

**Visualizing the Nano-Particles Flow through Porous Media
using Transparent Micromodels**

by

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CERTIFICATION OF APPROVAL

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In partial fulfillment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(PETROLEUM)

Approved by,

(AP. Dr. Syed M. Mahmood)

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.

MUHAMMAD ARIF BIN MOHD RASHID

Abstract

The nano-particles flow study in reservoir is still new for the petroleum extraction field. The increasing need for visualizing the nano-particles effects towards fluids flow in porous media is the result of the importance of nano-particles study in understanding deeper about particles-flow behavior and its effects towards other fluids especially in EOR process. In this research, few transparent micromodels have been constructed to resemble the reservoir rock as close as possible. The best micromodel is selected and tested for fluids flow. The fluids flow were tested with and without the presence of nano-particles and the comparison was made during the observation. The presence of nano-particles effects towards fluids behavior were highlighted.

Few micromodels have been constructed as resemble as possible with the reservoir rock properties although there are few constraints limiting the capacity of the model. Each micromodels were made differently in terms of their grains size and structure which later on induced the porosity and visibility value. One micromodel has been chosen due to its good visibility under microscope lens while maintaining the least minimum porosity (among all the micromodels). This is important as the fluids flow process in the porous media can be visualized as good as possible like the process of fluids flow in the reservoir rock if good micromodel is selected.

The fluids flow test were carried out upon the micromodel selection. The effect of nano-particles towards fluids during the displacement process of two fluids were observed thoroughly under the microscope. Few founts were discovered in the resultant images of the fluids flow and analysis were carried out qualitatively for the mean of understanding their correlation with the nano-particles effects.

The visualization of nano-particles effects towards fluid flow in porous media by using the selected transparent micromodel has been successful and the aim to see if there is any effects of nano-particles towards fluids has indeed yield positive results.

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Chapter 1 : Introduction

1.1 Background

The subject of nano-particles flow in porous media has become a new aim for the advancement in petroleum study. The emerging of nanotechnology application is quite sudden recently. The discoveries of the nano-technology potential to become solutions towards several problems in petroleum industry has become encouraging in recent research. Enhanced Oil Recovery (EOR) is the most speculated area for the potential enhancement by this nano-technology application. The rise of energy demand in global scale which expected to be occurred in the oil and gas industry has made EOR the most important tools as to meet this expectation. The advantage of nano-particles in EOR is the ability of it to alter certain factor in the formation and fluid properties. This involves introducing these nano-particles into formations and studying its effects on oil recovery. Nano-particles study has been a subject of interest especially in researching the EOR elements such as single phase fluid flow, chemical flooding, miscible displacement and multiphase fluid flow in the porous rock. The determination of performance in the hydrocarbon reservoirs affected by nano-technology has made the nano-particles effects towards rock fluids study the important subject to be known about.

Few methods are available for the modelling approach in visualizing nano-particles flow in porous media. Transparent micromodel is one of them. The method utilize from the transparent micromodel is the application of fluid flow through the model matrix (that being fabricated from almost exact porous media with their interconnected pores) plastic or polymer formulated model (Martin, 2014) which can be observed directly under light influence. The behavior of the fluids flow effects by nano-particles can be study from the optical microscope and the result obtained can be used to derive the explanation for the nano-particles flow and its effect in the targeted subject of interest.

Visualizing the nano-particles effects towards fluids flow in porous media has become the primary focus in this project. By using transparent micromodels, the depiction of the nano-particles effects in fluids flow can be made possible with the optical microscope. Hence, it should be helpful in improving the understanding of the nano-particles with its flow and effect characteristics correlate with porous medium

element such as reservoir rock. The inter-relationship explanations between the fluids flow with the rock properties such as porosity, permeability, capillary effect, pore and grain sizes, grain surface roughness, wettability, and shapes, the average number of flow entrance/exit paths from pores in the network, and pore network size and coordination number can be made from this visualization (Martin, 2014). The interpretation of the correlation between nano-particles effects toward fluids flow with the effect of changing parameters like types of injection fluids, injection rate and pore properties also can be made with the visualization of micromodel. The utilization of this understanding should help EOR process in increasing its effectiveness.

