

Comparative studies on the effect of surfactant used in optimizing the hydraulic fracturing technique for shale gas formation

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Supervisor: Dr Sonny Irawan

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

Petroleum Engineering Programme

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2013

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.

MOHD RIDZUAN BIN HAMID

ABSTRACT

Hydraulic fracturing plays a major role in enhancing petroleum reserves and daily production. It consists of blending special chemical to make the appropriate fracturing fluid and then pumping the blend fluid into the pay zone at high rates. The project done is based in the study of the effect of surfactant in hydraulic fluid used in optimizing hydraulic technique towards alteration of shale gas formation. The objectives are to estimate reduction of interfacial tension by applying the surfactant in hydraulic fluid, to investigate the effect of surfactant in hydraulic fluids towards improving the distribution of strain and stress in the shale formation and to estimate the tolerance level for the fracture pressure after surfactant and hydraulic fluids is injected in the formation. Reason of choosing shale formation as a field of study is due to increasing in demand for unconventional drilling for the natural resources. In this project the surfactant used in the hydraulic fluid can act as the de-emulsifier or as emulsifier. Surfactant could also lower the surface tension and reduce the capillary pressure which result in lower the energy required to move the hydraulic fluid across the boundaries and through the formation. In this project, the author will study on the effect of strain and stress distribution after the hydraulic fluid which contains surfactant is injected in the formation. Besides, addition of surfactant in the hydraulic fluid will help in reducing the surface tension. The theory is brought into laboratory work where clean shale formation is tested in tri-axial equipment test for stress and strain distribution measurement in hydraulic fracturing technique by using surfactant as the additive medium in the injected fluid. Three different concentrations of surfactant solutions are planned to be prepared by varying the percentage of additive. The result of core samples for the distribution of strain and stress measurement will be able to describe concisely the effect of surfactant used in the hydraulic fluid. Based on the hypothesis, additive agent (surfactant) in hydraulic fluid will help in improving the shear and strain stress distribution.

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CHAPTER 1

INTRODUCTION

1.1 Back ground study

Over the year, the technology associated with fracturing has improved significantly. A host of fracturing fluid has been developed for reservoir ranging from shallow, low temperature formation to those in deep or hot area. New design models and analytical and diagnostic methods have emerged and the service industry has continually developed new equipment to meet the merging challenges.

This include in practice for the unconventional resources for shale gas formation. Unconventional resources require fracture stimulation to achieve hydrocarbon production at economic rates. Organic-rich, low permeability shale deposit are becoming increasingly vital to the production of natural gas. The primary purpose of stimulating fractured shale formations is extend the drainage radius by creating a long fracture that connect natural fracture and increase flow channel to the wellbore.

Compatibility of treating fluid with the formation and reservoir fluids should not be overlooked. Damage often occurs when formation containing swelling and migratory clays which expose to the aqueous fluid. This paper describes the laboratory experiment which compares the study on the effect of surfactant used in fracturing fluid for optimizing hydraulic fracturing technique towards alteration of shale gas formation. Besides this study will mainly focuses in improving the effective stress in the formation. Once the hydraulic fluid is injected in the formation will give a significance impact on the distribution of pressure. Surfactant or surface acting agent will help in reducing the surface tension and reduce the capillary pressure in the formation.

1.2 Problem statement

1.2.1 Problem identification

Critical analysis towards hydraulic fracturing technique is one way to have better understanding of the application or the limitation in designing the suitable surfactant to be used for shale gas formation. In the case of shale formation, surfactant used need to be compatible in order to adapt with the formation. Alteration of shale gas formation will be main focus as injection of fluid will effect in distribution of stress. Does the surfactant used in hydraulic fluid will be able to improve and indicate the effective stress of the formation.

In shale formation with low permeability and porosity, there are possibilities that the hydraulic fluid will impair the formation and reduce the tendency to penetrate in the formation. Does the surfactant used in hydraulic fluid will have the capacity to reduce the surface tension and capillary pressure in the formation. This possibility should be tested and analyzed in order to see if the improvement can be further described for betterment of the hydraulic technique.

In order to do so, laboratory work can be done in determining the effective stress in formation cause by the hydraulic fluid injected in the formation where its limitation and expectation can be drawn.

1.3 Objective and scope of study

1.3.1 Objective of the project

By using understanding the concept of hydraulic fracturing technique towards alteration of shale gas formation, there are two main goals to be achieved from this project which are:

- To estimate reduction of interfacial tension by applying the surfactant in hydraulic fluid
- To investigate the effect of surfactant in hydraulic fluid towards improving the distribution of strain and stress in the shale formation
- To estimate the tolerance level for the fracture pressure after surfactant and hydraulic fluid is injected in the formation

1.3.2 Scope of study

In completing the research regarding this project, there are several scopes of study will be emphasized and explained throughout the project process flow. The basic understanding starts with the earliest and fundamental of hydraulic fracturing technique. Then the scope study is narrowed to the designing the preferable hydraulic fracturing fluid by adding the additive agent. Study will move on the additive used or the surfactant in hydraulic fluids for the shale formation.

In this project, Tellus 46 of hydraulic oil is used as the oil based fluid and sodium lauryl sulphate (SLS) as the surfactant or additive agent. Although modified hydraulic fluid introduces additional effect for shale formation especially in maintaining the distribution of strain and stress. Besides, the additional additive agent in hydraulic fracturing fluid will reduce the surface tension and capillary pressure. To prove this presumption, the theory is brought into lab work to be studied further.

The scope of study will be concluded in the form of research data to see whether or not additional additive agent (surfactant) can affect the distribution of stress and strain in the shale formation.

1.4 The relevancy of the project

The project is relevant to the author as a Petroleum Engineering student who had already completed course related to the well stimulation technique and formation evaluation study. Moreover, the understanding about the subsurface formation and their properties such as distribution of stress and strain in rock formation is crucial for hydraulic fracturing technique in determining the preferable injection rate and to avoid from damaging the formation from excessive injection pressure. This project could also provide critical analysis and giving new exposure for future engineer as unconventional drilling for shale gas formation has come into practice in oil and gas industry. The analysis process that is supported with experimental data will be able to improve author's ability to make significant reasoning.

CHAPTER 2

LITERATURE REVIEW

This chapter discusses about the theories and paperwork reviews related to this project. There are few areas of focus which contribute in completing this project and have become guidance as reference. The area covers in shale gas well formation, hydraulic fracturing technique, fracturing fluids and additives, oil-based fracturing fluid, surfactants and fracture mechanic (In situ stress)

2.1 Shale Gas well formation

According to the Brandon.N (Sep,2007) in his paper regarding predicting cumulative production of Devonian Shale Gas wells from early well performance data, Appalachian Basin of Eastern Kentucky explains about the entire shale gas well properties and reservoir characteristic in detail. In this paper states that the Appalachian Basin is dominated by a sequence of black and gray shale which often organic rich units are thought to be the source beds for much of the hydrocarbon produced in the basin. The shale itself can be a reservoir containing free gas in the natural fracture system and absorbed gas. In this Appalachian Basin, organic rich unit alternate with gray shale consisting mostly quartz and clay minerals. The shale range in thickness from 0 meters in placing along the crest of the Cincinnati Arch to more than 1097 meters in west Virginia. In the gas productive areas of Kentucky, the shale is typically 60 meters to 480 meters thick. The shale ranges in depth from the outcropping on the western margin of the basin to more than 1200 meters. Shale gas production was discovered on eastern Kentucky during 1892 and today there are estimated to be more 6000 shale gas well producing between 50 and 70 billion cubic feet of gas annually.

2.2 Hydraulic fracturing Technique

The hydraulic fracturing plays a major role in enhancing petroleum reserves and daily production. It consists of blending special chemicals to make the appropriate fracturing fluid and then pumping the blended fluid into the pay zones at high enough rates and pressure to wedge an extended a fracture hydraulically (adapted from John L.G and et al, 2001 in recent advances in hydraulic fracturing handbook). First, a neat fluid called a “pad” is pumped to initiate the fracture and to establish propagation. This is followed by slurry of fluids mixed with a propping agent. This slurry continues to extend the fracture and concurrently carries the proppant deeply into the fracture. After the materials are pumped, the fluid chemically breaks back to lower viscosity and flows back out of the well, leaving a highly conductive propped fracture for oil or gas to flow easily from the extremities of the formation into the well. Fracture has two wings extending in opposite directions from the well and is oriented more or less in the vertical plane. Other fracture configuration such as horizontal fracture is known to exist some have been observed at relatively shallow depths which are less than 200 feet or 610m. Fracturing has made a significance contribution in enhancing oil or gas producing rates and recoverable reserves. The fracturing process, introduced to the industry in 1947 is a standard operating practice. By 1981 more 800,000 treatments had been performed. As 1988 this has grown to exceed 1 million. About 35 to 40 % of all currently drilled wells are hydraulically fractured and about 25 to 30 % of the total U.S oil reserve has been made economically produced by the process.

2.3 Fracturing Fluids and additive

According to the John L.G and et al in their Recent Advance in hydraulic Fracturing handbook volume 12 (2001) describes the purpose of a fracturing fluid is basically to wedge open and extend a fracture hydraulically and to transport and distribute the proppant along the fracture. The fluids selected for a treatment can have a significant influence on the resulting effectively propped fracture length, fracture conductivity and treatment cost. Fluid properties strongly govern fracture-propagation behavior and the placement of the propping agents. Fluid leak off rapidly into the formation have a low efficiency in hydraulically wedging and extending a fracture. Fluid leak off may also result in undesirable concentration of

residue in the fracture. The effective viscosity of the fluid controls the internal fracturing pressure and the proppant transport characteristic. Some of the desirable features of a fluid for the majority of the fracturing treatments which are low fluid loss to obtain the desired penetration with minimum fluid volume, sufficient effective viscosity to create the necessary fracture width and to transport the proppant in the fracture. Fracturing fluid should be compatible with the formation material. If the chemical nature of the fracturing fluid causes swelling of naturally occurring clays in the formation, thereby plugging pore channel and the treatment will be a failure. If the fracturing fluid causes migration of fines and clay, the success of the treatment will be nullified. If the fracturing creates emulsion and sludging of the crude oil, then plugging rather stimulation will occur. If the fracturing fluid dissolves the cementing material that holds the grains of the sandstone together, spalling of the formation can occur and failure will result. The fracturing fluid should not cause scaling or paraffin problem. Compatibility is therefore critical and necessary of a fracturing fluid. The ideal fracturing fluid should be moderately efficient. A high percentage of the fluid should remain in the fracture and not be lost to the formation. Fluid efficiency is normally attained by combining high fluid viscosity with fluid-loss additives. These fluid-loss additives may consist of plastering agents, bridging agents, microemulsions or emulsified gas. A low efficiency fracturing fluid would not create the desired formation penetration of most of the fracturing fluid leaks off during treatment.

2.4 Oil-Based fracturing fluids

The most common oil-based fracturing gel available today is a reaction product of aluminum phosphate ester and a base typically sodium aluminates. Reaction of the ester and the base creates an association reaction, which in turn creates a solution that yield viscosity in diesel or moderate to high gravity crude system (John L.G and et al, 2001). The aluminum phosphate ester gel have been improved to gel more crude oils and to enhance temperature stability. The earliest viscosities oils were napalm type fluid of aluminum octoate. Later fluids were reaction products of caustic and tall oil fatty acids, in fact some of these fluids are still in use. These fatty acid soap, although useful as fracturing fluid, frequently cause permeability problems. Aluminum phosphate esters can be used to create fluids with enhanced stability at

high temperature and good proppant carrying capacity for use on wells with BHT's excess of 260 °F [127 °C]. Using gelled hydrocarbon is advantages in certain situation to avoid formation damage to water sensitive oil producing formation that may be caused by the use of water based fluids. If the produced crude has high enough gravity, typically above 35° [0.85 g/cm³], then produced crude oil can be used to fracture the formation. The primary disadvantage of using gelled oil systems is the fire hazard. In most cases, the pumping friction of an oil based fluid is higher than a delayed, cross linked water based fluid system. Pumping pressure is also higher because of a lack of hydrostatic head of the hydrocarbon compared with water. Additional, when one fractures a high temperature well (Above 260 ° F [127 °C]), the temperature stability of a delayed, cross linked water based system is more predictable and such a system is less costly than typical oil based fluid system. It should also be mentioned that preparation of oil based fracturing fluids requires a great of technical capability and quality control. The preparation of water based fracturing fluids is relatively straightforward by comparison. In particular the preparation and quality control of gelling crude oil require much care than those of water based fluid.

- Aluminum phosphate ester hydraulic fluid

According to Maberry L. J and et al in their SPE paper which is Field evaluation of wells fractured in North La Barge Field Using continuous mix gelled oil state that the characteristic of the fluid. Phosphate ester gelling agents are blend of mono di- and trialkyl ester. The dialkyl ester is the major component with monoalkyl and triakyl ester present in lesser amount. Aluminum salts, such as aluminum chloride, aluminum acetate, and aluminum isopropoxide, were used in prior art to crosslink phosphate ester. The use of iron compound to crosslink phosphate ester was suggested as early 1970, but aluminum phosphate ester chemistry is more prevalent at this time. Aluminum cross linking in phosphate ester occurs at the hydroxyl site. The alkyl groups on the phosphate ester have an affinity for hydrocarbon fluids and keep the phosphate ester in solution. The dialkyl ester has one hydroxyl group and good solubility in hydrocarbon fluids due to the presence of two alkyl group. Monoalkyl ester has two hydroxyl groups available. However solubility is limited by the presence of only one alkyl group. Trialkyl ester has no hydroxyl groups, but has three alkyl groups and excellent solubility in hydrocarbon fluids. The crosslink

aluminum phosphate ester is affected by water, acid and base (attracted to the polar aluminum ion) that disrupt orientation resulting in weakened gel structure, limited thermal stability, and a premature loss of viscosity. The crosslink fluid is shear thinning but not shear degradable.

