Percolation Effects in Electrical Resistivity of Fine-grained Rock Material

by

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Project Dissertation submitted in partial fulfilment of the requirements for the Bachelor of Engineering (Hons) (Petroleum) JAN 2015

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CERTIFICATION OF APPROVAL

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

Approved by,

(Dr. Shiferaw Regassa Jufar)

UNIVERSITI TEKNOLOGI PETRONAS

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Jan 2015

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.

GOO MEI SAN

ABSTRACT

Percolation is a probabilistic phenomenon used in studying the system with phase transition. This concept is most widely used in studying semiconductor where the critical concentration of conductive filler is determined in order to start an electrical flow. In this study, percolation phenomena is used to study the effect of concentration of fine grained conductive rock materials (in this case, clay is used to represent the fine grained rock material) on the overall electrical conductivity of the system. As the critical concentration is determined, further increase in the concentration is done to study the trend afterwards. Besides the concentration of fine grained conductive particles, the amount of water salinity on the system's conductivity is also investigated using the same approach. The numerical simulation is designed to input user defined concentration of conductive particles, after that it will distribute the conductive particles into the system randomly using Monte Carlo method according to the concentration assigned. We will then check if there is any continuous path exist throughout the system for it to percolate. Laboratory work is done to match with the simulation results, by using synthetic cores made from sand and fine grained shale. The core is prepared by mixing sand and clay material in a fixed proportion, with sodium silicate solution as the binding agent. After that, the cores will be saturated with brine with different saturation and the effects of both the factors of clay concentration and water salinity on the core resistivity will be investigated. The results clearly show the decrement of core resistivity as the clay content increases, and when the water salinity increases as well. The effect of clay on resistivity becomes less obvious as the clay content continue to increase. In the case for clean sand core, water salinity gives the most obvious impact on resistivity values compared with those core samples that contain clay. The higher the water salinity, the lower the resistivity values measured.

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CHAPTER 1

INTRODUCTION

1.1 Background

Extraction of oil and gas resources from shale formation has received a lot of attention especially during these two decades. This is mainly due to the depletion of conventional resources and the discovery of the potential to recover from the shale formation. However, the production of oil and gas from shale formation remains a challenge until today mainly due to the high fracture treatment pressure required for the low permeability shale formation (Integrated Solutions for Unconventional Applications, 2003).

Due to the cation exchangeable property of shale, it can conduct electricity and in turns reduce the reading for resistivity log, which as a result lead to the overestimation of water saturation and porosity. It's also important to note that Archie's equation does not account for the effect of shale (ChengBing, 2004). In order to correct for the shale effect, different types of models have been developed by researchers to interpret the data for this type of formation, which includes the dual electric layer model. In ChengBing's research, they successfully introduced a new form of equation which does not rely on the need of assuming uniform porosity across different layers. They also propose the method of high-resolution measurement to better analyse shaly formation.

It is also important to note that different distribution patterns of shale in the sandstone formation will affect the reading of resistivity in a different way even though there is same volume of shale present (IHRDC, 2014). Typical types of shaly sandstones distribution include laminar shale, structural shale and dispersed shale. These distributions are further illustrated in the figure below:



Figure 1 Different Shale Distribution (http://www.ihrdc.com/els/ipimsdemo/t26/offline_IPIMS_s23560/resources/data/G4105.htm)

As shown in the Figure 1, laminar shale describes the shaly sandstones formation where the shale are in the form of thin layers, sandwiched between sandstones. On the other hand, structural shale is a type of distribution where the shale become part of the rock matrix and is randomly distributed in the sandstones formation. Last but not least, the dispersed shale are shale that accumulate in between the open space of the sandstone matrix.

Percolation theory is a probabilistic approach most commonly used to describe a system exhibiting phase changes. For instance, in the study for semiconductor, when the amount of the conductive filler exceeds certain threshold value, electricity start to flow. This condition has some similarity with the shale formation system where shale acts as the conductive filler while the sandstones acts as the non-conductive matrix. If the threshold value for shale content to starts electricity flow in the formation is known, its effect can be implemented and corrected during the interpretation of log data.

1.2 Problem Statement

Since the presence of clay minerals in reservoir rocks affects the resistivity log reading, which in turn leads to the overestimation of water saturation and porosity, its effect must be studied in order for correction to be made when dealing with this resistivity log reading. In this research, the critical concentration of clay material for electrical conductivity to occur is determined and further increment in the clay material is done to study its impact.

Besides the effect of clay content, the effect of salinity also affect the resistivity reading log, where high salinity brine will give a lower resistivity reading. Thus, the effect of water salinity is also studied in the research in order to further confirm the trend of resistivity reading with respect to clay content. For instance, the trend of relationship between clay content and resistivity should be similar for all different types of salinity, even though the values might be different from each other.

Throughout the progress of this research, further insight will be given to determine the critical composition of fine-grained rock (clay) in order to initiate an electrical conductivity. In addition, the effect of volume increase in fine-grained rock materials (clay) upon the electrical resistivity below, at, and above the percolation threshold will also be examined in this research. Lastly, the effect of salinity upon the electrical conductivity below, at, and above the percolation threshold will also be examined in this research. Lastly, the effect of salinity upon the electrical conductivity below, at, and above the percolation threshold will also be examined in the short of the percolation threshold will be investigated in the laboratory work.

1.3 Objectives and Scopes of Study

The main objectives of the study include the following:

- 1. To estimate the threshold concentration of clay content to start electricity percolation by using a 2D numerical model.
- 2. To investigate the effect of amount of clay on core resistivity from the synthetic cores prepared with different clay-sand ratio.
- 3. To determine the impact of different brine salinity on core resistivity.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Shale Formation

Shale gas production has received much attention especially during the last two decades, due to the fact that the conventional oil are facing the problem of depletion and high increase in price (Alternative Energy Sources- Unconventional Oils, 2007). In United States for instance, development strategy has been implemented in several shale gas formations such as Mercellus Formation, the Haynesville Formation, the Eagle Ford Formation, Woodford shale and Barnett Shale which in turn contributed towards the huge increase in the US's natural gas production (Soua, 2014). These shale gas formation have rapidly become important hydrocarbon targets, with the possibility that the recovery of shale gas in other countries can be made commercially profitable, across the world including North America.

