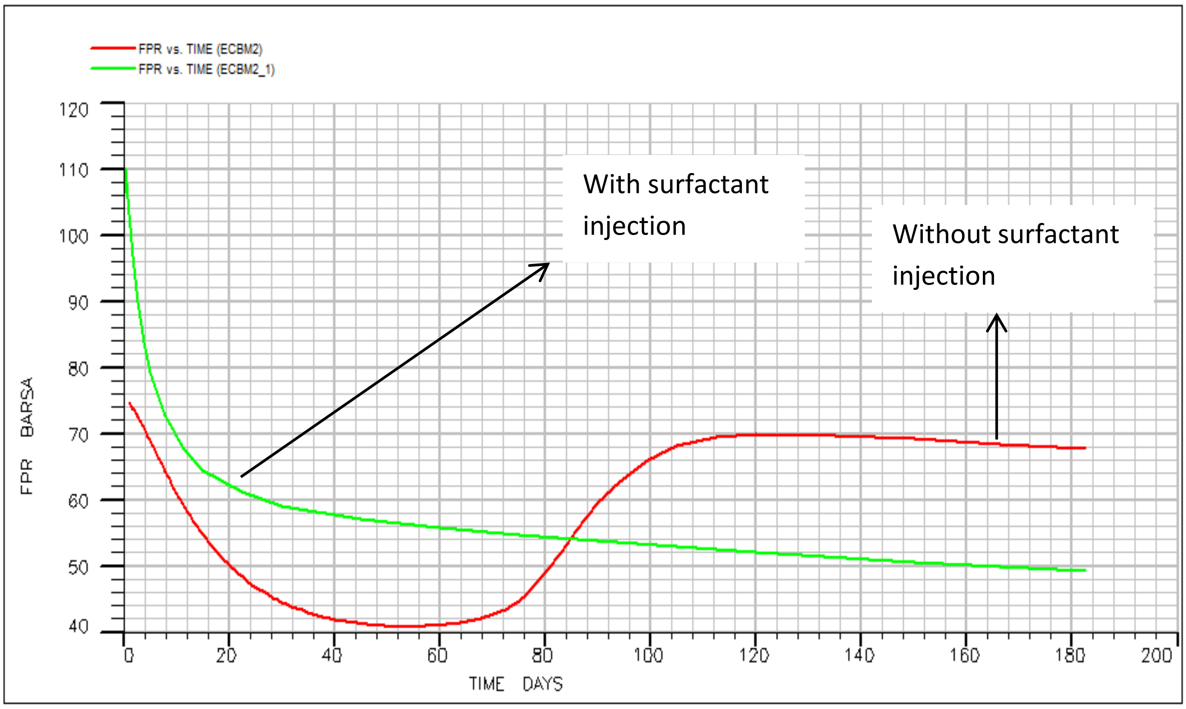
1. Aminian, K. (2009). Coalbed Methane – Fundamental Concepts. Petroleum & Natural Gas Engineering Department West Virginia University
2. Conway M. W. and etc (November 10, 1994). "Stimulation of Coalbed Methane Production". Retrieved from http://www.google.com/patents/WO1994025730A1?cl=cn on 15th June 2013
3. Iqbal Ahmed, Ani Idris, Ahmed Hussain, Zainal bin Abidin & Saad Khan (2013). Influence of Co-Solvent Concentration on The Properties of Dope Solution and Performance of PES Membrane. Accepted article in Press of Chemical Engineering & Technology, 1 July 2013.
4. Keith K. and Bauder J. (2003). "Frequently Asked Question- Coal Bed Methane (CBM)". Montana State University-Bozeman John Wheaton, Montana Bureau of Mines and Geology
5. Nilabjendu Ghosh (2011). "Permeability of Indian Coal". Department of Mining Engineering National Institute of Technology, Rourkela-769008
6. Nimerick, K.H and etc. (June 1990). "Design and Evaluation of Stimulation and Workover Treatments in Coal Seam Reservoirs". *Society of Petroleum Engineers*, paper no.: SPE 23455
7. Paulina Mwangi (December 2010). "An Experimental Study of Surfactant Enhanced Waterflooding". Lousiana State University and Agricultural and Mechanical College
8. Schlumberger (2003). Coalbed Methane Reservoirs, *Oilfield Review Magazine.* Retrieved from www.slb.com/resources/publications/oilfield_review.aspx on 14th June 2013

.