1.2 Problem Statement

The nano-particles flow study in reservoir is still new for the petroleum extraction field. The subject of flow of fluids and particles in porous media is important in determining the performance of underground hydrocarbon reservoirs especially during production and EOR. The increasing need for visualizing the nano-particles effects towards fluids flow in porous media is the result of the significant importance of nano-scale study in understanding deeper about particles-flow behavior and its effect towards other fluids especially in EOR mechanisms since its manipulation should be helpful in the process of increasing the chances of high hydrocarbon recovery from deep earth.

1.3 Objective

There are several objectives identified in this project

1. To experiment and find the suitable method of construction and develop a successful transparent micromodel which can represent a good porous media.
2. To visualize the nano-particles effects towards fluids flow in porous media by using transparent micromodel so that the effects of nano-particles as the additive in EOR fluids in the rock can be understood.

1.4 Scope of Study

The scope of study comprise of few elements;

1. The study on the petroleum field which the fluids flow study is narrowed into the study of hydrocarbon with its related phases and fluids/reservoir rock properties especially with regards of Enhanced Oil Recovery (EOR) application.
2. The factors behind nano-particles with its characteristic which can affects the fluids behavior.
3. The analysis of pore-scale level condition with the help of microscope and the analysis will be carried out macroscopically. Experimenting the fluids flow, fluids behavior, nano-particles interference and grains-simulated-reservoir rock.
4. The study and experimentation to develop a good model that can exhibit the properties of formation rock. If this process is possible, then the study can be further analyze with regards of petroleum/reservoir condition.
5. EOR characteristics with regards of nano-particles should be understood well in doing this project since it is the primary reason of this study.

1.5 Relevancy of Study

This FYP is highly relevant to the Petroleum Engineering as nano-particles application has become the new alternative in increasing the effectiveness of EOR technique in Oil and Gas (O&G) field. This study focus on investigating and documenting the performance of different fluid flows with the presence nano-particles effects, which can be a very beneficial research to the industry. In addition, this research expose the author to experimental values and practices, which are crucial in developing the technical skills in petroleum field. This experience will be very valuable and useful for the future.

1.6 Feasibility of the Project within the Scope and Time Frame

This project is feasible as it is a simple experimental study, therefore it is expected that technical problem that will be face is not really much. However, a few limiting factors and problems do exist and need careful planning in approaching them.

The experimental study was implemented by using optical microscope equipment. This equipment is available in the environmental laboratory at Academic Block 14. The usage of the equipment is simple and easy, and it is free for the student use, thus this project can be implemented with relatively at very minimal cost. Although the problem of developing a good micromodel and finding a suitable nano-particles for the project will be expected, few alternatives are present in chartering this matters. But, as long as the proposed program for the research is progressing well, then the project can be run smoothly and at the same time, minimizing the above factors effect.

In terms of time frame, time losses was expected as the author is new in the research field. Since it will required a lot of initiative and dedication in completing this project, the self-learning sessions and active interactions are needed in building the momentum for the research. In early time of the project, the targeted objectives was to test different type of micromodels in fluid flows study and deducting the phenomenological process happening in the micro level of the micromodel pores. However, due to the limiting time and cost factors, the objectives of the project was redefined to limit on the study of fluid flow effect by the nano-particles and developing a good transparent micromodel that can resemble a good rock properties.

Chapter 2 : Literature Review

2.1 Nano-Particle

Nano-particle is defined as a small object that behaves as a whole unit with respect to its transport and properties which cover a range between 2,500 and 10,000 nanometers for coarse particles, 100 and 2,500 nanometers for fine particles, and 1 and 100 nanometers for ultrafine particles. The flow of nano-particles of fluids in a medium is the defined terminology of nano-particles flow in this paper.

Additive with particle size in the range of 1-100 nanometers available in drilling, completion, stimulation, drill-in or any others fluids used in the gas and oil exploration and exploitation, is what defined as nano-fluids. Physically, a dimension that is thousand millionths of a meter is the usual perspective of nano-particles size. 100 nanometer fibers or particles have diameters that are about 800 to 1000 times smaller than the diameter of a human hair and roughly equal to the width of 10 hydrogen atoms (Saeid et al. 2006). Nano fluids can be classified as advanced nano-fluid and simple nano-fluid with regards to the number of nano-sized additives available in the fluid. Advanced nano-fluids is the fluid with more than one nano-sized additive meanwhile simple nano-fluids is the fluid with one nano-sized additives. Nano-particles can be stand with multifunctional or single functional type. A multifunctional nano-additive can perform several jobs in the fluids systems to complete the functional tasks of the fluid with a dramatic reduction in total solids and chemical content of a mud and also the overall fluid cost. In nano-based smart fluids, it has the significantly higher functional ability with a reduction in overall fluid cost in spite of high cost of individual additive.