- Tellus oil 46

High performance of hydraulic fluids which provide outstanding protection and performance in most manufacturing operation. This fluid has the capacity of long fluid life and minimizes sludge formation by providing excellent performance. Besides, proven zinc-based anti wear additives are incorporated to be effective throughout the range of operating conditions. This is included in low load and severe duty high load condition. Outstanding performance in a range of piston and vane pump test. This fluid is tested with superior cleanliness, excellent filterability and high performance of water separation. Moreover, helps reduce the impact of contamination on filter blocking. This type of oil is formulated for fast air release in order to help efficient hydraulic power transfer and minimize fluid impact on the cavitations induced oxidation that can shorten fluid life.

2.5 Surfactant and non-emulsifier

According to the John L.G and et al in their Recent Advance in hydraulic Fracturing handbook volume 12 (2001) also describe in details about the surfactants. A surfactant (surface-active agent) can be defined as a molecule that seek out an interface and has the ability to alter the prevailing condition. A surfactant is almost always composed of two parts which are a long hydrocarbon chain that is virtually insoluble in water but soluble in oil and strongly water soluble tail. Because there is partial solubility in oil and water, the surfactant will tend to accumulate at the interface of these fluids. The water soluble portion of the molecule may be ironically positive (cationic), negative (anionic) or mixed amphoteric. The ionic charge of the various surfactants used in oilfield stimulation is important in terms of wettability imparted to a given formation. The inherent ionic characteristic of particular formation cause cationic surfactant to leave carbonates water wet and sandstone oil wet. Anionic surfactants tend to leave sandstones water wet and limestone oil wet. Amphoteric surfactants are organic molecules whose ionic charges depend on the pH of the fluid. Almost all formations are naturally water wet condition is preferred the

ionic nature of the surfactant is an important consideration and one should be aware of the charge of a surfactant in its selection. It is generally inadvisable to mix cationic with anionic because of the possibility of forming precipitates. Because a large number of formations throughout the world are heterogeneous, limy sand or sandy lime, it is often useful to select nonionic surfactant, provided that it meets certain non-emulsification criteria. An emulsion consists of two immiscible fluids in which one phase exist as fine droplets dispersed throughout the other phase. Oil field emulsions are either oil in water (where oil droplets exist in the continuous water phase) or water in oil (where oil droplets are the continuous phase). The viscosity of an emulsion can vary from several to several thousand centipoises. If an emulsion is created near wellbore, severe productive blockage may occur. Because of their surface active nature, surfactant can act as de-emulsifier or as emulsifiers. Effectiveness of a surfactant as de-emulsifiers in a particular crude oil system must be determined experimentally. Test should be run according to specification set out in API RP-42 to determine the proper type and concentration of surfactant required to prevent emulsification of particular crude with a treating fluid. The surfactant should be maintain its surface activity at reservoir temperature and should not be easily stripped out of solution by adsorption from contact with the reservoir rock. As discussed earlier, some fracturing fluids are composed of hydrocarbon and water that are emulsified to build fluid viscosity. When emulsified fluids are used, it is desirable to the surfactant to adsorb on the formation so that the emulsions will break. Surfactant can also used to prevent or to treat near wellbore water blocks. Although not as severe as emulsion, a water block can impair production. Surfactant lowers the surface tension of the water and reduces capillary pressure which results in lower energy required to move water across boundaries and through the formation matrix. Another form of well damage that may be treated by surfactant is blockage by fines. Fines can be silt, clay mineral or drilling fluid solid. If a surfactant that wets the individual fine particles is used in the fracturing, the particles can be removed from the formation more easily when broken fracturing fluid is produced back.

- Conventional surfactant

According to the Paktinat . J and et al, 2006 in their SPE paper (104306) which is Case study: Optimizing hydraulic fracturing performance in Northeastern United States fracturing shale formation define the conventional surfactant used in the

hydraulic fracturing technique. Surfactants are defined as a group of chemical consisting of hydrophobic and hydrophilic tails that alter the surface activity in an aqueous media. When a surfactant is dissolved in an aqueous solution its hydrophobic group distorts the hydrogen bonds between the water molecules around the hydrophobic group resulting in decreased surface tension between hydrophobic group and water. Both hydrophobic and hydrophilic groups of surface active agents play an important role in this phenomenon. The hydrophobic portion is normally made up of hydrocarbon ranging from C8-C18 and can be aliphatic, aromatic, or a mixture of both. The main sources of hydrophobe are normally natural fats, oil, petroleum fraction, synthetic alcohols or polymer. The classification of the surfactant comes from the hydrophilic group of the surfactant. This portion identifies surfactant as being anionic, cationic or non-ionic.

- Sodium Lauryl Sulphate

Sulfonic acid is a compound with general formula RSO_2OH , where R is an aliphatic or aromatic hydrocarbon. It is a derivative of sulfuric acid (HOSO_2OH) where an OH has been replaced by a carbon group or a compound where a hydrogen atom has been replaced by treatment with sulfuric acid; for example, benzene is converted to benzenesulfonic acid (water-soluble). Sulfonic acid has a sulfur atom bonded to a carbon atom of a hydrocarbon and bonded also to three oxygen atoms, one of which has been attached to a hydrogen atom. Sulfonic acid is acidic due to the hydrogen atom, stronger than a carboxylic acid. Sulfonic acid is one of the most important organo sulfur compounds in organic synthesis. Sulfonic acids are used as catalysts in esterification, alkylation and condensation reactions. Sulfonates are salts or esters of sulfonic acid. Sulfonic salts are soluble in water. Sulfonic acid and its salts present in organic dyes provide useful function of water solubility and or improve the washfastness of dyes due to their capability of binding more tightly to the fabric. They are widely used in the detergent industry. Alkylbenzene sulfonic acid is the largest-volume synthetic surfactant because of its relatively low cost, good performance, the fact that it can be dried to a stable powder and the biodegradable environmental friendliness. Sodium lauryl Sulphate (SLS), prepared by sulfation of lauryl alcohol and neutralisation with sodium carbonate, is another common surfactant which has amphiphilic properties due to C12 chain (lipophilic) attached to a sulfate group (hydrophilic). This bifunctionality in one molecule

provides the basic properties useful in cleaners and detergents. SLS is used as a wetting agent in textiles, foaming and cleaning agent in detergent, cosmetic emulsifier, and sometimes in toothpastes.

2.6 Fracture mechanics (In-situ stresses)

2.6.1 Mechanic of hydraulic fracturing

According R.M Holt and et al, in Petroleum Related Rock Mechanics 2nd Edition stated that Hydraulic fracturing in rocks takes place when the fluid pressure within the rock exceeds the smallest principal stress plus the tensile strength of the rock. This results in tensile failure or splitting of the rock. A hydraulic fracture may be initiated by natural, geological processes in the earth whereby the fluid pressure increases and/or the smallest principal stress decreases. Artificial or man-made hydraulic fractures in petroleum activities are normally initiated by increasing the fluid pressure in the borehole to the point where the smallest principal stress at the borehole becomes tensile. Continued pumping at an elevated pressure causes the formation to split and the fracture will grow in the direction of least resistance. Some distance away from the borehole the fracture will always propagate in the direction normal to the smallest principal stress in that specific formation.

2.6.2 Fracture initiation and formation breakdown

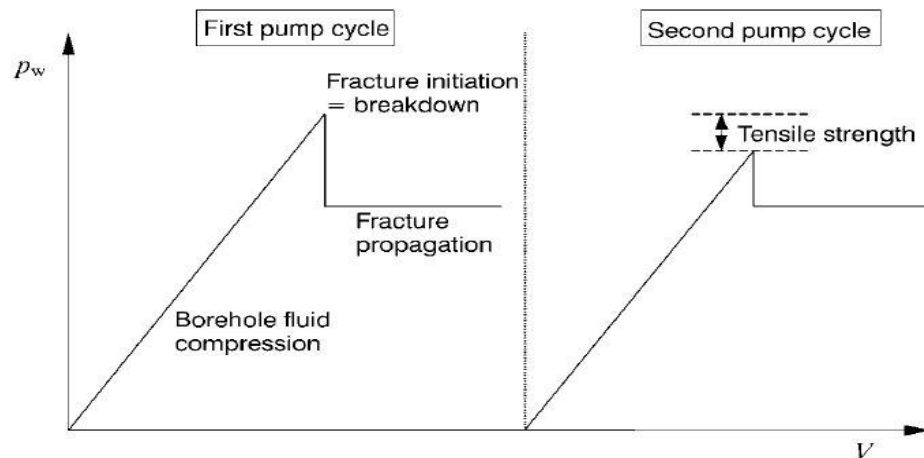


Figure 1 : Idealized borehole pressure response during hydraulic fracturing of a vertical wellbore. Two pressure cycles are included

The first linear part represents the elastic deformation of the system in and around the borehole, primarily compression of the fluid in the borehole. The peak

represents the fracture initiation condition, i.e. the creation of a vertical fracture on the borehole wall. The well pressure drops instantaneously at this point. This implies a situation of unstable fracture growth, whereby the volume of the fracture is growing at a higher rate than the rate of fluid injection. Continued pumping will eventually result in stable fracture growth, represented by the constant well pressure level. In this idealized case the point of fracture initiation and formation breakdown are thus identical.

The second curve in Fig. 1 shows the response which would occur if a second pressure cycle was run. Then the only resistance to fracture initiation and formation breakdown would be the stress concentration around the borehole. The tensile strength is now zero, since the fracture already exists. The difference between the first and the second peak would thus ideally be a direct measure of the tensile strength of the formation. In practice, however, the presence of the fracture may make the effective stress concentration smaller in the repeat cycle than in the first, meaning that the difference is not only related to the tensile strength.

2.6.3 Failure Criteria

Based on the Mohr-Coulomb Criterion where he suggested that rock failure in compression takes place when the shears stress , τ , that is developed on a specific plane (plane *a-b* in Figure 2) reaches a value that is sufficient to overcome the natural cohesion of the rock, as well as the frictional force that oppose motion along the failure plane.

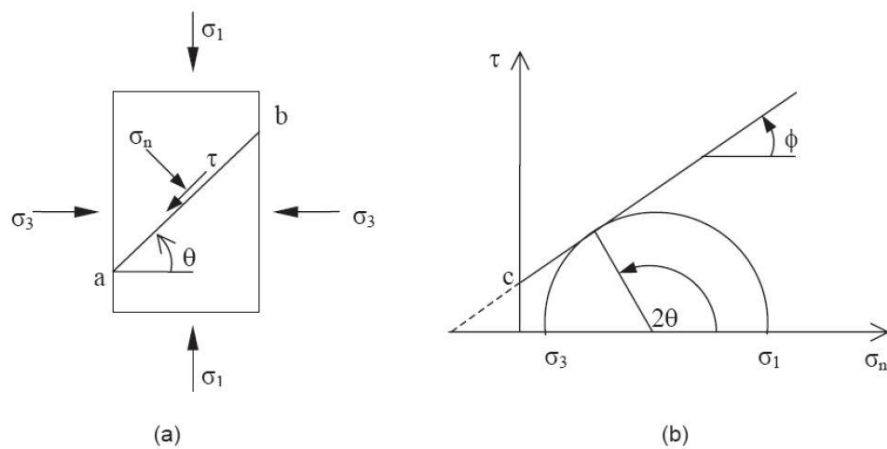


Figure 2 : Mohr-Coulomb failure criterion, (a) Shear failure on plane a-b. (b) Strength envelopes in terms of shear and normal stresses

The criterion can be written as

$$\tau = C + \sigma_n \tan \theta$$

Where σ_n is the normal stress acting on the failure plane, C is the cohesion of the material and θ is the angle of the internal friction. Figure 1 shows the strength envelope of the shear and normal stresses. This criterion can be interpreted as being intended to apply only to situation in which $\sigma_2 = \sigma_3$. The coulomb failure therefore can be represented by the maximum principle stress, σ_1 and minimum principle stress, σ_3 . Where:

$$\sigma_n = \frac{1}{2}(\sigma_1 + \sigma_3) + \frac{1}{2}(\sigma_1 - \sigma_3) \cos(2\theta)$$

$$\tau = \frac{1}{2}(\sigma_1 - \sigma_3) \sin(2\theta)$$

The coulomb criteria can also be expressed in term of the maximum shear stress, τ_{max} and the effective mean stress $\sigma_{m,2}$:

$$\tau_{max} = \frac{1}{2}(\sigma_1 - \sigma_3)$$

$$\sigma_{m,2} = \frac{1}{2}(\sigma_1 + \sigma_3)$$

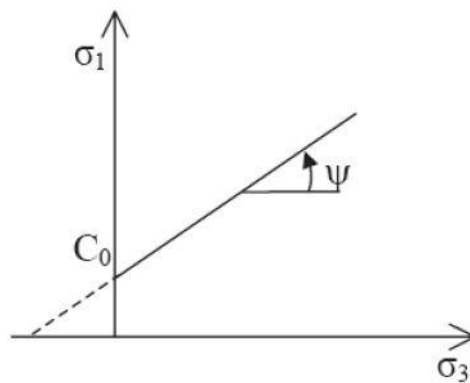


Figure 3 : Mohr-Coulomb strength envelope in term of principle stresses

CHAPTER 3 METHODOLOGY

3.1 Experimental procedure / Project Activities

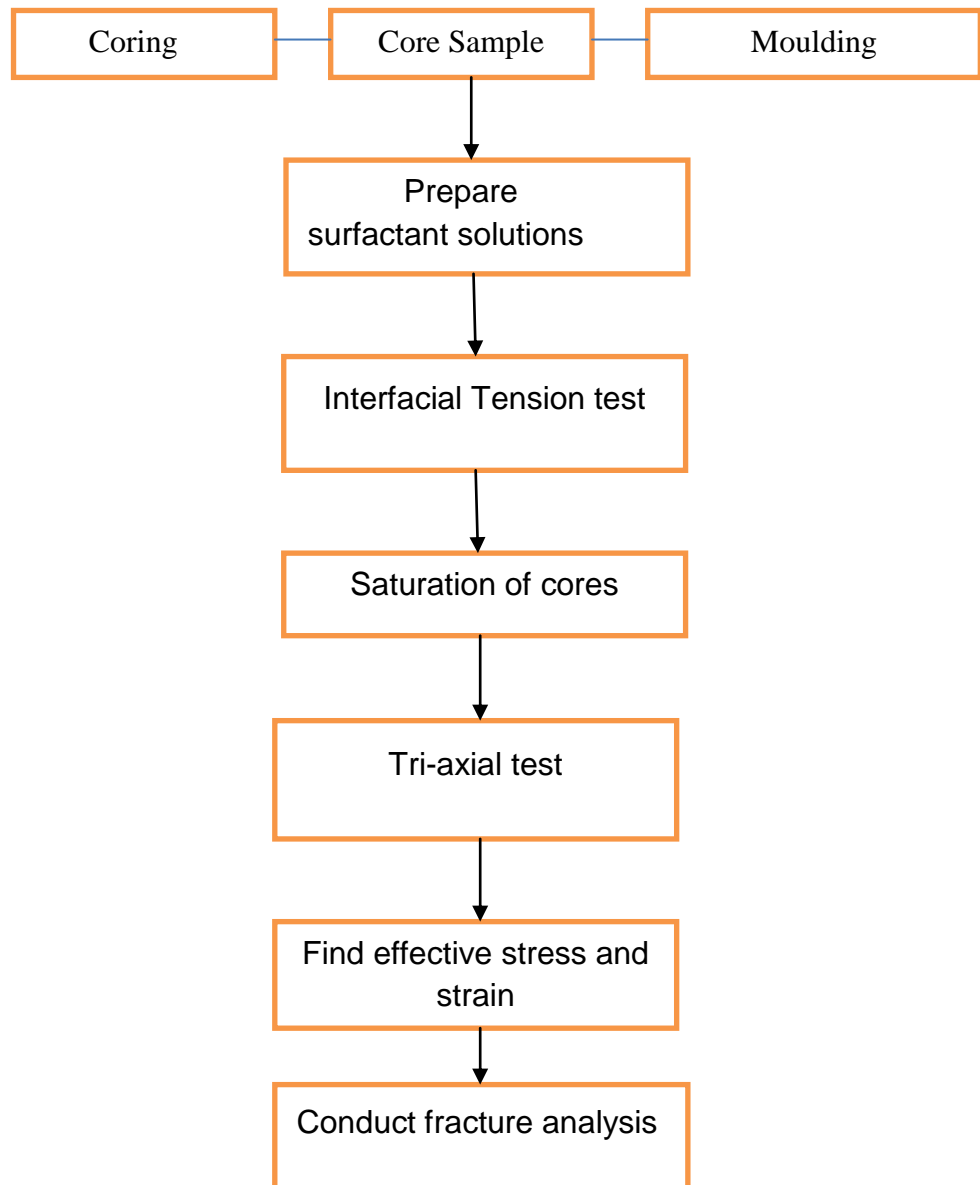


Figure 5 : Project Activities

3.2 List of experiment used in order to fulfill project objectives

Table 1 : List of experiment used

Objective	Methodology	Tools
1. To estimate reduction of interfacial tension by applying surfactant in hydraulic fluid	<ul style="list-style-type: none"> > Interfacial Tension Test (IFT) > Prepare fluid sample with different density and concentration 	>Spinning Drop Equipment
2. To investigate the effects of surfactant in improving the distribution of stress and strain 3. To measure the fracture pressure after surfactant and hydraulic oil is injected in the formation	<ul style="list-style-type: none"> > Stress and Strain measurement > Conduct compression test for saturated shale core sample 	> Tri-axial equipment- Hydraulic fracturing stimulation for core sample

3.3 Experimentation setup

Part 1: Core Sample Preparation

I. Core sampling

Raw materials of core samples are taken from Sri Iskandar, where the geological setting is mostly sandstone interbedded with shale. The study area is easily accessible which located beside the local main road. It is covered with roughly 70% of vegetation and about 30% of the area is open outcrop with minimal amount of vegetation covering it that makes the rock easy to study. Raw materials are taken by manual hand core machine on targeted shale spot or potential location. On site location there are many potential shale spot location with less weathering effect and can be easily drilled by the handy core machine. Below is the surface map of Sri Iskandar.

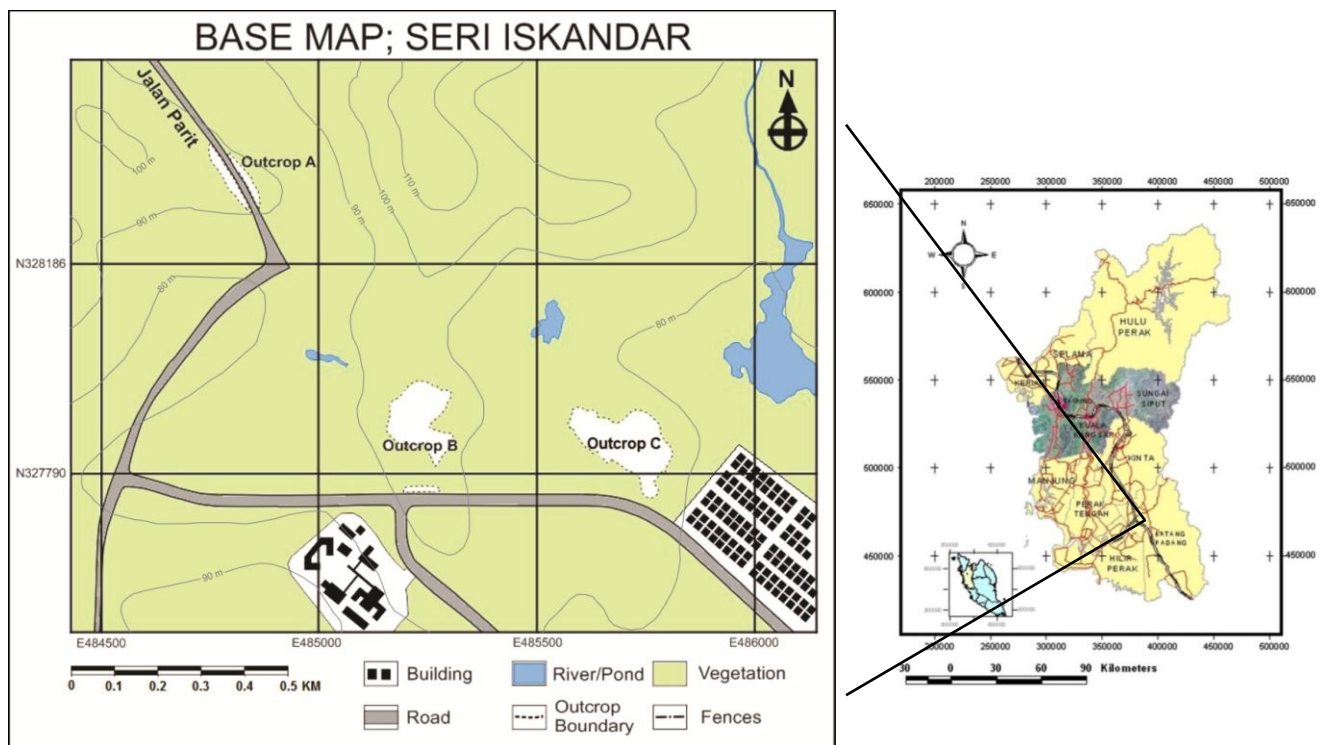


Figure 6 : Surface Map of Sri Iskandar



Figure 7 : Raw Material for Shale



Figure 8 : Coring on site for Shale

II. Core samples preparation with desired dimension

1. A raw material of shale is mould in the steel mould for making the raw material in good shape.
2. Moulded core is drilled with core machine in lab core analysis
3. Saturate the core samples with KCl to stabilize the clay content
4. Record the weight of core sample
5. Measure the porosity and permeability of each core sample



Figure 9 : Mould for core samples



Figure 10 : Shale core samples

III. List of apparatus for core sample preparation

1. Mortar mixture
2. Steel cube Mould
3. Coring Device (Hand Drill machine)



Figure 11 : Hand drill machine

Part 2 Interfacial Tension Test (IFT)

This experiment is done by using the spinning drop equipment.

1. Prepare fluid samples with different density and concentration
 - Hydraulic oil (Tellus 46) + brine solution (30000 ppm)
 - Hydraulic oil (Tellus 46) + 1% surfactant concentration (Sodium lauryl sulphate)
 - Hydraulic oil (Tellus 46) + 10% surfactant concentration (Sodium lauryl sulphate)
2. Fill the capillary with liquid with higher density
3. Inject liquid with lower density
4. Scaling of image window and calibrate the camera movement
5. Calibrate the needle in order to get the correct image and size
6. Accelerate the rotational speed to give smooth shape
7. Capture the contact image and measure the interfacial tension
8. Repeat step 2 until 7 with different sample and density

Part 3: Stress and Strain measurement

In order to proceed with the tri-axial compression test, shale core samples undergo the saturation process. Shale core samples are being saturated in vacuum chamber (Decantor unit). All core samples are left for 2 days in order to complete the saturation process. The saturation mediums are listed below:

1. Sample A : Clean Shale with brine solution
2. Sample B : Clean shale with 1% of surfactant concentration (SLS)
3. Sample C : Clean shale with 10% of surfactant concentration (SLS)

Tri-axial compression test

The procedures for conducting a tri-axial compression test are for the most part relatively standardized. The assembled sample and instrumentation fixture are installed in a pressure vessel. After this, typical procedure might include the following steps:

1. A saturated shale core sample with dimension of (55mm x 110mm) is installed between hardened steel end-caps and this assembly is sealed with a thin, deformable, heat shrink jacketing material.
2. Axial and radial strain measurement devices are mounted on the core sample in order to perform the measurement of strain.
3. Pressure vessel is filled with hydraulic fluid (Tellus 46). The confining pressure (σ_3) is raised to a nominal value (100 psi) at servo-controlled rate. The initial confining pressure is applied so that there will always be at least a small difference between confining pressure acting outside of the jacket and pore pressure in the rock (inside the jacket). Otherwise leakage will occur.
4. If additional saturation medium measures are required they are often undertaken at this time.
5. The confining pressure (σ_3) and pore pressure (Pp) are simultaneously increased at a desired controlled rate.
6. The pore pressure is maintained constant and the confining pressure is increased at a controlled rate until (σ_3) reaches a specified value.

7. The axial stress difference ($\sigma_1 - \sigma_3$) is increased at a rate corresponding to an axial strain rate of $10^{-5}/s$. Alternatively rather than controlling the axial strain rate, the axial stress rate can be controlled. Loading is continued until the sample fails. If behavior is not brittle, loading is continued so that the post-peak regime is adequately defined.
8. The sample is unloaded slowly, the pressure vessel is emptied and the sample assembly is disassembled. The sample is examined, documented and archived in a specified manner.

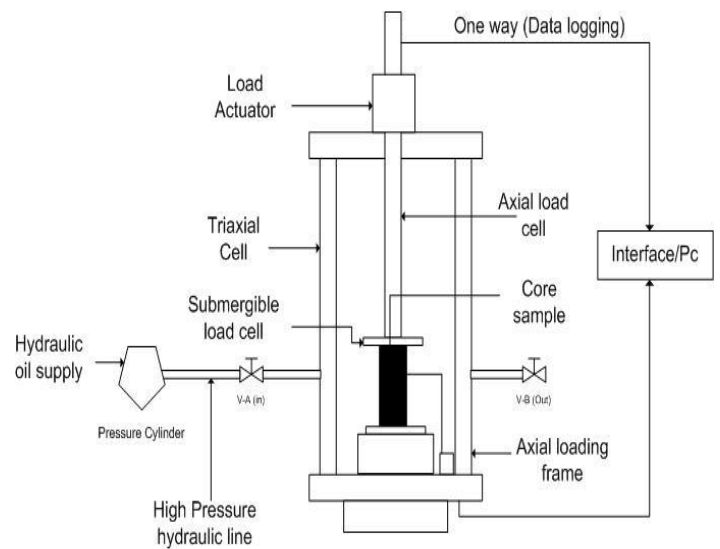
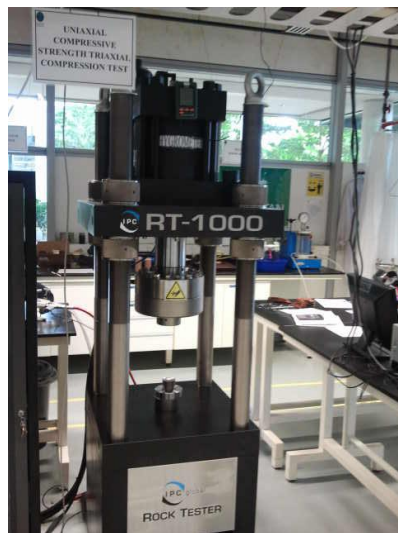


Figure 12 : Tri-axial compression test (Rock Tester)

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Preparation of core sample and general properties

The first step done for the project is to prepare the core sample and test in the tri-axial equipment for the stress and strain measurement. General properties of core sample are as follows:

Table 2 : General Properties of core sample

General Properties of core sample	
Type of rocks (Raw material)	Shale
Weight (g)	44.8
Diameter (mm)	55
Length (mm)	110
Average porosity	0.22
Average permeability (md)	0.15

4.2 Interfacial Tension Test Result (IFT)

This test is to measure the interfacial tension between the hydraulic oil with the different concentration of surfactant. Three different of samples are prepared and the details of samples are as follow:

Table 3 : IFT different fluid properties

Properties	Hydraulic oil (Tellus 46)	Brine solution, 30000ppm	1% concentration of surfactant (Sodium lauryl sulphate)	10% concentration of surfactant (sodium lauryl sulphate)
Density g/ cm ³	0.879	1.273	1.278	1.274
Refractometer Index (RI)	-	1.33547	1.33826	1.33448

4.3 Interfacial Test result (IFT)

The interfacial forces either take from forces on the interface between two fluids or the fluid and solid matrix of the rock. Both effect rely upon difference in the relative strength of inter molecular of fluids. Interfacial tension is one of the important criteria in designing the hydraulic fracturing technique. The ability of fracture fluid to give the significant impact on the rock formation is also depending on the interfacial tension. Once the interfacial tension of the fluids reduce will help the fracture fluid to penetrate into the formation. In this experiment, the fracture fluid used is Tellus 46 and the additive agent is Sodium lauryl sulphate as the surfactant. The interfacial tension is tested between Tellus 46 and different concentration of Sodium lauryl sulphate from 1% and 10% concentration. The interfacial tension is done by using the spinning drop test.