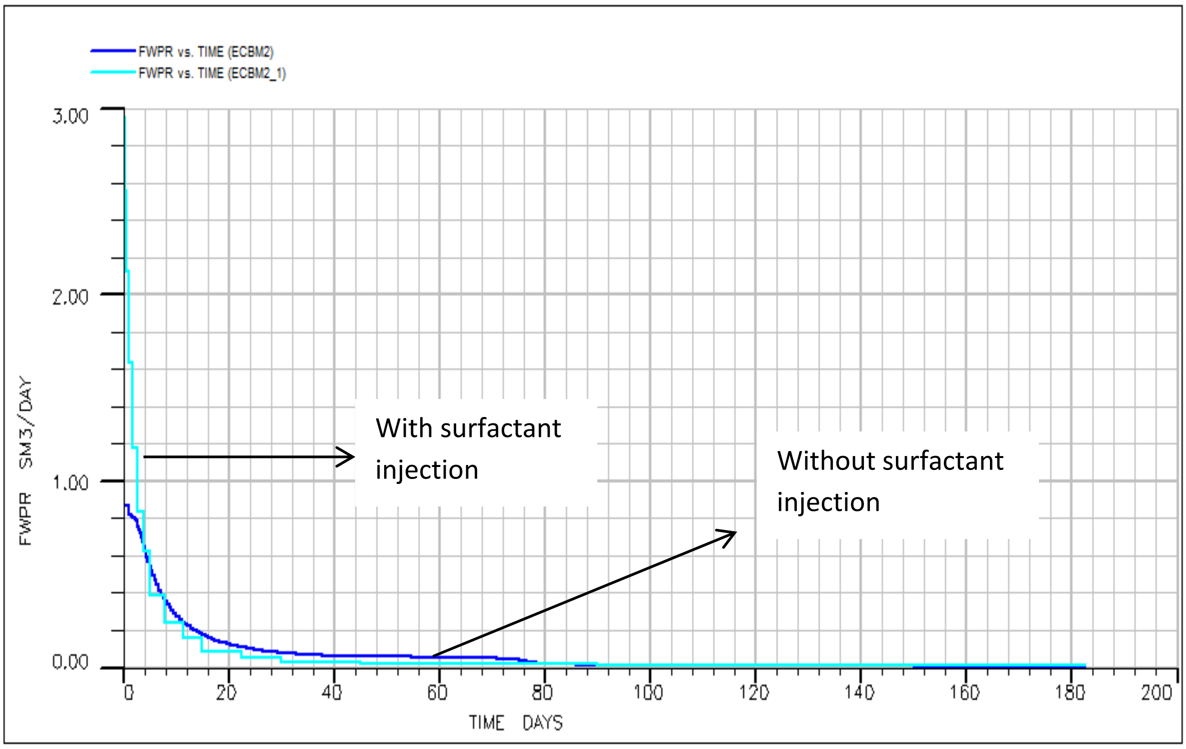
APPENDIX

Appendix 1

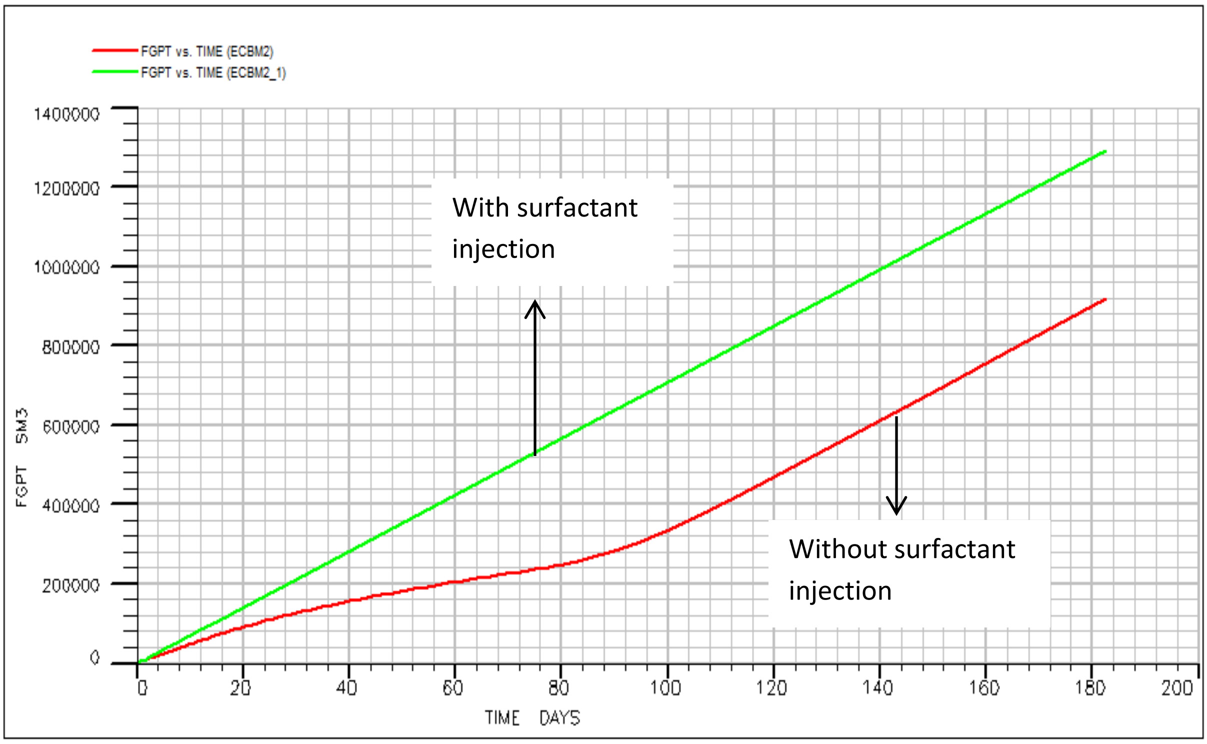
(a) Field Pressure Rate vs Time/ Days



(b) Field Water Pressure Rate vs Time/ Days



(c) Field Gas Production Total vs Time/ Days



Appendix 2

GACU (Gas Adsorption Column Unit)

Mercury Porosimeter