The laws that govern nanoscale material behavior are drastically different than the laws governing the macro and microscale behavior due to its size with the close proximity to the atomic scale compared to macros and micros that beyond colloidal sizes of nano-particles. This is reflected by the significantly different behavior of carbon nanotubes and fullrenes from those of graphite even though these materials were derived from the graphite mother source (Smalley and Yakobsonb, 1998 and Zhou et al. 2005). Characteristics such as nano-particles grain boundary, size of the particles surface are per unit/mass or volume, purity and perfection of the particles, and etc. will show the superiority comparison of nanomaterials compared with the

parent materials. The excellency of the unique properties own by the nanos that are non-existent in the mother source is the size dependent properties of the nanomaterial compare to the macro and micromaterials of the same mother source. Another magical tool when enable them to behave differently compared to the parent particles with regards of the influence of particle boundaries is the enhanced quantum effects of nano-sized particles. Comparing nanomaterials with the macro and micro materials of the same mother source especially with it extremely high surface area to volume ratio properties, it make them significantly low concentration of material but high interaction potentials in the system.

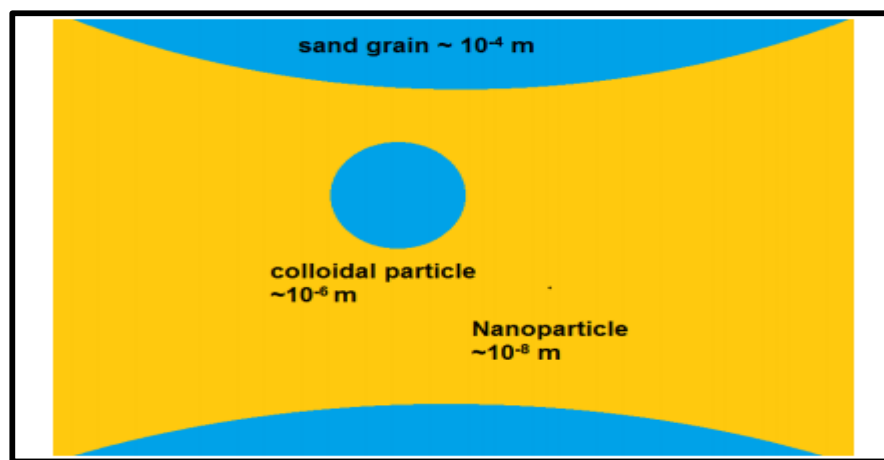


Figure 1 : Scale scan of sizes of nano-particles, colloid particle and sand grains

The engineering behind nano-particles has a lot of potential applications especially in oil reservoirs with its unique properties. For example, emulsions and foam can be stabilize with the surface active nano-particles for mobility control in improved oil recovery processes (Zhang et al., 2009). Tracer or sensor to detect certain reservoir fluid and rock properties can be made from functionalized nano-particles. The prerequisite for some of these applications is the ability for long distance propagation. Retention which is understood as the amount of particles retained in porous media during transport is the key parameter in term of identifying the transportability of nano-particles in porous media.

2.2 Porous Media

Porous media is a complex structure of pore bodies and throats covering a wide range of sizes. Numerous example of porous media are available. Porous or fissure

rocks, fibrous aggregates, soil ceramics, sand filters, filter paper and a loaf of bread are just a few. Heterogeneous or multiphase matter will occupy a portion of space in porous media. The least is one of the non-solid phases which might be gaseous or liquid phases should comprise in this matter. The 'matrix' or 'bulk' is the term for solid-grainy portion of the medium. Meanwhile void space is referring to the space that is not part of the solid matrix within the porous medium domain. Figure 50 in the appendix show the closer view of rock grains and its porosity.