Shale is a type of fine-grained sedimentary rock that forms due to the compaction of silt and clay size mineral particles which are commonly known as mud, this property causes shale to be categorized as mudstones (Shale, 2007). The laminated and fissile properties of shale make it different from the other types of mudstones. By the term "laminated", it means that shale is made up of many thin layers while the word "fissile" implies that the shale can be easily broken into thin pieces along the lamination. There is one common definition that shale should contain at least 67% of clay mineral.

In one of their publication, Caineng et al. (2013) define shale as rock formed from fragment, clay an organic matter, with the grain diameter of less than 0.0039 mm. Shale is commonly distinguished for its laminated, finely layered and fissile properties. They also mention in their book that shale is the most abundant type of sedimentary rock found on earth, where the total amount of sedimentary rocks account for 55% among the rock types that cover earth surface. Shale has a very complicated mineral compositions. They contain both clastic minerals such as quartz, feldspar, calcite and also the clay minerals, which include kaolinite,

turface, illite and hydromica. Different proportions of these constituent mineral will in turn lead to the great diversity of shale.

There are many different types of shale according to their compositions as mentioned before (Caineng et al.). For instance, shale which contains high amount of silicon dioxide is known as siliceous shale while shale rich in organic carbonaceous matter is known as carbonaceous shale. On the other hand, shale which contain significant amount of scattered organic matter and iron sulphide is known as organic-rich shale or another name, the black shale (Zhang et al., 1987).

National Park Service (2015) explains about the depositional environment of shale formation. Due to its small particle size, shale is generally known to be deposited in a relatively deep and calm water, where the water is calm enough that it is no longer able to suspend such fine particles in it. On the other hand, Caineng et al. mention in the book that shale can be deposited in marine, terrestrial and also marine-terrestrial environments. The depositional environment for black shale is typically of poor oxygen and high hydrogen sulphide, such as deep shelf, closed bay, under-compensation basin, deep lake and lagoon. Generally, both sources agree with each other that shale is deposited in deep environment.

In the past, reservoir rocks such as the sandstone formation, carbonate rock and volcanic rock were the main targets in the oil and gas development. During the drilling operations, shale formation is also penetrated by the well and significant amount of the oil and gas have been discovered from these sections. However, shale is still considered as a source rock instead of reservoir rock due to the fact that it has a very poor reservoir physical properties, with porosity of less than 10% and permeability of less than 0.001 square micro meter (Editorial Board of Series of Shale Gas Geology and Exploration and development, 2009). For the past, shale is only considered as source rock or seal with no major reservoir development interest until when North America successfully develop shale reservoir by fracturing it.

2.2 Electrical Conductivity Phenomenon of Shale Formation

There are several methods to determine the shale formation. The most common method to determine shale formation is by using gamma ray logs (SPE, 2013). This is due to the fact that shale formation contains radioactive materials such as plutonium, uranium and thorium. Generally, gamma ray logging tool is used to determine the volume of shale in order to determine net to gross ratio for volumetric calculation. As mentioned before, shale formation is generally associated with low permeability and porosity. Thus, shale formation can also be implied from these logs with reference to other properties such as gamma ray log. One more important indicator but at the same time confusing parameter to determine shale formation is the resistivity log. Normally, low resistivity is associated with water bearing sandstone formation. However, it is also observed that sometimes this shaly formation also contributes towards the low resistivity log reading.

The research of the effect of clay content and water salinity can be traced back to year 1957, with a study conducted by Hill and Milburn. It was implied by them that the presence of clay minerals further complicates the interpretation of the complex electrical logs. This type of phenomena is further studied by Worthington (1982). The root cause of this shaly-sand problem in hydrocarbon evaluation is due to the effect of these relatively fine-grained minerals upon the electrical parameters measured. This influence is shown as an excess conductivity. If the formation is having low shaliness, this excess conductivity is normally regarded as insignificant. Worthington also emphasized in his study that the degree of shaliness does not depend only on the lithology (presence of shale) itself, but also the salinity of the saturating solution. A formation or its section is thus classified as effectively clean or effectively shaly, depending on whether it obeys or defies the fundamental empirical law of Archie.

Hill and Milburn have correlated the resistivity of the fully saturated granular formation, R_0 with resistivity of saturating electrolyte, R_w using the following equation:

$$F = \frac{R_o}{R_w} = f^{-m} \qquad \qquad \text{------ Equation 1}$$

Where F the formation resistivity factor is the ratio between R_o and R_w . f is defined as the formation porosity while m is the slope of the average line when the data is plotted on a log-log graph paper. Worthington further explained in his study that F actually serve as a geometric factor to represent the tortuosity of the pore channels. By the definition above,

the formation resistivity factor of the rock should be an intrinsic quantity while as a fact shall not be vary with respect to the saturating electrolyte salinity. However, it was later discovered that actually the value of F is not a constant for the particular reservoir but in turn, varies with the resistivity of the electrolyte saturating the reservoir. A conclusion has been drawn where this phenomenon is actually contributed by the presence of clay or shale element in the rock sample or reservoir. Thus, the value F_a (apparent formation resistivity factor) is introduced instead of F, for a shaly reservoir. In a reservoir with very high shale content, the value of F_a might be as low as only 10% of the F value. Hence, if this shale effect is ignored, interpretation regarding this reservoir will yield a significant error. To further explain this phenomenon, a basic correlation has been introduced to describe the relationship between F_a and F:

$$\frac{F_a}{F} = (1 + XR_w)^{-1} \qquad \qquad \text{------ Equation 2}$$

Where X may be considered as a generalized parameter to describe shaliness. From this equation, it can be implied that when the product of XR_w approaches zero, the ratio of apparent and real formation factor approaches one, indicating a potential clean sand. An analogous equation proposed by Waxman and Smiths, 1968 has the similar form:

$$\frac{F_a}{F} = (1 + BQ_v R_w)^{-1} \qquad \qquad \text{------ Equation 3}$$

Where Q_{ν} represents the volumetric concentration of clay exchange cations and B is the equivalent conductance of these cations. The product these two parameters is equivalent to the excess conductivity associated with the shale effects. Attention should be given where these excess conductivity caused by shaliness is electrolytic in nature and shall not be mixed up with electronic conduction which is observed in reservoir rock containing disseminated ores.