In this test a small drop is placed in a denser liquid enclosed in a glass tube in which normally brine solution and surfactant solution are filled. The glass tube is subjected to rotation at high angular velocity about its horizontal axis. The method is based upon the principle of gyrostatic equilibrium, which is the state of uniform rotation in which every bit of the fluid inside the tube is at rest with respect to the wall of tube. Gyrostatic equilibrium is achieved at high angular velocities when the gravitational force perpendicular to the axis of rotation is negligible as compared with the centrifugal force. When the tube rotates with high velocity, the drop migrates which is from the hydraulic oil to the axis of rotation and assume a cylindrical shape with hemispherical ends. For each angular velocity, the drop comes to an equilibrium shape which is characteristic of that velocity.

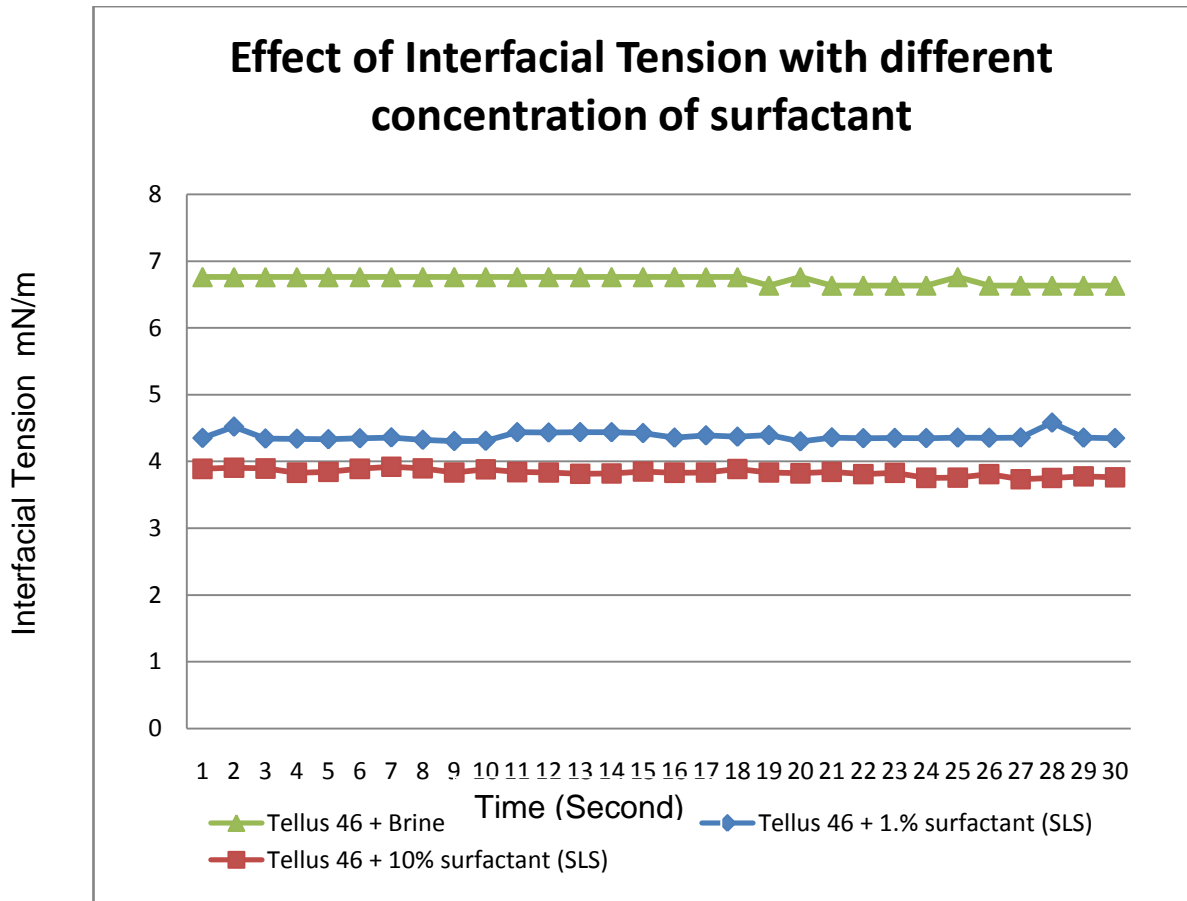
At low rotational velocities, the fluid drop will take on the ellipsoidal shape, but when rotational velocity is sufficient large it will become cylindrical. Under this latter condition the radius of cylindrical drop is determined by the interfacial tension. Take also the consideration of the density difference between the drop with the surrounding fluid and the rotational velocity of the drop. The spinning method has been very successful in examination of ultralow interfacial tension down to 10^{-6} mN/m.

The result gained from this experiment is the measurement of interfacial tension between the hydraulic oil with different concentration of surfactant for the shale core sample. The measurement reflected the interaction of different fluid properties when come into contact. The table 6 shows the overall results of the calculated interfacial tension from this experiment.

Num of Run (Second)	Fluid interfacial tension with hydraulic oil (mN/m)		
	Brine Solution	1% surfactant concentration (SLS)	10% surfactant concentration
1	6.764	4.351	3.888
2	6.763	4.52	3.906
3	6.763	4.342	3.893
4	6.763	4.339	3.83
5	6.763	4.332	3.844
6	6.764	4.346	3.888
7	6.763	4.359	3.919
8	6.764	4.323	3.897
9	6.764	4.305	3.833

Table 4 : Calculated Interfacial Tension for different fluid properties

The results show in table 6 are plotted into graph that depicts the calculated interfacial tension against the number of run and the relation changing different type and concentration of fluids towards the hydraulic oil. Below is the result from the spinning drop test to measure the interfacial tension of the fluids.



Constant value of average interfacial tension for hydraulic oil (Tellus 46) with brine solution at **6.721 mN/m**. This shows that the interaction of hydraulic oil for the brine solution indicates the highest surface tension compared to other fluids. However the average value of interfacial tension between hydraulic oil (Tallus 46) and 1% concentration of surfactant (sodium lauryl sulphate) is **4.355 mN/m** and has reduced to 35% from previous interfacial tension. The promising result for interfacial tension is showed by the interaction of hydraulic oil (Tallus 46) with the 10% concentration of surfactant (Sodium lauryl sulphate) which is **3.807 mN/m** and which mean the interfacial tension has reduced to 50% from the original value.

4.4 Tri-axial compression test (Rock tester)

In this experiment failure rate and strength of rock sample are being tested in designing the hydraulic fracturing technique. Experiment results are represented as stress strain curves and tabulated value of elastic constant of strength. In this tabulated value of elastic constant of strength the value of fracture pressure required will be determined experimentally. After the exact concentration of surfactant (Sodium Lauryl Sulphate) in 10% concentration has the ability to reduce 50% of the interfacial tension of the interaction with the hydraulic oil, the surfactant is brought to be tested in tri-axial compression in order to give enhancement for hydraulic fracturing technique by reducing the required fracture pressure. In this experiment two test were conducted by testing shale core sample with injection of hydraulic oil (Tellus 46) without surfactant and injection of hydraulic oil (Tellus 46) with the 10% concentration of surfactant (Sodium Lauryl sulphate).

For a typical stress-strain curve showing axial and radial strain as a function of the axial stress difference. In a brittle or elastic-perfectly or strain softening materials, confined compressive strength at the confining pressure used in a tri-axial test is taken as the maximum effective axial stress accommodated by the sample. Below is the result obtained from the conducted tri-axial test:

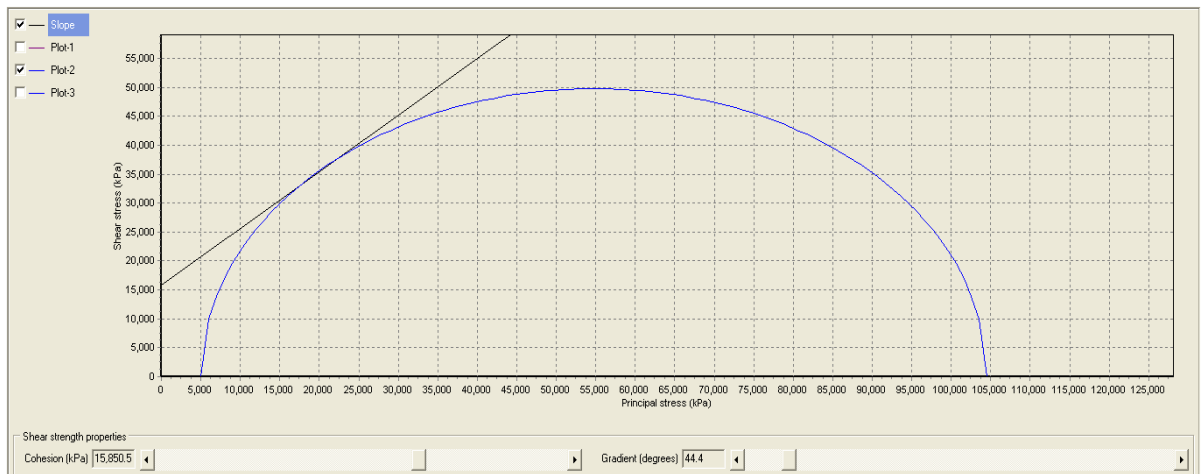


Figure 13 : Stress-Strain curve (Hydraulic oil injection without surfactant-SLS)

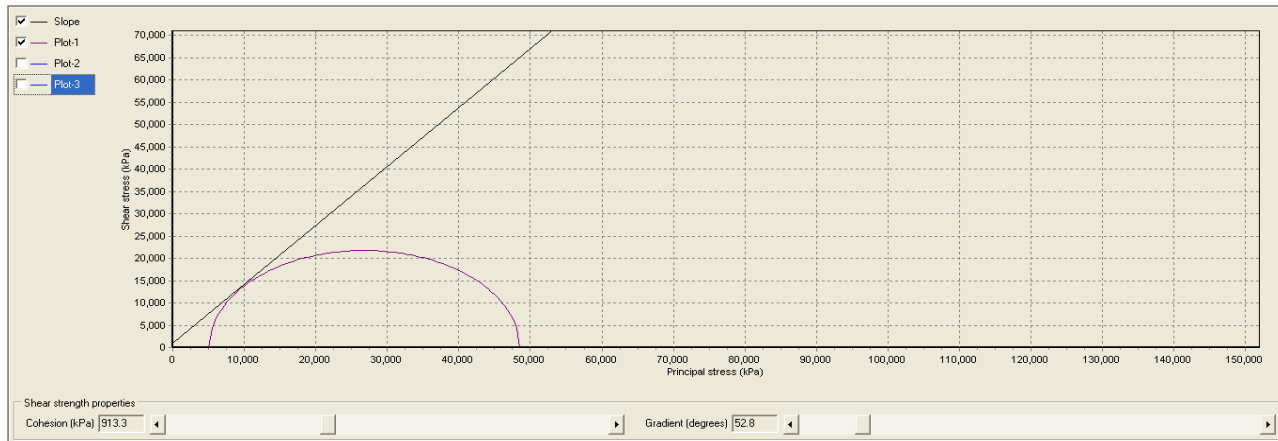


Figure 14 : Stress-Strain curve (Hydraulic oil injection with 10% concentration of surfactant-SLS)

Result Criteria	Hydraulic oil (Tellus 46) injection without surfactant-Figure 26	Hydraulic oil (Tellus 46) injection with 10% concentration of surfactant (SLS)-Figure 27
Confine Pressure (σ_3) kPa	5029	5042
Axial Pressure (σ_1) kPa	104569	48534
Shear Pressure kPa	13174.9	913.3
($\sigma_1 + \sigma_3$) / 2 kPa	49770	21746

Table 5 : Result for the Stress and Strain Curve

This simplest representation is known as Coulomb failure envelope. This failure locus is a best fit tangent to Mohr's circle, constructed from multiple tri-axial compressions. Mohr's circles are plotted using the effective axial and confining pressure at failure as the relevant major and minor principle stresses. In this experiment, only one confined pressure is being compared to determine the Mohr's circle. From the result obtained, injection of hydraulic oil (Tellus 46) with 10% concentration of surfactant (SLS) has the ability to reduce the axial pressure from **104569 kPa** to **48534 kPa**. Besides the shear pressure also decreases from **13174.9 kPa** to **913.3 kPa**. This shows that the surfactant used is helping in increasing the failure rate and has the significant effect in changing the distribution of the effective strain and strain pressure. This experiment also will show the ability of the surfactant to reduce the anticipated stress and strain pressure or helps the fracture fluid to break the formation easily.