Porosity, capillary tension, permeability, tensile strength, and etc. are among the properties characterized by the porous medium. In each elementary volume, the representative of solid phase must be present and it should be distributed throughout the porous medium within the domain occupied by a porous medium. Various openings comprising the relatively narrow void space in the porous is among another basic feature of the rock. Porous media usually has interconnected channels throughout the length and diameter that sometimes being termed as the effective pore space. The unconnected channel still considered as part of the solid matrix with regards of the fluid flow through porous media. As far as flow through the medium is concerned, certain portions of the interconnected pore space in fact, may be assumed as ineffective and a good example is dead-end pores (blind pores); no flows occur through them due to the channels or pores with only a narrow single connection of interconnected channel space. However, the existence of concept of closed or tight porosity in this medium should not be neglected. Acquiring a curve that lies completely within from any two points in the effective pore space is the another way to explain the porous media characteristic. Explain below is among the main factors that hugely influence the porous media.

a) Porosity

Porosity is the fraction of the pore or void space that occupying inside the bulk volume of the porous sample. "Porosity" is not a measure of void fraction but rather of void size which cause sometimes, this word is used inaccurately with a different meaning; for example, in the expression "graded porosity" of a filter consisting of layers of different grain sizes. The porosity may vary from near zero to almost unity depending on the porous medium type.

It is important to distinguish between two kinds of pore or void space, which called interconnected or effective pore space for the one with a continuous phase within the porous medium or “effective” pore space, and the other which cannot contribute to transport of matter is the one consists of dispersed “isolated” or “non-interconnected” pores or voids in the medium. Interconnected from one side only is called blind pores. They are unable to do transport even though they are penetrable.

b) Permeability

Permeability is the term used for the conductivity of the porous medium with respect to permeation by a Newtonian fluid. “Permeability”, used in this general sense, is of limited usefulness only because its value in the same porous sample may vary with the properties of the permeating fluid and the mechanism of permeation. It is both more useful and more scientific to separate out the parameter that measures the contribution of the porous medium to the conductivity and is independent of both fluid properties and flow mechanisms. This quantity is the specific permeability “k”, which in this monograph is called permeability, for short, unless otherwise stated. Its value is uniquely determined by the pore structure.

2.3 Transparent Micromodel

For the smallest scale relevant for the petroleum recovery, micromodels are the most suitable for experimental study of flow in porous media of pore scale. According to Department of Physics and Technology of Bergen University (2012), micromodels contain enclosed pore networks where flow can be observed visually in a microscope, making it possible to study how pore scale events affect flow patterns and displacement efficiency at larger scales and this visual studies enable several key features regarding fluid flow and recovery to be identified, and the effect of changing parameters like injection rate, types of injection fluids and pore properties to be observed. A paper by Hardy (1986) describe micromodel as the set of interconnected pores which flow resistance of porous media is assigned to pore connectors and the pore volume of porous media is assigned to the pores as the description of the porous model. By dividing the flowing fluid into discrete fluid elements, the model will

simulates fluid flow perfectly. Meanwhile Wan et. al (1996) provided that creating photographically printing a pair of mirror images of a pore network pattern on two layer plates, followed by chemically etching the pattern onto the plates and then fusing them together, the sample of good micromodel will be formed which is transparent and two-dimensional representations of porous medium networks. Transport of various fluid phases and solutes are permitted through the inlet and outlet port micromodel making the channel network and pore scale phenomena observation possible. Conclusively, transparent micromodel can be used to simulate transport processes at the pore scale by reproducing the resemblance with porous media properties in its domain, either natural or synthetic. In simpler term, the complexity of porous materials either natural or artificial that can be captured by networks of pores is what defined micromodel in true form.

Micromodel consist of a digitally or manually constructed pore network etched into a material and enclosed by a transparent surface which typically is glass or plastic. It can be constructed from 2D thin-sections of real porous rocks or manually after a desired design by using certain software or manual method to represent a realistic pore structure. The first method is normally preferred when to study fluid flow in real porous rocks is the objective, while the second method is used for elementary study of flow physics with controlled flow conditions. A wide range of values concerning pore and grain sizes, porosity, permeability, grain surface roughness, wettability, and shapes, the average number of flow entrance/exit paths from pores in the network, and pore network size and coordination number can be made in the micromodel. (Martin, 2014).