In Worthington's study, he has also presented the relationship between F_a/F with R_w . All his sample showed that as the value of electrolyte resistivity decreases, the value of F_a/F approaches unity. In his interpretation also, it was mentioned that cleaner sand will have a higher tolerance for the value of F_a/F to approach unity over a larger range of electrolyte resistivity. On the other hand, the F_a/F value for shaly sand will start to deviate from unity when the value of R_w is still low, as compared with cleaner sand.

2.3 Cations Exchange Capacity (CEC)

As mentioned in the previous section, clay acts as one of the most common constituent of the shale. And in fact, the clay content in shale is the one that causes the excess electrical conductivity of shaly formation. Clay is made up of different minerals such as kaolinite and muscovite due to the process of isomorphous substitution (Paulus, 1999). Often, an atom of higher valence in a mineral will be replaced by one with a lower valence, which in turns creates a deficit of positive charge and an excess negative charge. This excess of negative charge is compensated by the adsorption on the layer surfaces of cations, which are too large to be accommodated in the interior of the lattice. When there are water and other cations present in the solution, the cations on the layer surfaces can be easily exchangeable, and these cations are called exchangeable cations. The amounts of these cations can be determined analytically and is expressed in milliequivalents per 100 gram of dry clay. This property of shale is known as cation exchange capacity (CEC). It is this property that contribute towards the electrical conductivity of shale.

Schlumberger (2015) define CEC as the ability of a clay's negatively charged surface to accommodate positively charge ions or cations. Most of clay consists of aluminosiliates whereby parts of the aluminium and silicon ions can be replaced by elements with different value of charge. For instance, aluminium ion (Al^{3+}) might be replaced with magnesium (Mg^{2+}) or iron (Fe^{2+}) ions. This deficient in positive charges will attract the cations when the clay is suspended in electrolyte, causing the formation of an electrical double layer. This electrical double layer consists of the Stern and Gouy layer. Stern layer is formed when the extra cations attracted to the clay surface also come together with their hydration water molecules. This layer is next to the clay surface and no anion is present. The Gouy layer is outside the Stern layer, where the ion concentration gradually approaches the one as in the bulk electrolyte. The thickness of Stern layer is inversely proportional to the electrolyte salinity. Several methods are available to measure the CEC, such as membrane potential, wet chemistry and multiple salinity where some of these methods will permanently destroy and alter the rock.

2.4 Shale/ Clay Distribution Patterns in Reservoir

One of the most important property that affect resistivity response is the shale distribution pattern. In laminar shale distribution, the total conductivities of clean sand and shale laminae determines the rock resistivity (Paul, 2006):

$$C_t = C_{sand} + C_{sh}$$
 ------ Equation 4

Expressed in terms of resistivity:

$$\frac{1}{R_t} = \frac{\left(1 - V_{sh}\right)}{R_{sand}} + \frac{V_{sh}}{R_{sh}}$$
 ------- Equation 5

Where:

Rt = total resistivity; Rsand = clean sand resistivity; Rsh = shale resistivity;

Vsh = shale fraction

Dispersed shale system generally has a low porosity due to the fact that the open spaces between sand matrixes have been filled by shale. To describe the resistivity of dispersed shale system, Simandoux equation is commonly used:

$$S_{w} = \left[A R_{w} \phi^{-m} \left(\frac{1}{R_{t}} - \frac{V_{sh} S_{w}}{R_{sh}} \right) \right]^{1/n}$$
 ------- Equation 6

In structural shale distributed formation, the resistivity is generally lower compared to other system because there is a larger effective surface for conduction to occur (Paul, 2006). Waxman and Smit have developed an equation to describe the resistivity of this type of formation, where its resistivity depends only on water conductivity and CEC in water saturated condition. Their equation does not account for shale distribution.



Figure 2 Conductivity of a shaly sand versus formation water conductivity (Paul, 2006)

Wang et. al. has conducted a study using three dimensional percolation network model to describe pore structure, fluid distribution and electrical conductivity in shaly reservoir. The coding and generation of their model is carried out using computer simulation language C. They have used Weibull function to describe the reservoir pore structure. In their simulation, effect of different salinity level is studied and analysed. The critical salinity where shale role changes from reducing resistivity to increasing resistivity has also been determined in their study. They found out that at low water salinity, the shale plays as a resistivity reducing agent while the opposite case is true when the water salinity is higher.

2.5 Percolation Theory

Percolation is a random probabilistic process that involves a phase transition (Azita, 2011). In a particular system of percolation, clusters of different sizes and shapes may present. Percolation threshold is defined as the critical value of concentration when formation of infinite or long-range connectivity in any system first occurs. The application of percolation is very broad, including the field of mathematics, biology, hydrology, ecology, petroleum, chemistry, physics and material sciences. This is due to the fact that percolation is a very general phenomenon applicable in almost every area of science as the simplest model of spatial disorder. In the introduction of Jared and Alan's (2012) research, they quoted that percolation system is used by geologists to study the diffusion of water and other nutrients through soil while in biology field, percolation system is used to study the diffusion has been given especially during the study of semiconductors.

In an infinite system, the percolation threshold is well defined by a distinctive phase transition at a finite value of ϕc where the infinite system will definitely not percolate if the concentration is lower than this ϕc and when the concentration exceeds this critical value, the system percolates. On the other hand, the situation is different for a finite system. A discrete phase transition technically does not occur for a finite system, meaning that system of finite size might have a percolating cluster for concentration lower than ϕc and might not be able to percolate at concentration higher than ϕc (Azita, 2011). In practice, the numerical computer simulations are done on a limited-size percolation models and extrapolated to estimate the percolation threshold for and infinite-sized system using size scaling theory.

Typically, the conductivity of a composite material can be expressed in the formula below:

$$\sigma = (\phi F - \phi C) t$$
 ------ Equation 7

Whereby the electrical conductivity of the composite is represented by σ , while ϕF and ϕC represent the concentration of the filler and percolation threshold respectively. A is a proportionality constant while t is a dimensionless exponent.