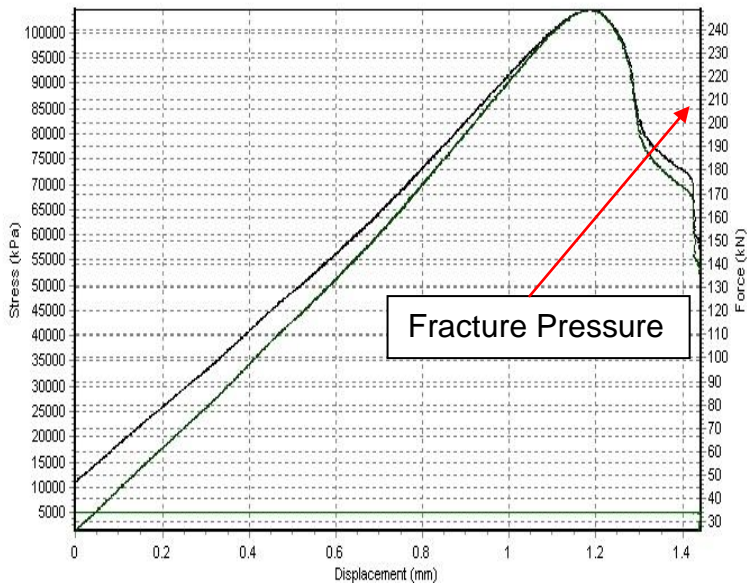


Figure 15 : Stress -Failure rate for hydraulic oil injection without surfactant

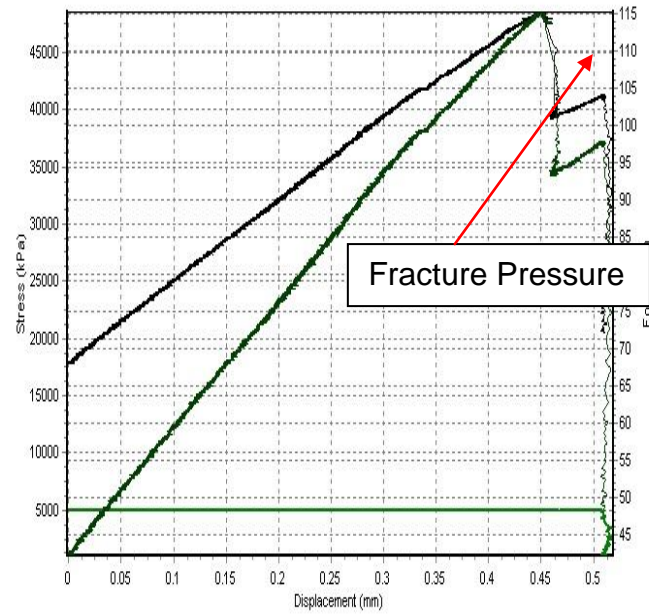


Figure 16 : Stress-Failure rate for hydraulic oil injection with 10% concentration of surfactant

Result Criteria	Hydraulic oil (Tellus 46) injection without surfactant-Figure 28	Hydraulic oil (Tellus 46) injection with 10% concentration of surfactant (SLS)-Figure 29
Confined Pressure (kPa)	5029	5042
Maximum axial force (kN)	248.440	115.310
Minimum axial force (kN)	42.240	26.188
Fracture Pressure (kN)	210	110

From the result in triaxial compression test, Stress-Failure graph shows the estimated value for fracture pressure after the shale rock sample has been injected by two different medium from hydraulic oil without surfactant and hydraulic oil with 10% concentration of surfactant (SLS). By injecting hydraulic oil with 10% concentration of surfactant (SLS) the fracture pressure has reduced from **210 kN** to **110 kN**. Which means shale core sample is easily fracture when the additive is applied in the injected hydraulic oil.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

In conclusion, based on the study of using surfactant in hydraulic fluid in optimizing the hydraulic fracturing technique in shale gas formation should give a significant effect in determining the effective stress and stress of the formation. This is highly desired in hydraulic technique in order to optimize well performance after the treatment. The main characteristics of surfactant in hydraulic fluid that make it desired choice is its compatibility fluid with formation and its ability to reduce the surface tension and capillary pressure inside the rock formation. As the main objective to investigate the effect of the surfactant on improving the stress –strain pressure distribution of shale formation and ability in reducing the surface tension has been proved experimentally to justify the hypothesis. These are the results obtained after surfactant is applied for improving the hydraulic fracturing technique for shale gas formation.

1. Average interfacial tension for hydraulic oil without surfactant = 6.721 mN/m
2. Average interfacial tension for hydraulic oil with surfactant= 3.801 mN/m
(reduced 50% from original value)
3. Axial pressure reduce from 104569 kPa to 48534 kPa
4. Shear pressure also decreases from 13174.9 kPa to 913.3 kPa
5. Fracture pressure has reduced from 210 kN to 110 kN.

However few recommendation or improvement should be made especially in getting the core sample for the project. Core sample should be taken from hydraulic fractured field which originated from shale environment. Besides, used of micro-emulsion surfactant for an alternative additive in order to replace the usage of the conventional surfactant. As for now in our field region, shale gas formation is not widely explored which make the limitation in study the project. Experiment should be maintained in a controlled environment which means that other related factor should be turned constant. The continuation of study in this field is important especially for the unconventional drilling due to increasing in demand for hiring new researcher or expertise. This kind of exposure will help for the future engineer to have a better understanding in this field.

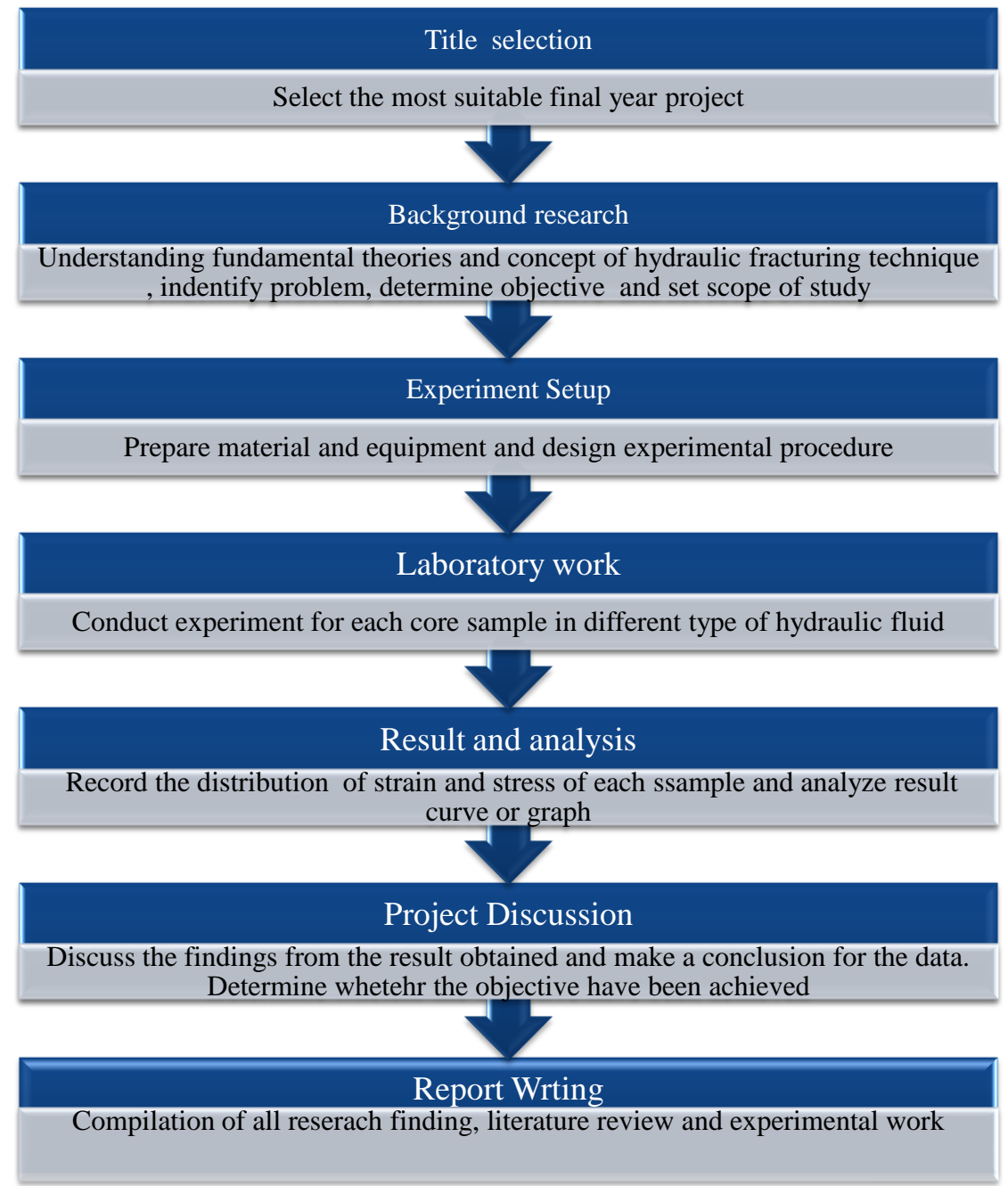
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APPENDICES

Appendix i: Project Planning

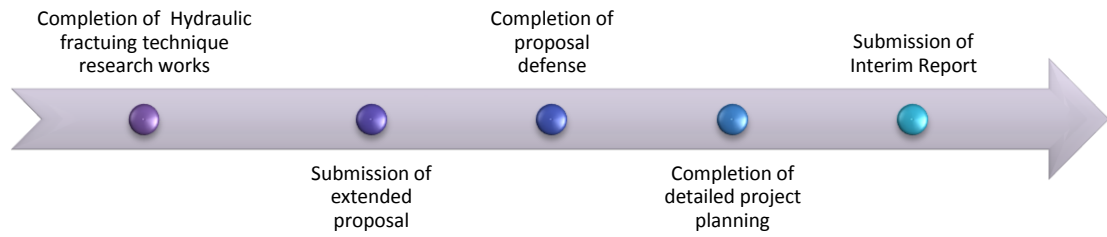


Appendix ii: Project Gantt chart for Final Year project 1 and 2

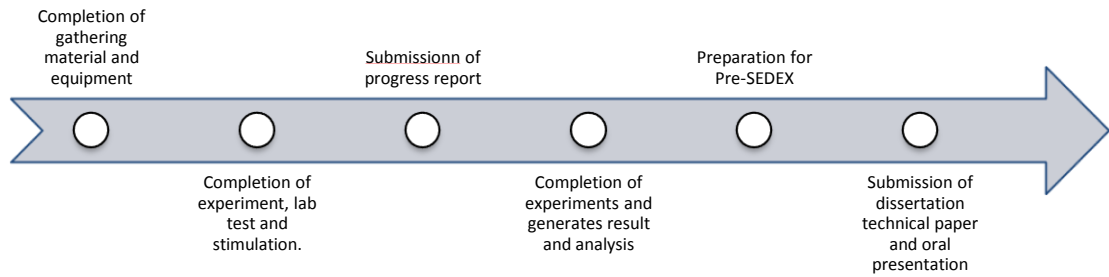
Name : <u>Mohd Ridzuan Bin Hamid</u> Student ID :12688 Final Year Project I & II																													
Petroleum Engineering																													
Project Title : Comparative studies on the effect of surfactants used in optimizing hydraulic fracturing technique towards alteration of shale gas formation																													
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								█	█																				
Interim Submission										█	█	█	█																
Gathering Material and Equipment																█	█	█											
Completion of experiment ,lab test and stimulation																			█	█	█	█							
Documentation Project																				█	█	█	█	█	█	█	█	█	

Figure 17 : Final Year Project Gantt chart

Appendix iii: Key Milestone Final Year Project 1 and 2



Final Year Project 1



Final Year Project 2

Figure 18 : Final Year Project Key Milestone

Appendix IV: Interfacial Test Result (IFT)

- i. First run between hydraulic oil (Tellus 46) with the brine solution (30000 ppm).