The ability of micromodel to enable fluid flow to be studied visually is obviously a huge advantage for research. Figure 5 in the appendix show among the sample of procedure in micromodel fabrication. However, the conductance and trapping of fluids in real porous media will be hugely affected due to micromodel inability to realize real dimensionality of real porous media in 2-dimension micromodel as some pores in the real porous media that are connected in 3-dimension will be isolated in 2-dimensional version of micromodel. This again does limit the ability to perform direct measurement of many relevant flow properties like relative permeabilities and fluid saturations due to this restrictions put to the dimensionality of micromodel. A pore network with a high coordination number will be less affected by the reduced dimensionality.

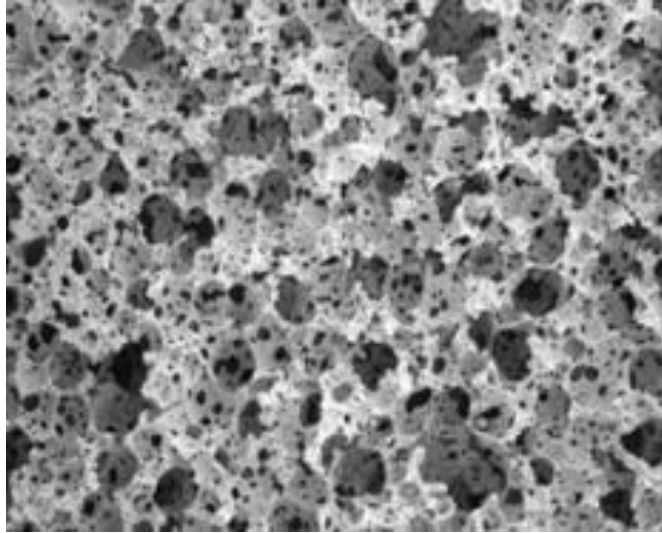


Figure 2 : Micro-scale view of the rock

It should be understood that the micromodel is exposed to several limitations; not suitable for experiment with high pressure and temperature due to material limitation and typical micromodel is just suitable with little number of inlet/outlet port of fluid which drastically can give much impact on experiment with regard of change of fluids in the flow into porous media.

Chapter 3 : Methodology

3.1 Research Methodology

Figure below show the research methodology for this project.