One of the examples of percolation system is the clustering of conductive particles in an insulating matrix (Sahimi, 1994). In fact, one of the most important roles of percolation effects is the study of electrical conductivity in various system, including the conductor-

insulator composite system. In this study, percolation system will be used to study the electrical conductivity and resistivity of shaly formation. The electrical conductivity of a composite is affected by the conductive filler loading. When the conductive filler concentration is low, the system acts as an insulator. By increasing the amount of filler, the conductivity of the system increase at a very slow rate. When the concentration finally reaches the percolation threshold, the conductivity increases by several orders of magnitude. Continue increasing the conductive filler content will increase the conductivity very little until it behaves like a conductive filler itself.

Azita has quoted in their research that huge amount of effort has been rendered in order to model the percolation phenomenon. They mentioned about the two primary formulations which are popular in finding the exact and approximated values of percolation threshold in various systems. The first type of modeling is the lattice modeling, where common two dimensional lattice models are square, triangular and honeycomb structure. For three dimensional lattice models, cube and diamond are the common ones. Lattice modelling analyses percolation systems through the concepts of bonds and sites percolation.

Another type of percolation models is the continuum modelling. The research by Meester and Roy (1996) regarding continuum modeling has also been quoted by Azita. This type of modelling is also known as off-lattice models. In this type of modelling, the geometrical objects or the filler can be placed randomly and individually in any spot of the continuum space and a connectivity is formed when the objects overlap with each other.

In this research, the clay particles act as the conductive filler while the sand matric act as the nonconductive medium. Once the percolation threshold for the clay content is reached, the system start to conduct, in other words, additional conductivity is achieved due to the formation itself, and not only because of the water salinity.

2.6 Synthetic Cores

Synthetic core involves the preparation of reservoir rock core plugs from the unconsolidated sand and other materials, which include the resin or bonding materials under compression at certain temperature. In a research carried out by Orrin (1963), synthetic cores have been produced to study the effect of water sensitivity. They proposed this method of investigation due to the fact that natural core are expensive to obtain, which might be very appropriate in the time of their study. Besides, the composition of clay is also hard to be determined from the natural cores due to the technology of that age. With synthetic cores, the composition of the materials added can be clearly known and manipulated for better study. In their study, they mixed larger portion of unconsolidated sand with certain amount of clay and a thermoplastic resin as a binding agent. After that, the mixture is subjected to high compression pressure and suitable temperature under a hydraulically controlled piston. The pressure applied range from 12000 to 18000 psi, with a duration of 10 to 15 minutes. Attention should also be given to pressure applied during the preparation process, as the permeability is proportional to the pressure applied. The grains size of the mixture will affect permeability as well, where smaller grain size will lead to lower permeability value. The amount of resin to be added will affect the permeability as well. The resin added should be just enough to make the core consolidated. Too much resin added will plug the pores.

2.7 Cores Resistivity Measurement

The core resistivity measurement instrument can be traced back from year 1957 by a research conducted by Rust. In his study, he has introduce the use of two-electrode and four-electrode for measuring the resistivity of the core sample.

The two-electrode method, the core sample is located in between the two electrodes, where current of known magnitude is passed through the core sample. The potential drop across the core is noted and the calculation for resistivity is done using the data obtained from the measurement and also the geometry such as the length and cross section area of the core. On the other hand, the four-electrode method still uses two end plates as the current electrodes, but additional two electrode are used in between the core samples to measure the potential drop across the sample with different length. In the two electrode method, the excess resistivity when the current is passed through from the electrodes to the cores will affect the real resistivity value. In the four-electrode case, this problem will be alleviated since the potential drop is measured across the core sample itself.



Figure 3 Core Sample Resistivity Cell (Rust, 1957)

Currently, one model of device used for measuring the core sample resistivity is introduced by Core Laboratories (2015). This machine is known as Advanced Resistivity ARS-300. It consists of a pneumatic atmospheric core holder, programmable RCL meter and also a fourelectrode measuring system. The pneumatic atmospheric core holder enables the application of desired pressure of up to 10,000 psi onto the core during measurement to resemble the reservoir condition. It also has a panel to measure the brine resistivity, which is the data required to calculate the formation resistivity factor. This device is able to apply three test voltages of 300mV, 1V and 2V onto the core sample with the frequency of 50 Hz to 1MHz.



Figure 4 Advanced Resistivity ARS-300 (Core Laboratories, 2015)

CHAPTER 3

METHODOLOGY

The first part of the research is the use of percolation simulation by using MATLAB software to roughly determine the critical composition for clay material to cause extra conductivity in the core system. A two-dimensional lattice structure is created using MATLAB coding, and the conductive particles are assigned randomly using Monte Carlo simulation technique. The concentration of conductive particles are decided by user in order to study the critical threshold for percolation to first occur. Several test will be run in order to determine a more accurate threshold concentration.

After the estimation of percolation threshold from the simulation results, several experimental steps are involved to further investigate the effect of clay concentration on extra conductivity of the core, from the preparation of materials needed for the core creation process, designing and fabrication of the core mould, compression process, saturation of cores and finally to the measurement of the saturated core resistivity. Each and every step will be further explained below.

3.1 Preparation of materials for core preparation

There are three raw materials needed for the preparation of the cores:

1. Sand

The sand particles needed for the core preparation will be obtained from the Civil Engineering Department of UTP, as they have the sufficient amount of sand particles with ranges of specific sizes. Further sieving process will be carried out at the Civil Engineering laboratory in order to make sure the sand particles size are relatively constant, as this will severely affect the permeability of the core produced. The sand particles used for this study is 0.3 mm in grain size. This is categorized as medium size sand, with reference to IHRDC. The source of the sand is from Tronoh River sand, a place that is nearby UTP campus.



Figure 5 Sand Material Used for Experiment

PARTICLE TYPE	GRADE LIMITS (mm)	GRADE LIMITS (microns)	AGGREGATE
SAND	Range: from 2 to 0.625 mm	Range: from 2000 to 50 microns	Sand / Sandstone
Very coarse sand	2 to 1 mm	2000 to 1000 microns	Sand / Sandstone
Coarse sand	1 to 1/2 mm	1000to 500 microns	Sand / Sandstone
Medium sand	1/2 to 1/4 to mm	500 to 250 microns	Sand / Sandstone
Fine sand	1/4 to 1/8 mm	250 to 125 microns	Sand / Sandstone
Very fine sand	1/8 to 1/16 mm	125 to 63 microns	Sand / Sandstone



2. Clay (Shale)

The clay solid sample has been obtained from my co-supervisor, Mr. Asif. The sample came from a clay outcrop in Sarawak, Malaysia. The solid clay is first crushed into small pieces using the hammer, before being sent to be grinded into powder form. In order to understand further the composition of the clay material, parts of the powder form will be sent for X-Ray Diffraction Analysis.