Run-No (Second)	Interfacial Tension [mN/m]
1	6.764
2	6.763
3	6.763
4	6.763
5	6.763
6	6.764
7	6.763
8	6.764
9	6.764
10	6.764
11	6.764
12	6.764
13	6.763
14	6.764
15	6.764
16	6.764
17	6.763
18	6.763
19	6.637
20	6.764
21	6.636
22	6.637
23	6.636
24	6.637
25	6.764
26	6.637
27	6.637
28	6.637
29	6.636
30	6.636

Table 6 : IFT hydraulic oil + Brine solution

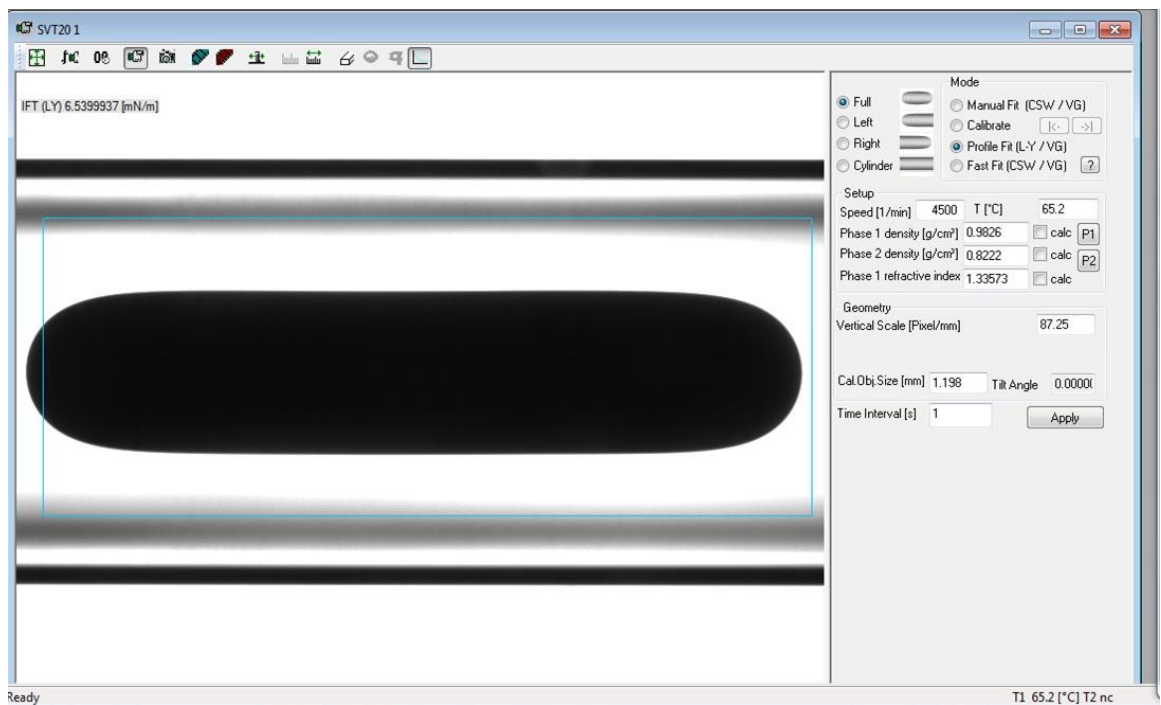
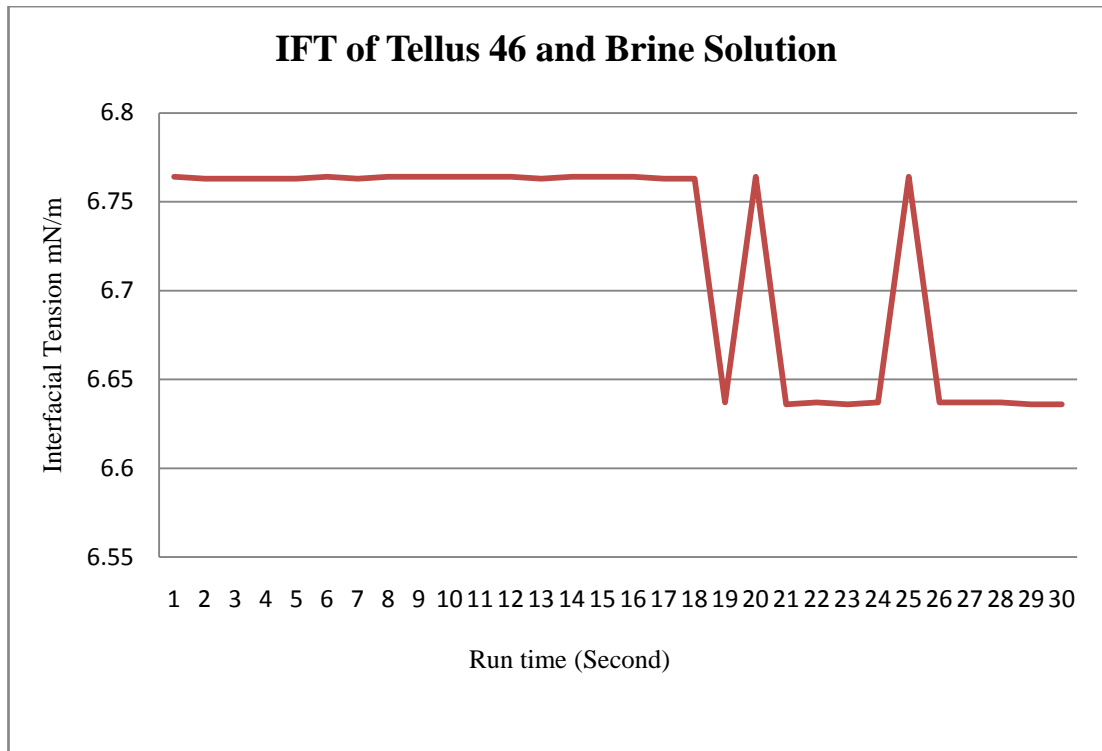


Figure 19: Interfacial Tension of Tellus 46 with brine solution

- ii. Second run between hydraulic oil (Tellus 46) and 1% concentration of surfactant (Sodium Lauryl Sulphate)

Run No (Second)	Interfacial Tension mN/m
1	4.351
2	4.52
3	4.342
4	4.339
5	4.332
6	4.346
7	4.359
8	4.323
9	4.305
10	4.308
11	4.437
12	4.432
13	4.438
14	4.437
15	4.424
16	4.356
17	4.39
18	4.37
19	4.394
20	4.3
21	4.359
22	4.347
23	4.351
24	4.349
25	4.356
26	4.353
27	4.358
28	4.58
29	4.355
30	4.349

Table 7 : IFT Tellus 46 + 1% concentration of surfactant (SLS)

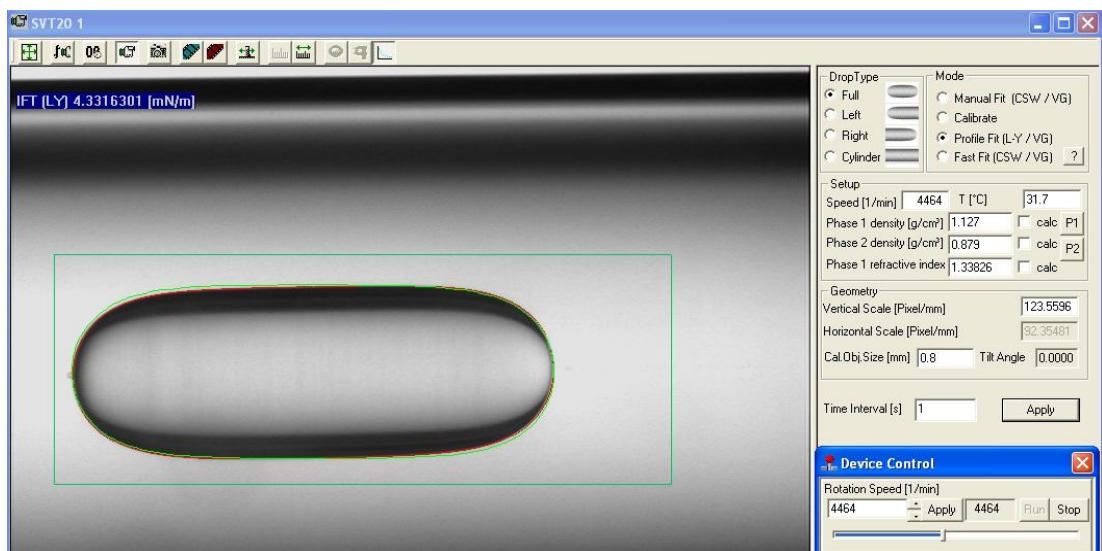
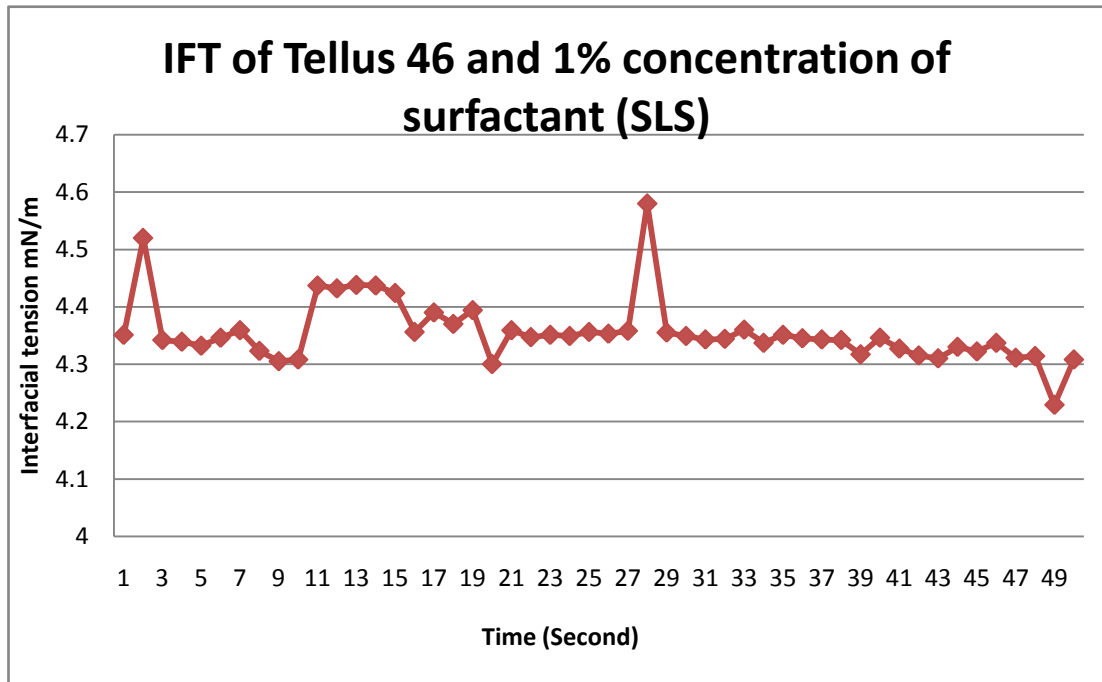


Figure 20: Interfacial Tension between Tellus 46 + 1% concentration surfactant (SLS)

- iii. Third run between hydraulic oil (Tellus 46) and 10% concentration of surfactant (Sodium Lauryl Sulphate)

Run No (Second)	Interfacial Tension mN/m
1	3.888
2	3.906
3	3.893
4	3.83
5	3.844
6	3.888
7	3.919
8	3.897
9	3.833
10	3.882
11	3.84
12	3.833
13	3.813
14	3.819
15	3.849
16	3.83
17	3.834
18	3.886
19	3.831
20	3.821
21	3.844
22	3.809
23	3.828
24	3.753
25	3.754
26	3.81
27	3.732
28	3.752
29	3.776
30	3.76

Table 8 : IFT Tellus 46 + 10% concentration of surfactant (SLS)

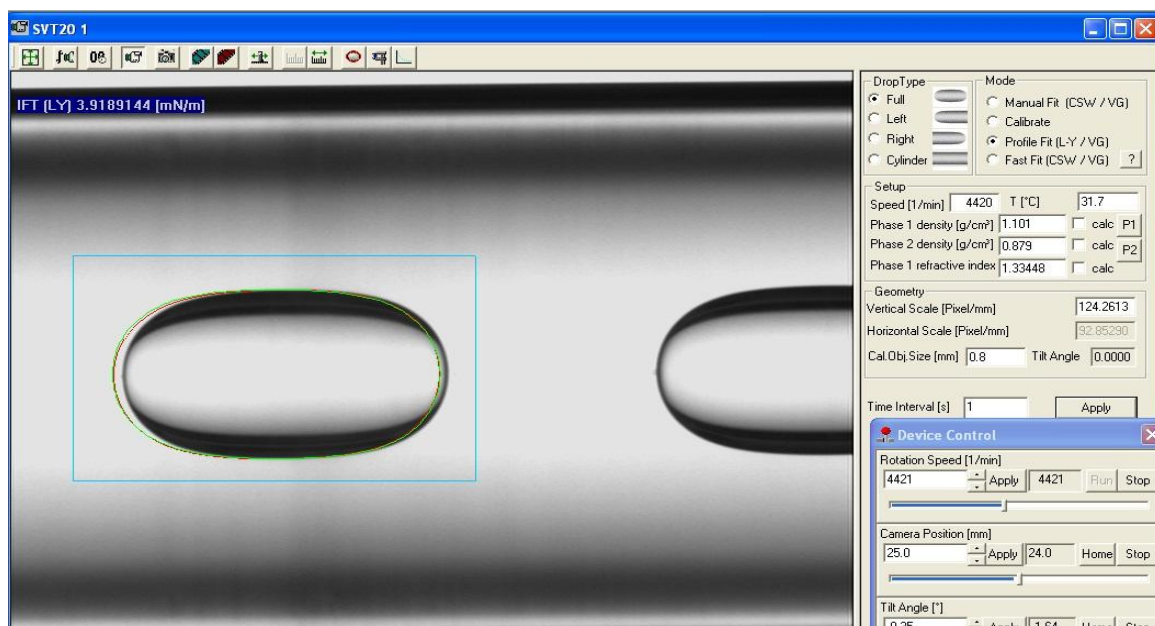
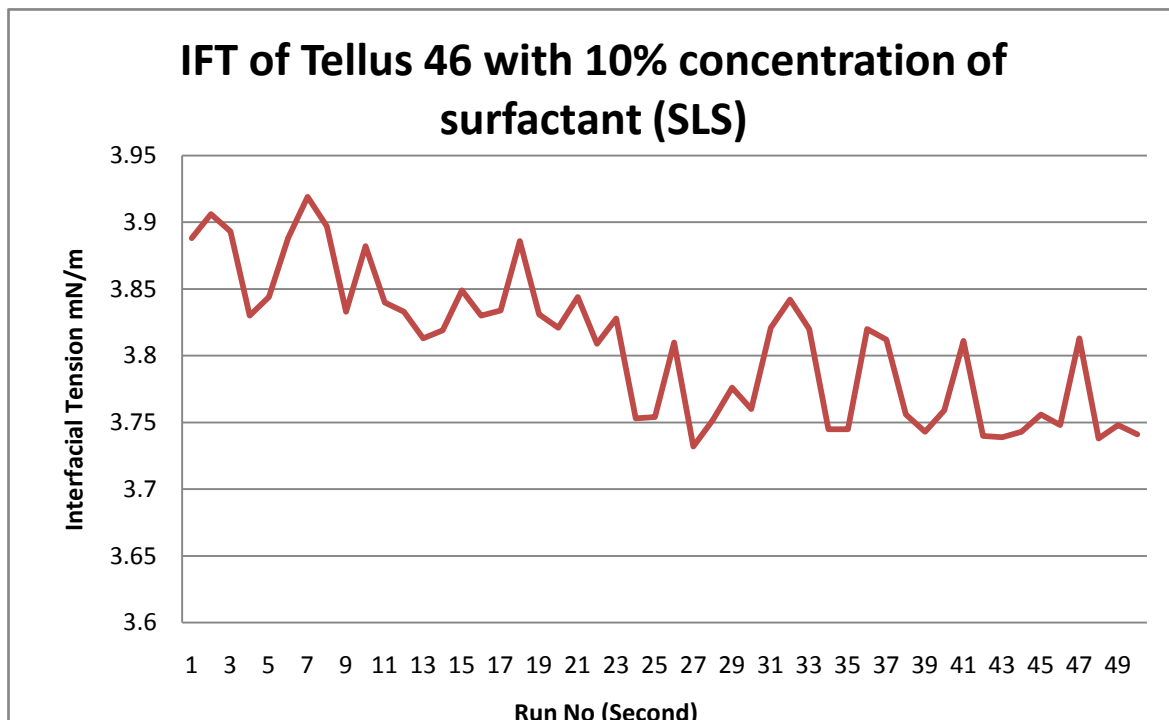