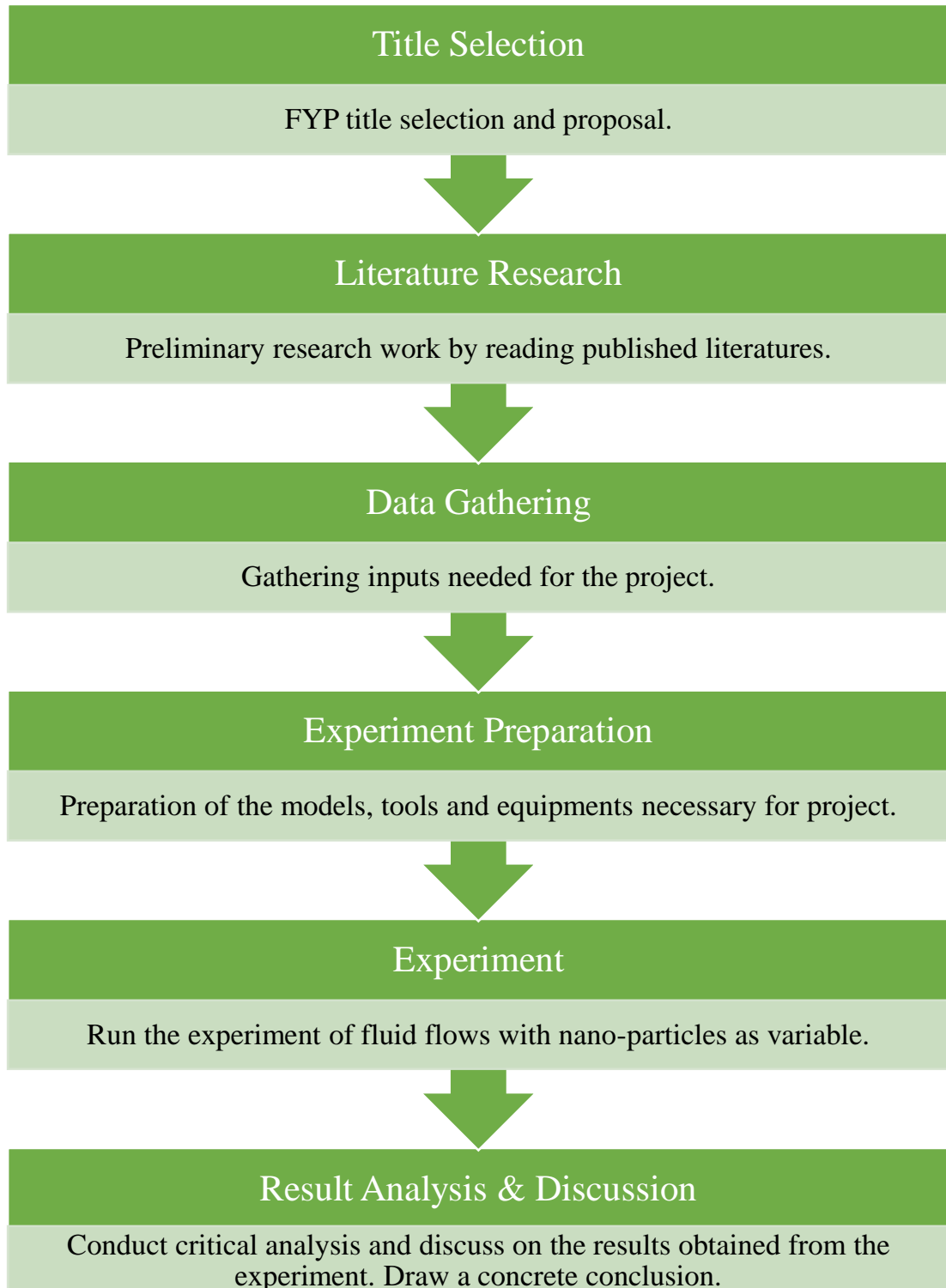


Figure 3 : Schematic diagram of project flow

The subsequent paragraphs describe the methodology of this FYP in brief. Following the selection of FYP title, the project started with the literature review of the SPE papers and other online journals related to visualization of fluids flow, nano-particles and its effects, micromodels, microscope, and nano-particles researches done by the previous engineers and researchers. The objective of this stage is to gain thorough understanding on the concept of project and thus forming strong basic knowledge to assist the future study.

The next stage is to collect the parameters and data for the inputs for the studies, mostly from literature review of the published papers. The data collected will be put into the scope of study for further analysis towards the development of this project. The inputs from the key personnel such as UTP researchers, lecturers and technicians also have been contributed much towards this stage. Important aspects of the project have been recognized and thoroughly emphasize during the time of project.

After the preparation of the models and chemicals, required tools and equipments, the project experiment was conducted as per objectives. The determination of suitable micromodel and fluid flows experiment with the nano-particles as the key variable are the aim in this stage. The data was collected through the microscope and capture images. Subsequently, the critical analysis of the results was carried out and the conclusion would be drawn upon.

Finally, the literature reviews, simulation works, research outcomes, findings and discussion will be documented in the Final Report.

Basically, the project activities for this FYP can be generalized into few groups;

- a) Literature review and data gathering
- b) Experiment
- c) Analysis
- d) Documentation

The first item that is literature review and data gathering was conducted in FYP 1 and the rest of stages were carried out in FYP 2.

3.2 Procedures

In order to successfully complete the project, series of steps and procedures are identified earlier. Below is the research procedures with description of each of the phases happen in the project.

3.2.1 The Construction of Micromodels

Few type of micromodels have been constructed to meet this objectives. Each model are developed differently in terms of their material, grain/pore structure and process of construction. The micromodel created must resemble as much as possible like the rock in the reservoir to be selected as the porous media for the next stage of the experiment. Among the elements prioritized in this process are;

- Porosity
- Visibility

Since there are few limitation in this micromodels construction such as minimum porosity value, pressure, and temperature, therefore the emphasize of the porosity is not highly stress much in this micromodel as long as it can still be come a good factor in the micromodel testing. The parameters that become variable in the construction of the transparent micromodel are the type and size of the grains.