Figure 6 Clay Material Used for Experiment

3. Sodium Silicate

This chemical is needed in liquid form to act as the binding agent of the core sample during compression. This chemical is available in highly concentrated form, further dilution with 97% wt. sodium silicate and 3% wt. water is needed before it can be used for the preparation process.



Figure 7 Sodium Silicate Solution Used for Experiment

3.2 Creation of the Core Mould

Since the creation of core samples requires the use of hydraulic compression machine, there is a need for suitable sized and designed mould to contain the materials during compression. The mould is designed to be able to contain 20 cm X 30 cm core samples. This mould can be created using the CNC machine available in UTP laboratory. The mould contains of three parts, one outer hollow cylinder to contain the core materials, and two solid cylinder with the same diameter as the core samples to contain the mixtures and act as the surface for compression. The standard core size is supposed to be 1.5" X 3.0". However, due to the budget constraint, bigger size mould manufacturing is not feasible as the cost is more than double of the mould with smaller size. The material used for the mould is Stainless Steel 304 (SS304) due to its strength, as the mould will be subjected to high pressure during the compression. Before manufacturing, the design of the mould has been discussed thoroughly with the laboratory technician for its feasibility and usability.



Figure 8 Core Compression Mould

3.3 Mixing of Core Materials and Compression of Core Samples

The core samples is formed by mixing the clay, sand and sodium silicate together in the mould and compressed with certain suitable range of pressure for a time duration. Seven cores of different sand-clay ratio have been designed to be produced in this stage. The ratio is in volumetric basis. Thus, the volume of sand and clay are measured using measuring cylinder. After mixing the sand and clay, sodium silicate solution is used to act as the binding agent. The amount of sodium silicate solution to be added is suggested to be around 7% in weight with respect to the total weight of the sand and clay mixtures.

Same amount (2 ml) of sodium silicate solution is added for each core sample because the amount of sodium silicate solution will actually affect the porosity and permeability of the cores and should be kept constant. The sodium silicate is added using syringe because it is very vicious and hard to be measured and transferred using measuring cylinder repeatedly as part of the sodium silicate will stick to the measuring cylinder inner wall. After adding all the needed materials together, they will be mixed thoroughly using a spatula and constantly be compressed manually to make sure the materials are mixed homogeneously.



Figure 9 Measuring Volume of Sand and Clay Materials



Figure 10 Adding Sodium Silicate Solution to the Mixture

Hydraulic compression machine is used for this purpose and the maximum force it can apply is up to 160 kN.

The area subjected to pressure is calculated as follow:

$$A = \frac{\pi (2 \ cm)^2}{4} = 3.142 \ cm^2 \qquad ----- Equation 8$$

The force applied for all core samples is the same value of 35 kN, converting the value to pressure:

$$\frac{35 \ kN}{3.142 \ cm^2} \times \frac{224.809 \ lb}{1 \ kN} \times \frac{1 \ cm^2}{0.155 \ in^2} \approx 16,000 \ psi$$
 ----- Equation 9



Figure 11 Core Compression

After the core is formed, it is then removed from the mould. The cores would be heated to get them dry at 100 $^{\circ}$ C and 200 $^{\circ}$ C using oven for 8 hours and 4 hours respectively, and then cement crystallization at 300 $^{\circ}$ C by using furnace for another 4 hours.



Figure 12 Heat Treatment of Core Sample in the Oven

3.4 Saturation of Core Samples

After the core samples are treated in the oven, it can then be saturated with brine solutions with different salinity. Different salinity of brine solution is used at different stages in order to study their effect on resistivity behaviour. The saturation process is done using the vacuum core saturation set up, which is available in the core analysis laboratory of Petroleum Engineering Department.



Figure 13 Saturating Core Samples with Brine Solution

This device works by sucking the air out from the cores' empty pore and displacing the pores with brine solution the cores are being submerged in. A vacuum pump is connected to this glass container through an air tight hose.

For each stage, core samples with different sand- clay ratio will be saturated with the same brine and the resistivity is measured. After that, the core samples will be cleaned and again saturated with brine with different salinity. The resistivity behaviour of different core samples will be recorded and tabulated in the results section. The porosity of the cores are also obtained by measuring the weight difference before and after the saturation with brine solution.

The brine salinity to be used for the experiment is quoted from one of the research conducted by Wang et al. (2006):

Description	Salinity in mg/Litre
Low Salinity Brine	5000
Medium Salinity Brine	15000
High Salinity Brine	150000

Table 2 Salinity of Brine Solution Used

3.5 Measurement of Core Resistivity

The saturated core samples will need to have their resistivity values measured. However, there's no specific equipment for this type of measurement. Equivalent electrical set up will be determined and used in order to obtain the resistivity values. In this research, RCL meter (Model: FLUKE PM6303A) is used to measure the core resistivity as shown in the set up below:



Figure 14 Measuring the Electrical Resistivity of Core Samples Using RCL Meter

Instead of direct measurement using the clip to secure the cores, two copper plates are used to keep the cores in between in order to make sure that all the volume of the core will be measured for its resistivity instead of its surface conductivity. The measurement is taken from both the copper plates. During the measurement, the core is held in the air to prevent extra conductivity added to the system and affect the results.