Figure 21 : Interfacial Tension between Tellus 46 + 10% concentration of surfactant (SLS)

Appendix V: Tri-axial Test Results

First tri-axial test- Injecting hydraulic oil (Tellus 46) without any additive

Run 1	
Maximum axial force (kN)	161790
Minimum axial force (kN)	8711
Confining stress (kPa)	3756.700
Fracture Pressure (kPa)	120

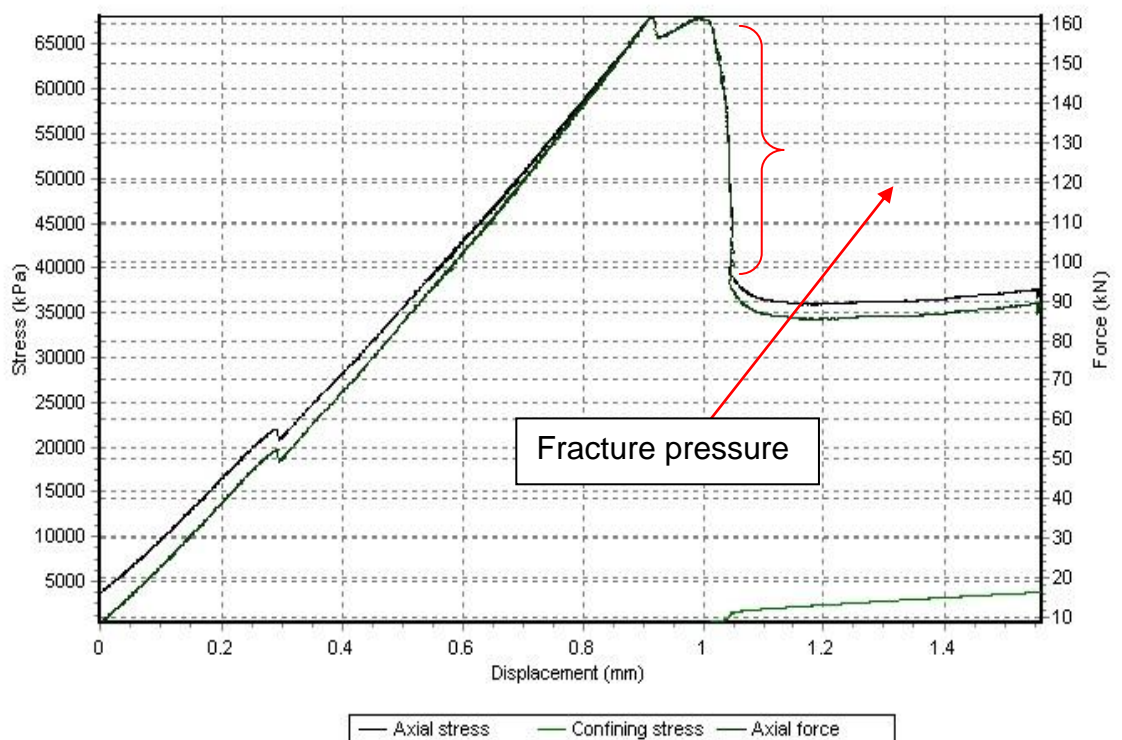


Figure 22 : Run 1- Tellus 46 injection of without additive in shale core sample

Run 2	
Maximum axial force (kN)	248.440
Minimum axial force (kN)	261.188
Confining stress (kPa)	5029
Fracture Pressure (kPa)	210

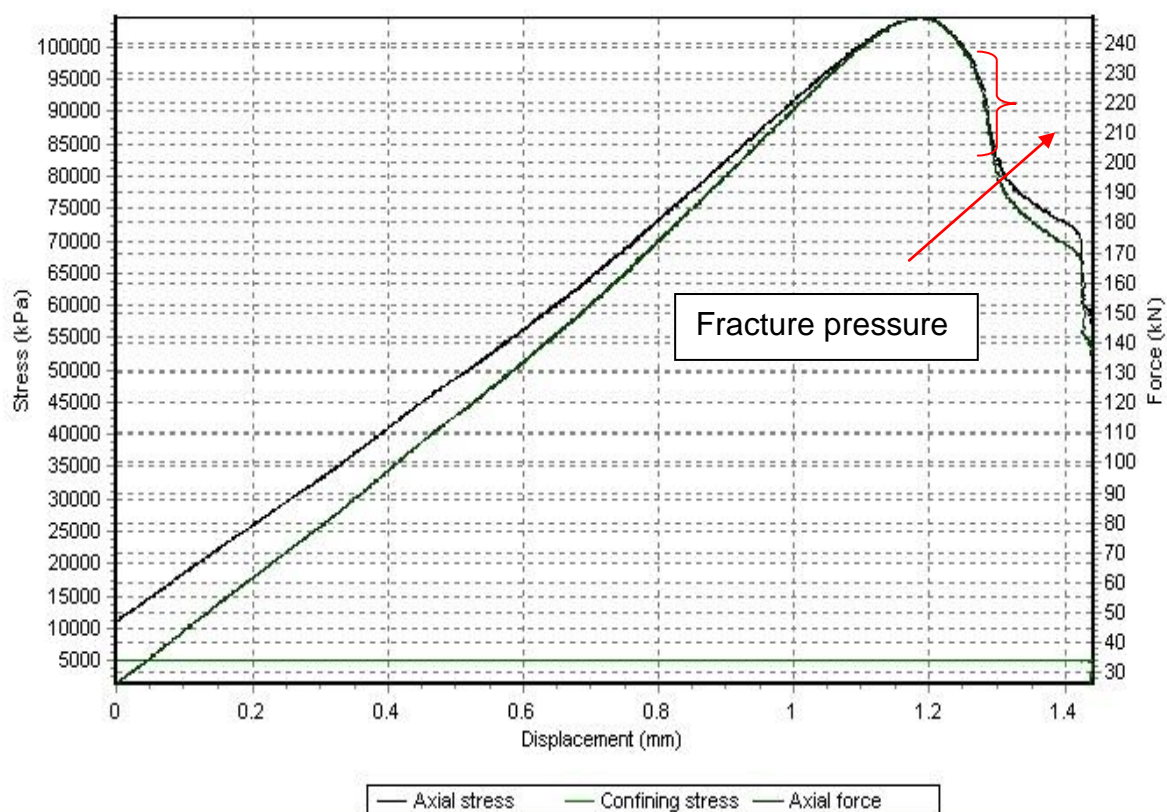


Figure 23 : Run 2- Tellus 46 injection without additive in shale core sample

Run 3	
Maximum axial force (kN)	304.410
Minimum axial force (kN)	110.570
Confining stress (kPa)	720.840
Fracture Pressure (kPa)	280

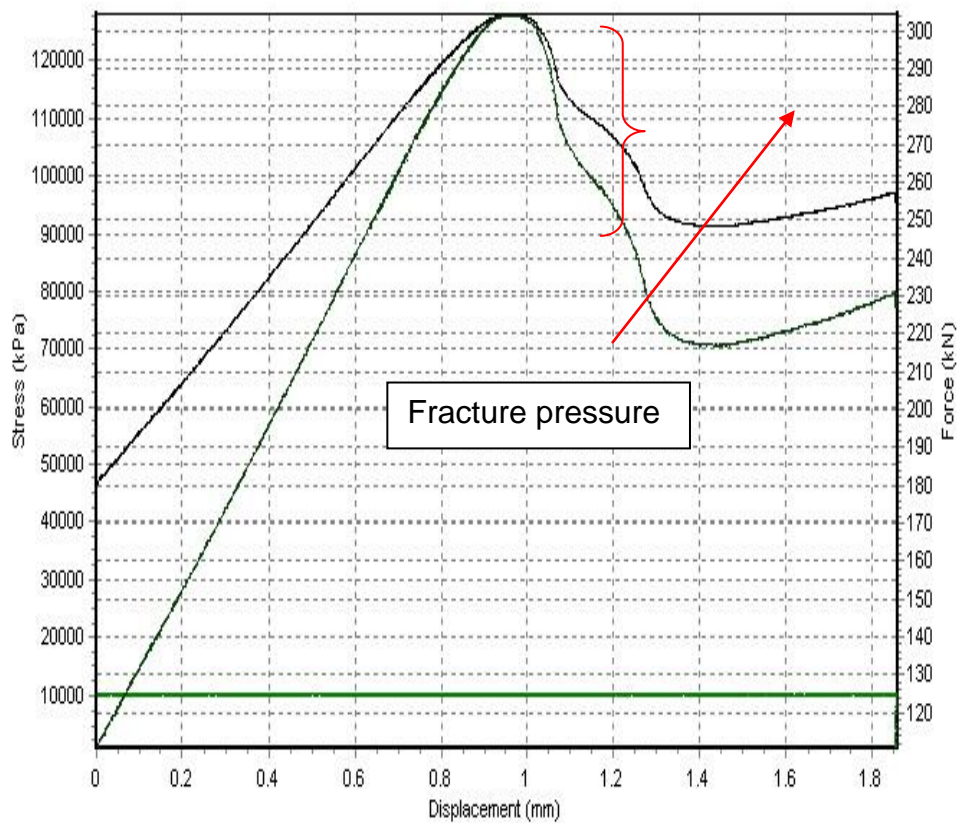


Figure 24 : Run 3- Tellus 46 Injection without additive in shale core sample

Mohr analysis for all runs by injecting hydraulic oil (Tellus 46) without any additive in shale core sample

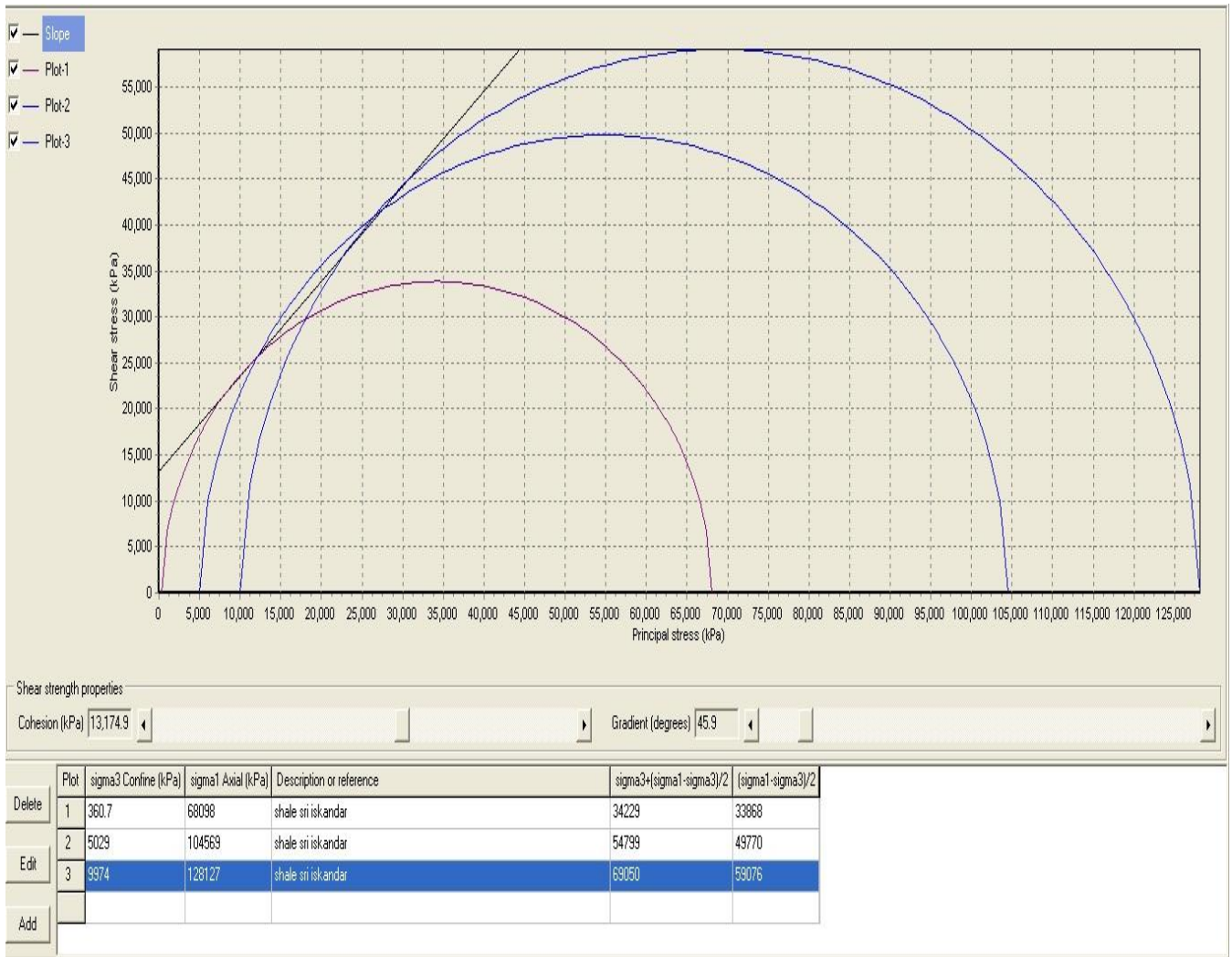


Figure 25 : Mohr analysis for Tellus 46 injection without additive in shale core sample