Three types of micromodel has been made and among of them are;

- a) Glass micromodel with medium size (15mm – 50mm) of sand grains

Procedures of constructions

- i. Few materials were prepared;
 - Glass slides with dimension 2.5cm x 7.5cm

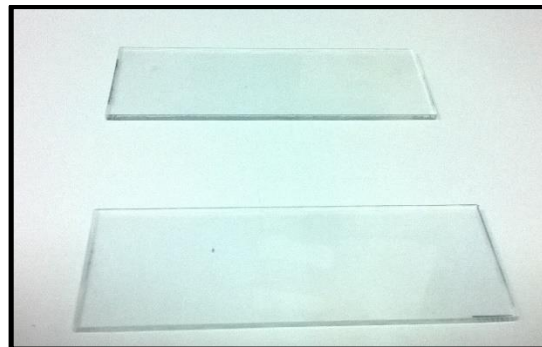


Figure 4 : The glass slides

- Sand grains

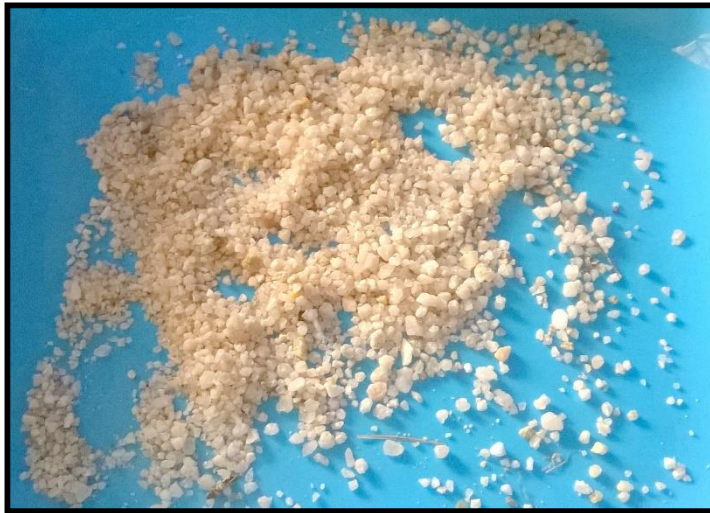


Figure 5 : Sand grains

- Silicon glue



Figure 6 : Silicon glue with gun

- Syringes and needles



Figure 7 : Syringes



Figure 8 : Needles

ii. The glass was put on the flat and horizontal surface. The needles will be glued first to the each end of the short sides of the glass (the ones with 2.5cm length). The reason why needles were put on the short sides of the glass is to increase the coverage area of fluid flows so it can move inside the micromodel, wholly.

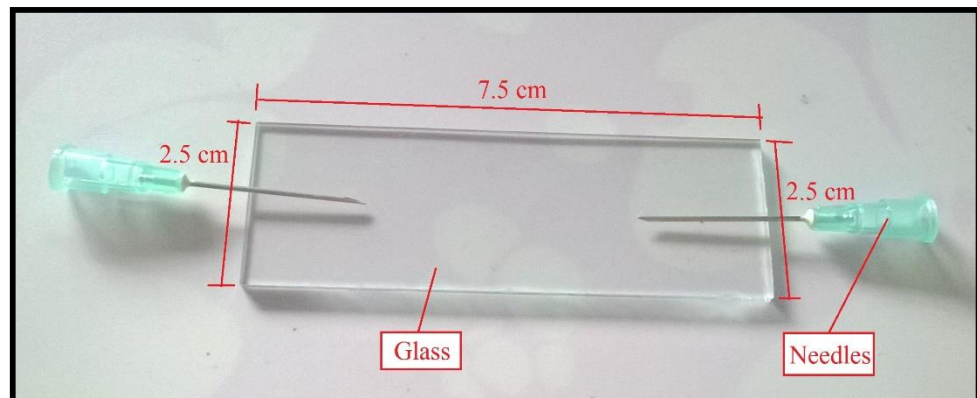


Figure 9 : Needles being glued on the glass slides

iii. The sand grains would be put on top of the glass surface. Another glass slide was put on top of this slide with the sand grains are between them. These slides will be sealed on the both short sides and one long side of the glass with silicon glue.