3.6 Cleaning of Cores after Resistivity Measurement

After each saturation, it is important to clean the remaining salt inside the pore space of the core, as the oven can only dry the cores, but not removing the solid particles. If the remaining salt particles in the cores are left unclean, it will affect the result for next saturation. This cleaning process is carried out using the Soxhlet Extraction equipment, with toluene as the cleaning agent. The setup is shown below:



Figure 15 Cleaning of Core Samples from Salt Particles Using Soxhlet Extractor

The Soxhlet apparatus is used to extract and clean the core samples from oil, water and any other materials such as salt. The apparatus is based on a heating mantle to boil the solvent, a sample chamber and a water-cooled system to condense the solvent vapours. The cores

are inserted into the sample chamber at the middle section. The bottom section of the setup consist of round bottom flasks. The cleaning agent, in this case the organic solvent toluene is used to fill up the round bottom flask to around ³/₄ of the total capacity of the flask. The setup is then heated using the heater at the bottom. Upon reaching its boiling point, the toluene vapour will move upwards and contact with the cores. The vapour will help to clean the impurity inside the cores, including the salt particles remaining. Then, the spoiled toluene will be evacuated from the chamber through a siphon and goes back to the flask where it will be redistilled.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 2D Percolation Simulation

For the first stage of the research, a simple 2D regular model with the size of 40 X 40 has been built and tested with different concentration of conductive particles in order to examine the trend of percolation. The model is visualize using the graphical function of MATLAB. Based on study proposed by Kim (2002), the approximated percolation threshold for 2D regular geometry is around 0.59. Thus the study starts with concentration of 0.4 to see if there's any percolation in the system.



Figure 16 2D System with Concentration of 0.4

Figure 17 shows the system for concentration equals to 0.4. It is observed that there is neither continuous path from top to bottom nor from right to left, meaning that the system is not conductive in this concentration. Note that the black color circles represent conductive particles while the white color circles represent nonconductive matrix. Thus, the concentration of the system is increased to 0.45 for further study and observation.

For the first run of concentration of 0.45, we can see that there is one connected path from top to bottom and no path from right to left is noticed. This one and only path is also tortuous as the only way to connect is through the diagonal particles.



Figure 17 2D System with Concentration of 0.45 (1)

Repeated run is carried out for the same concentration of 0.45 and this time it is shown that no conductive path is found throughout the system. Further runs need to be carried out to examine the behavior of percolation with this concentration of conductive particles.



Figure 18 2D System with Concentration of 0.45 (2)

The concentration of conductive particles are further increased to 0.5 to study the percolation behavior, and the result is shown in Figure 20.



Figure 19 2D System with Concentration of 0.5

As shown above, more conductive particles are seen as we increase the concentration, and there is a connecting path for this concentration. Figure 21 shows the system for concentration equals to 0.6:



Figure 20 2D System with Concentration of 0.6

More paths are available for the conductive particles as compared to previous cases which has lower concentration. With the results presented, the approximated percolation threshold for the system is around 0.4 to 0.5. However, since this is a two-dimensional representation, in addition to its regular geometry, which is not the case as in real core condition. However, this result does give a good estimation for the approximate volume percentage of clay to cause the system to percolate, in turn provide extra conductivity to the cores.

4.2 Sand-Clay Mixture Proportion Calculation

The proposed volume weighted proportion for the sand-clay system based on the 2D simulation results is as in Table 3:

Core Number	Clay Volume Percentage (%)	Sand Volume Percentage (%)
1	0	100
2	15	85
3	30	70
4	45	55
5	60	40
6	75	25
7	90	10

Table 3 Sand-Clay Volume Percentage of Cores

The final length of the core after compression will be only 3 cm. However, the amount material prepared is enough to fill the mould to 5 cm before the compression process. The total volume of the core is calculated as in Equation 10:

$$V = \frac{\pi (2 \ cm)^2}{4} \times 5 \ cm = 15.71 \ cm^3 \qquad \qquad \text{----- Equation 10}$$

The volume of each component to be added will be calculated using the formula as follow:

$$Clay Volume = Clay Volume Percentage (\%) \times 15.71 cm^{3} \quad \text{------ Equation 11}$$

Sand Volume = Sand Volume Percentage (\%) × 15.71 cm³ \quad \text{------ Equation 12}

The volume of each component are tabulated in Table 4 for each core:

Core Number	Clay Volume (cm^3)	Sand Volume (cm^3)
1	0.00	15.71
2	2.36	13.35
3	4.71	11.00
4	7.07	8.64
5	9.43	6.28
6	11.78	3.93
7	14.14	1.57

4.3 Compression Mould Design

Suitable design of mould is needed in order to produce a high quality core. After discussion with the supervisors and laboratory technician, the following design is proposed, as shown from Figure 22 to 24:



Figure 21 Compression Mould- Hollow Cylinder



Figure 22 Compression Mould - Top Plunger



Figure 23 Compression Mould - Bottom Stopper

Figures above show the design and dimension of the mould in side views. The mould consists of three parts:

- i) Hollow Cylinder: this part act as the main container for the sand mixture
- ii) Top Plunger: this component acts as the top plug where it is in direct contact with the source of pressure. During the ejection of the core sample, it will also be used to push the sample out from the hollow cylinder.
- iii) Bottom Stopper: this part acts as the base of the mould mainly to support and contain the sand mixture before and during compression.

4.4 Brine Solution Resistivity



Figure 24 Resistivity of Sodium Chloride Solution (Schlumberger Chart Gen-9)

The resistivity of the brine solution used for this research is extracted from the graph above and expressed in the table as follow:

Table 5 Resistivity of B	Brine Solutions
--------------------------	-----------------

Description	Salinity in mg/Litre	Resistivity (ohm)
Low Salinity Brine	5000	1.05
Medium Salinity Brine	15000	0.13
High Salinity Brine	150000	0.03 (Extrapolated)

4.5 XRD Results for Clay

As mentioned in the previous section, several types of clay mineral actually contribute towards the electrical conductivity of the sand-clay system. Figure 26 illustrates the structure of the clay minerals:



Figure 25 Clay Mineral Structures

Using the X-Ray Diffraction facility available in UTP, the content of the clay used in this research is further interpreted. The metal used for this XRD analysis is copper. The X-ray is produced by bombarding a beam of high energy electrons emitted from a hot filament onto the copper surface. Each crystalline solid has its unique characteristic X-ray powder pattern which may be used as a "fingerprint" for its identification. X-Ray Diffraction is one of the most important tools used for solid characterization.

The raw results of the interpretation is summarized in Table 6.