Second Tri-axial test- Injecting hydraulic oil (Tellus 46) with 10% concentration of surfactant (Sodium lauryl sulphate)

:

Run 1	
Maximum axial force (kN)	115.310
Minimum axial force (kN)	42.240
Confining stress (kPa)	5042
Fracture Pressure (kPa)	110

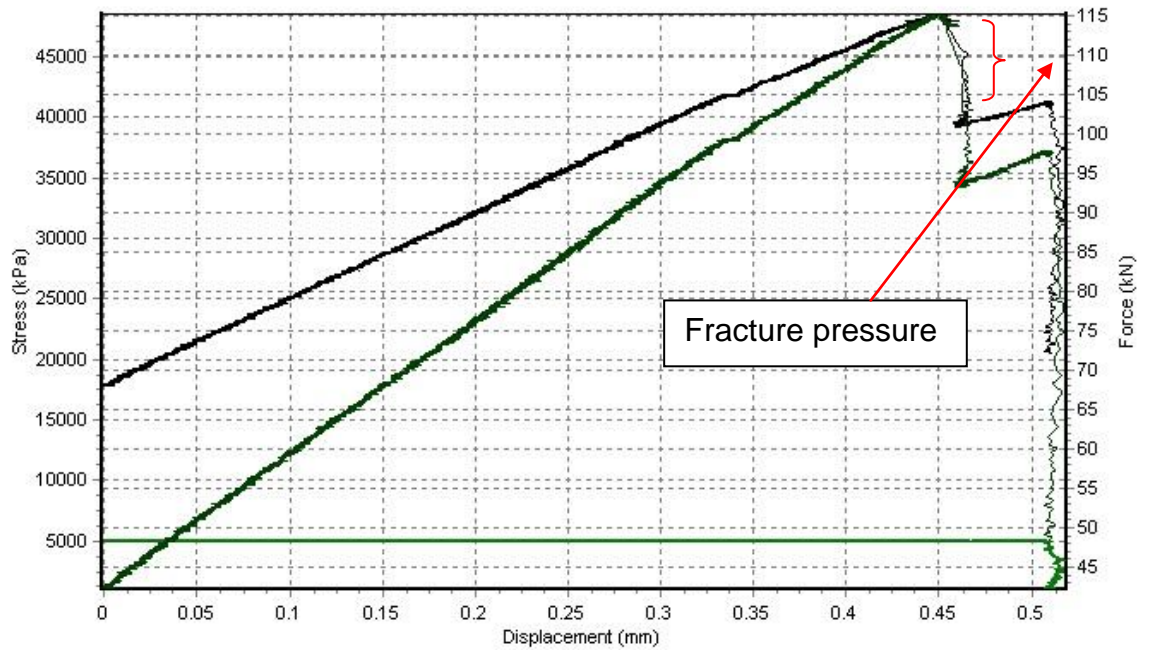


Figure 26 : Run 1-Injecting hydraulic oil (Tellus 46) with 10% concentration of surfactant (Sodium lauryl sulphate) in shale core sample

Run 2	
Maximum axial force (kN)	360.980
Minimum axial force (kN)	2.441
Confining stress (kPa)	3686.100
Fracture Pressure	300

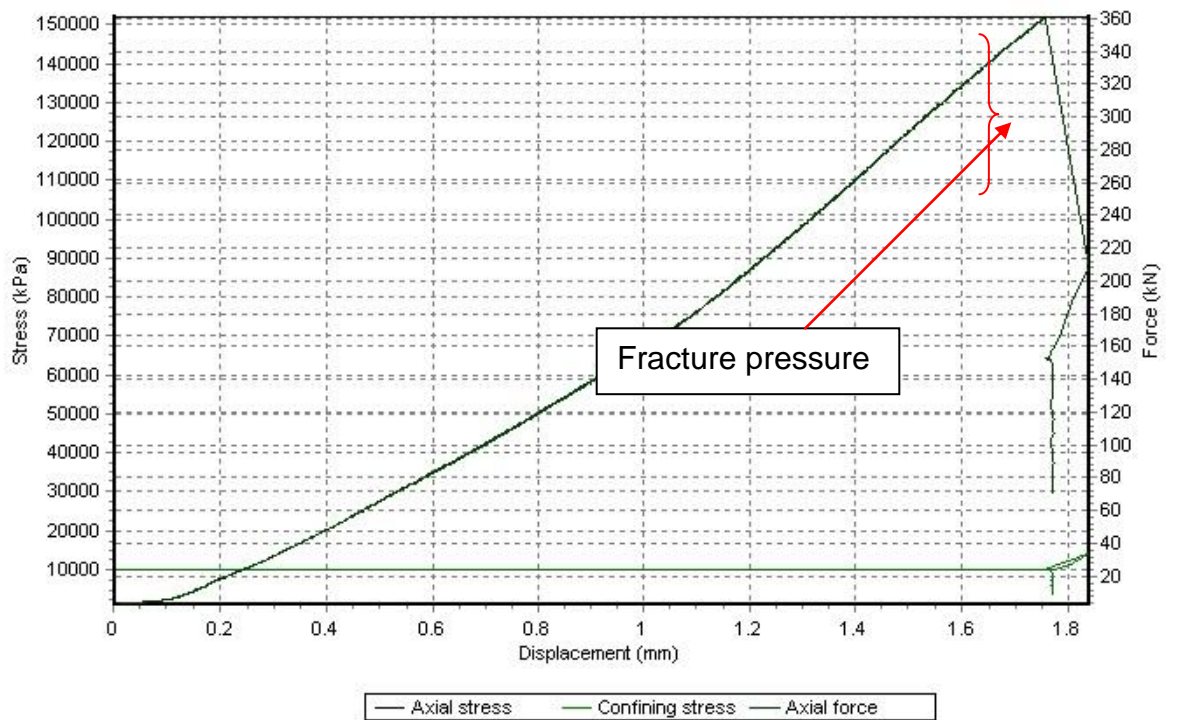


Figure 27 :Run 2- Injecting hydraulic oil (Tellus 46) with 10% concentration of surfactant (Sodium lauryl sulphate) in shale core sample

Run 3	
Maximum axial force (kN)	351970
Minimum axial force (kN)	1.551
Confining stress (kPa)	6982.100
Fracture Pressure (kPa)	

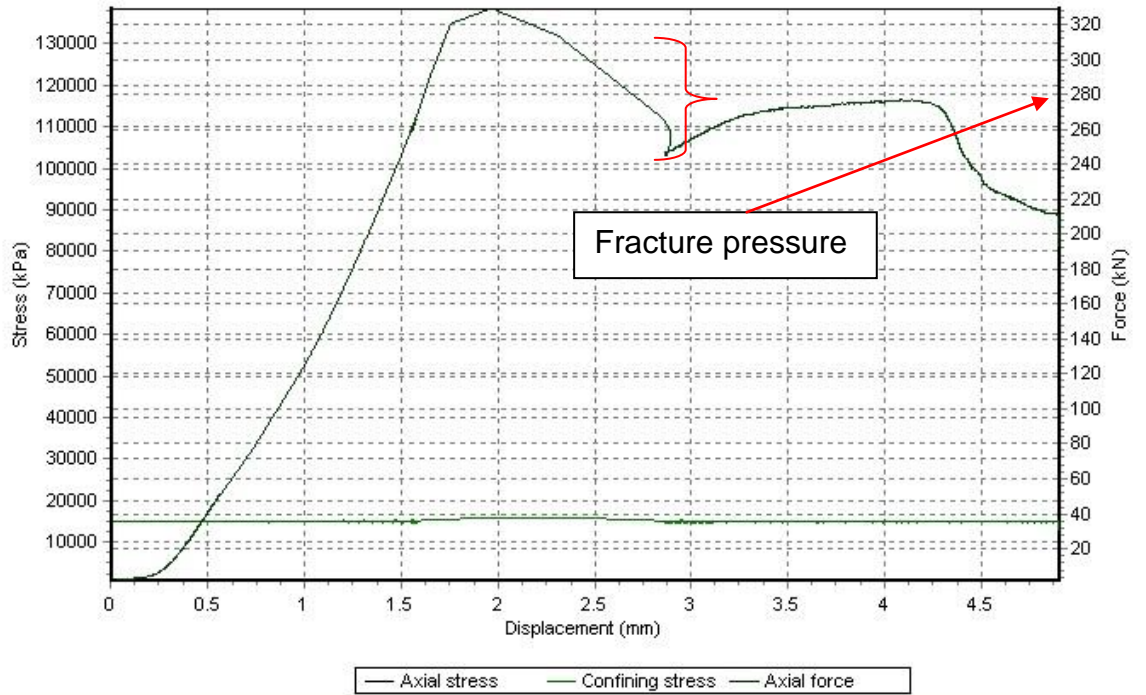


Figure 28 :Run 3- Injecting hydraulic oil (Tellus 46) with 10% concentration of surfactant (Sodium lauryl sulphate) in shale core sample

Mohr analysis for all runs by injecting hydraulic oil (Tellus 46) with 10% concentration of surfactant (Sodium lauryl sulphate) in shale core sample

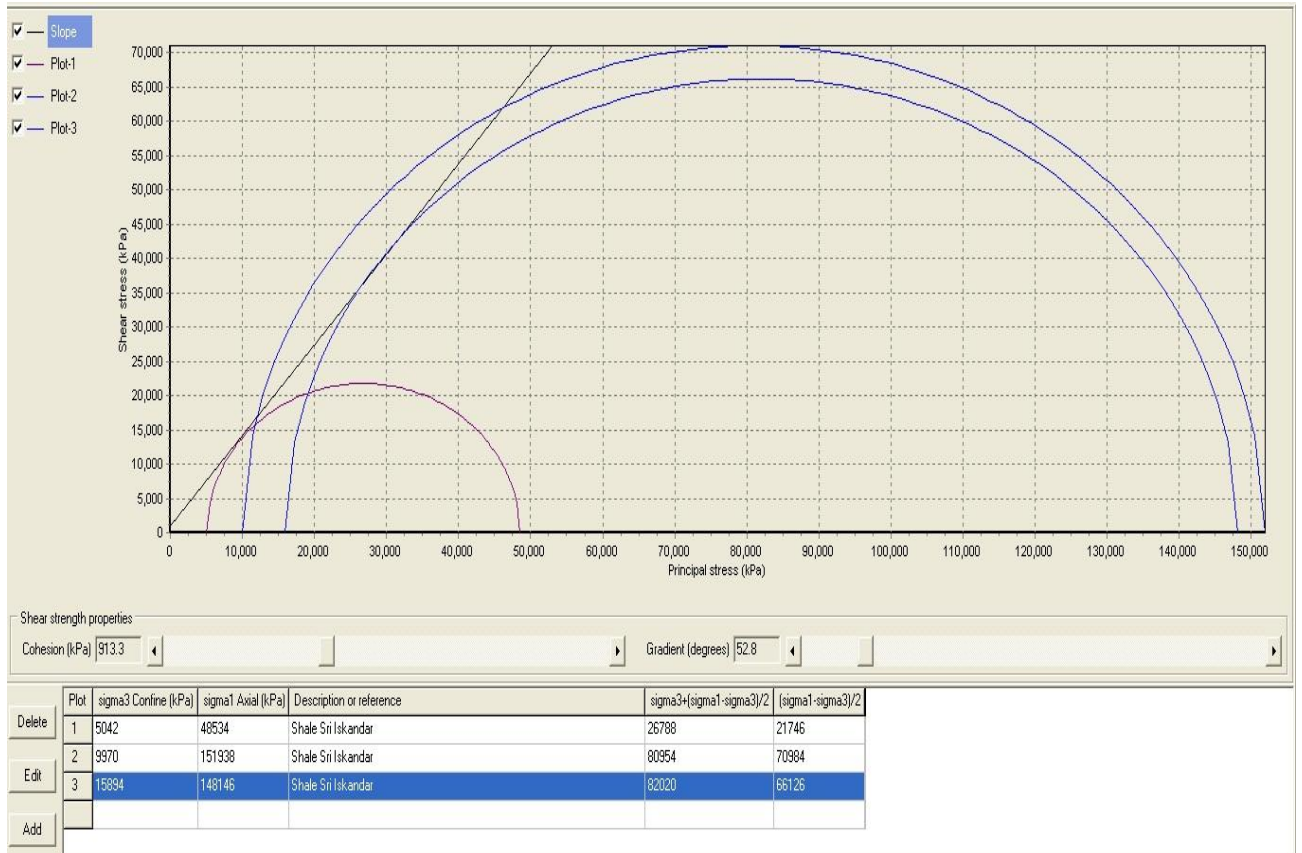


Figure 29 : Mohr analysis for Injection of Tellus 46 + 10% concentration of surfactant (SLS) in shale core sample

Appendix VI: Technical Paper