Caption	Angle	d value	Intensity	Intensity %
	2-			
	Theta °	Angstrom	Count	%
d=4.47828	19.809	4.47828	127	7.2
d=4.25276	20.871	4.25276	310	17.7
d=4.02811	22.049	4.02811	116	6.6
d=3.69544	24.063	3.69544	127	7.2
d=3.34048	26.664	3.34048	1752	100
d=3.21333	27.74	3.21333	138	7.8
d=3.18520	27.99	3.1852	145	8.3
d=2.56539	34.947	2.56539	94	5.4
d=2.45480	36.576	2.4548	158	9
d=2.27945	39.502	2.27945	143	8.1
d=2.23450	40.331	2.2345	96.7	5.5
d=2.12657	42.474	2.12657	110	6.3
d=1.97887	45.817	1.97887	97	5.5
d=1.81675	50.175	1.81675	207	11.8
d=1.67072	54.911	1.67072	104	5.9
d=1.54128	59.971	1.54128	124	7.1
d=1.45294	64.034	1.45294	47.9	2.7
d=1.38226	67.735	1.38226	74.3	4.2
d=1.37286	68.263	1.37286	104	5.9
d=1.28780	73.475	1.2878	53	3
d=1.25593	75.662	1.25593	62	3.5

Table 6 XRD Results

The output can be represented in a form of graph with several peaks, as shown in the Figure 27.



Figure 26 Original Clay XRD Curve

This figure is obtained after smothering the original graph and applying Fourier transform. All of these steps can be done automatically by clicking some buttons in the software interface. The highest peak here is d=3.3408. After obtaining this graph, it is used to match with the compound available in the library to determine the types of compound present in the clay sample.





Figure 27 Matching with Quartz

The first matching element here is quartz, which is as expected since most of the sedimentary rock will contain this compound in abundant quantity. Most of the peaks are found to be matching between the quartz database pattern and our sample here.



Matching with Illite 4.5.3

Figure 28 Matching with Illite

The next matching element is the illite mineral, one of the most common mineral present in clay sample. The highest peak and some of the lower peaks are found to be matching between the clay sample and the database pattern. However, it is found that not all of the peaks are matching.





Figure 29 Matching with Kaolinite

The clay sample is next tried to be matched with the kaolinite mineral profile in the database. It was found out that some of the peaks are matching between these two. Kaolinite is also one of the most common mineral found in clay that contributes towards Cation Exchange Capacity (CEC).







The common clay mineral montmorillonite is also found present in this sample as there are five matching peaks between the database and our sample. This mineral also enhance the CEC of clay.







It is also interesting to find out that the clay sample used contain some carbon as well, as can be seen from the matching of the highest peak between our sample and the carbon database file. In short, this clay sample used for the research contains quartz, carbon, minerals such as illite, kaolinite and montmorillonite. The minerals are the one that contribute towards the extra conductivity of the core sample.

4.6 Porosity Calculation for the Core Samples

Porosity is used to refer to how much empty space is present in the core samples, in between the matrix particles. It can be represented using the formula below:

Where pore volume represent the empty space between the core particles and bulk volume represent the overall volume of the core. Before the saturation (when the core is dry), the empty space is filled with air with negligible mass. After saturation, the pores are filled with brine solution. As long as the density of brine is known, the volume of pore spaces can be determined. Before using the brine for saturation process, its volume and mass are measured and recorded.

Brine Density =
$$\frac{\text{Mass of Brine}}{\text{Volume of Brine}} = \frac{30.065 \text{ g}}{30 \text{ cm}^3} = 1.002 \text{ g/cm}^3$$
 ------ Equation 14

The volume of brine entering the core (total core pore space) can then be determined as follow:

$$Pore \ Volume = \frac{(Core \ Mass_{After \ Saturation} - Core \ Mass_{Before \ Saturation})}{Brine \ Density} \quad ----- \ Equation \ 15$$

The porosity of the cores are determined from the first saturation data as shown below:

Core	Initial weight (g)	Final Weight (g)	Weight Difference (g)	Length (cm)	Bulk Volume (cm3)	Brine Volume (cm3)	Porosity	Percentage (%)
1	23.013	24.925	1.912	3.2	10.0544	1.907866	0.189754	18.98
2	24.078	25.736	1.658	3.2	10.0544	1.654415	0.164546	16.45
3	21.741	23.219	1.478	2.9	9.1118	1.474805	0.161857	16.19
4	21.976	23.579	1.603	2.9	9.1118	1.599534	0.175545	17.55
5	22.026	23.509	1.483	2.9	9.1118	1.479794	0.162404	16.24
6	21.69	23.351	1.661	3.1	9.7402	1.657409	0.170162	17.02
7	21.489	23.07	1.581	3	9.426	1.577582	0.167365	16.74

Table 7 Core Samples Porosity Calculation

It is noted that the core length has slight difference between each other after compression, which is unavoidable for experimental work. Their effect has been considered in calculating the bulk volume as shown in the table. Unfortunately permeability cannot be determined due to the core sizes (non-standard).

4.7 Resistivity Measurement of Core Samples

4.7.1 Resistivity after Saturated with Low Salinity Brine

Cores	Clay Percentage (%)	Resistivity
		(Ohm)
1	0	5550
2	15	2230
3	30	1850
4	45	999
5	60	850
6	75	557
7	90	425

Table 8 Resistivity after Saturated with Low Salinity Brine



Figure 32 Resistivity of Cores wrt. the Clay Content (Low Salinity)

As shown in the Figure 33, when there is no clay in the core (clean sand sample), the resistivity of the core is very high, meaning that brine salinity alone cannot contribute much to the conductivity of the core. However, when the clay content is increased to 15%, there is a drastic drop in resistivity, even though the 2D simulation results shown that there is a

need for 45% of lay to initiate an extra conductivity. This difference in results are due to some of the factors such as the difference between 2D and 3D arrangement of particles in simulation and experiment cases. In addition, we assume the particle sizes to be uniform in 2D simulation but in the real case, the clay particles and sand particles are not same in size and this will affect the particles distribution and arrangement as well. The smaller clay particles might be able to slip between the bigger sand particles easily in the experimental case.

When the clay content is further increased, the resistivity of the samples decreases, but at a much slower rate, which is in consistent with Wang et al.'s study. When the clay is increased from 30% to 45%, there is also a steeper decrement in resistivity compared with other clay content.

|--|

Cores	Clay Percentage (%)	Resistivity
		(Ohm)
1	0	3450
2	15	1360
3	30	1150
4	45	640
5	60	530
6	75	332
7	90	325

Table 9 Resistivity after Saturated with Medium Salinity Brine



Figure 33 Resistivity of Cores wrt. the Clay Content (Medium Salinity)

The experiment is repeated by using brine solution of medium salinity (15000 mg/L). Similar trend of resistivity is observed for both low and medium salinity. However, a big difference of resistivity is observed for the clean sand core between both types of salinity. Low salinity of brine solution gives a resistivity value of 5550 ohm for clean sand core while medium salinity only gives 3450 ohm. This is the extra conductivity contributed by the water salinity alone because same core is being used for both the experiment. Similar trend with low salinity is also observed when the clay content is increased from 30% to 45%.

4.7.3 Resistivity after Saturated with High Salinity Brine

Cores	Clay Percentage (%)	Resistivity
		(Ohm)
1	0	1283
2	15	638
3	30	433
4	45	335
5	60	245
6	75	183
7	90	132

Table 10 Resistivity after Saturated with High Salinity Brine



Figure 34 Resistivity of Cores wrt. the Clay Content (High Salinity)

The experiment is finally repeated by using the brine with high salinity (150000 mg/L). In this case, the resistivity value of clean sand core is even lower compared with previous experiments. However, the trend in between 30% to 45% is not obvious here. The steep decrement in resistivity is only observed from clean sand core to 15% clay core. At high salinity, the role of clay to decrease the resistivity has been greatly reduced as compared with previous cases. In Wang et al.'s study, clay actually increase the rock conductivity at high salinity. However, this phenomena is not observed here. The resistivity decrement is however very minor when the clay content is further increased in this case.

4.7.4 Comparison of Results for Low, Medium and High Salinity

Clay	Low Salinity	Medium Salinity	High Salinity
Percentage	Resistivity (Ohm)	Resistivity (Ohm)	Resistivity (Ohm)
0	5550	3450	1283
15	2230	1360	638
30	1850	1150	433
45	999	640	335
60	850	530	245
75	557	332	183
90	425	325	132

Table 11 Comparison of Results for Low, Medium and High Salinity



Figure 35 Resistivity of Cores wrt. the Clay Content (Comparison between different salinity)

A comparison between the three cases has been done in order to better illustrate the results. It can be implied from the Figure 36 that water salinity has a big influence on core resistivity in the clean sand core. This is due to the fact that in clean sand the only medium for electrical conductance is the water salinity.

As the amount of clay content increases the resistivity is also decreased but at a lower rate. This is because the electrical conductivity is gradually approaching its maximum value and further increase in clay will not contribute much to the conductance. This can be further supported by results of 90% clay core sample. For three brine salinity, the resistivity values do not differ as much as compared to lower clay content core samples.

4.7.5 Comparisons of Resistivity Values with Respect to Water Salinity

Core Clay							
Percentage							
Salinity							
(mg/L)	0	15	30	45	60	75	90
5000	5550	2230	1850	999	850	557	425
15000	3450	1360	1150	640	530	332	325
150000	1283	638	433	335	245	183	132

Table 12 Comparisons of Resistivity Values with Respect to Water Salinity



Figure 36 Resistivity of Cores wrt. the Water Salinity (Comparison between different clay content)

In this section, the resistivity values are plotted against the water salinity for all the different core samples. It is noticed from Figure 37 that clean sand core has the highest resistivity values for all the brine salinity values compared with other core samples that contains clay. As the clay content is increased gradually, the resistivity is decreased, however, there's a limit in it as we can see that the lines gradually flatten as the clay content is increased. This result is consistent with the simulation results done by Wang et al.

Throughout the experiment, same cores are being used repeatedly. This is important because as different cores are used, the distribution between clay and sand will be different even though the volume ratio is the same. This difference in clay-sand distribution will strongly affect the results we obtain. There might be some worries regarding the salt remained in the core after the previous saturation but this problem can be eliminated as there is Soxhlet Extractor facility available to remove the remaining salt particles before the next saturation.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

Based on the results from 2D percolation simulation, the estimated volume fraction of clay material needed for the system to percolate and add extra conductivity is around 0.45 to 0.50. The following steps include several laboratory works, starting from the manufacturing of core compression mould, preparing of sand-clay mixture and sodium silicate solution, compaction of cores and their heat treatment, saturation of cores with different brine salinity and finally the measurement of the electrical resistivity of the saturated cores.

After obtaining the results at the end, several graphs showing the relationship between resistivity and salinity and also clay content have been plotted in order to illustrate their relationship. It is shown that the higher the clay content, the lower the core resistivity while the higher water salinity also gives a lower resistivity reading for the cores. Among all the cores, water salinity gives the greatest impact on clean sand core resistivity.

In order to analyse the mineral content of the clay used in this research, part of it in powder form has also been sent for X-Ray Diffraction Analysis. The XRD results show that the clay samples contain quartz and other clay minerals that contribute towards the electrical conductivity.

In a nutshell, the objectives of the research have been met, where a 2D percolation simulation model has been created to estimate the percolation threshold, different clay content of core samples have been created and finally their resistivity values after saturated with three different brine salinity have been measured and presented in the results section.

In the future, further work can be done to further improve this research. One of the suggestions is to distribute the clay in laminated pattern to study their effects. In addition, the core samples can be saturated not only by brine, but also crude oil at the same time to alter its water saturation. However, these modifications require a re-design of experimental set up.

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APPENDIX

Gantt Chart and Key Milestone

Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Planned Work														
Identify the availability of														
facilities/equipment needed														
Booking of laboratory and equipment														
Planning of mould design for core compression														
Submission of mould design														
Preparation of sand-clay mixtures														
Submission of Progress Report							•							
Preparation of sodium silicate solution														
Core compression and heat treatment														
Core saturation with low salinity brine and resistivity measurement														
Poster Presentation										•				
Core saturation with medium salinity brine and resistivity measurement														
Core saturation with high salinity brine and resistivity measurement														
Submission of Technical Paper												•		

Submission of Dissertation						•	
FYP Viva							•