Figure 10 : The needles were glued

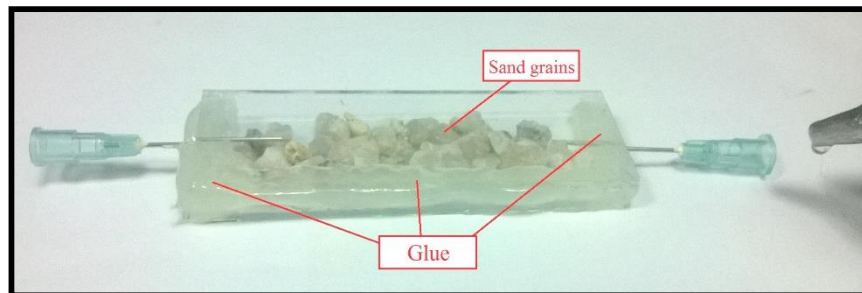


Figure 11 : Both glass slides has been sealed with glue

iv. More sand grains were added into the inner of glass layer through the available opening and being compressed later on. After the grain sands have been compact enough, the remaining opening was sealed with silicon glue. Any leakage on the seal would be checked and immediately patch with the glue so that no fluid loss will happen when fluids being flow inside of the micromodel.



Figure 12 : After the sand compaction and being sealed completely

b) Glass micromodel with small size (0.5mm – 10mm) of sand grains

Procedures of constructions

i. Few materials were prepared;

- Glass slides with dimension 2.5cm x 7.5cm
- Sand grains
- Silicon glue
- Syringes and needles

ii. The glass was put on the flat and horizontal surface. The needles will be glued first to the each end of the short sides of the glass (the ones with 2.5cm length). The reason why needles were put on the short sides of the glass is to increase the coverage area of fluid flows so it can move inside the micromodel, wholly.

iii. The sand grains would be put on top of the glass surface. Another glass slide was put on top of this slide with the sand grains are between them. These slides will be sealed on the both short sides and one long side of the glass with silicon glue.

iv. More sand grains were added into the inner of glass layer through the available opening and being compressed later on. After the grain sands have been compact enough, the remaining opening was sealed with silicon glue. Any leakage on the seal would be checked and immediately patch with the glue so that no fluid loss will happen when fluids being flow inside of the micromodel.



Figure 13 : Micromodel with smaller sand grains

- c) Glass micromodel by using beads (40mm-50mm) as the grains for the porous media

Steps of construction;

- i. Few materials were prepared;
 - Glass slides with dimension 2.5cm x 7.5cm

- Beads



Figure 14 : Beads

- Silicon glue
- Syringes and needles

ii. The glass was put on the flat and horizontal surface. The needles will be glued first to the each end of the short sides of the glass (the ones with 2.5cm length). The reason why needles were put on the short sides of the glass is to increase the coverage area of fluid flows so it can move inside the micromodel, wholly.

iii. The beads would be put on top of the glass surface. Another glass slide was put on top of this slide with the beads are between them. These slides will be sealed on the both short sides and one long side of the glass with silicon glue.

iv. More beads were added into the inner of glass layer through the available opening and being compressed later on. After the beads have been compact enough, the remaining opening was sealed with silicon glue. Any leakage on the seal would be checked and immediately patch with the glue so that no fluid loss will happen when fluids being flow inside of the micromodel.



Figure 15 : Micromodel constructed with beads

After the micromodels have been constructed, the comparison between the micromodels will be carried out in determination of the best micromodel to be used in next stage. The main parameters influencing the selection are;

- Porosity (as low as possible)
- Still have good visibility under the microscope observation

The comparison will be carried out qualitatively since it is sufficient for this level of project. The reason for the validity of this qualitative method is because it will make the process in this stage run smoothly without spending much time and these micromodel generated is not really sensitively affected by many key variables such as permeability, temperature and pressure change when conducting the experiment.

After the micromodel type has been selected, few more micromodels were constructed using the same concept. These micromodels will be used in later stages which would require the usage of a lot of same micromodels.

3.2.2 Flowing the Fluid With or Without Nano-particles Into the Micromodels and Conducting the Observation under Optical Microscope

Since the micromodel should have few openings or ports made available during the construction, these openings should be connected to the fluid container. However, as being stated by Javadpour (2007), to provide for more than one inlet and one outlet port in a micromodel is extremely difficult due to the material limitation and problem

with fluid flow transition as to make the model behave accordingly as good as a real porous media.

This limitation resulted in large uncertainties when a change in injection fluid occurred in the transition between fluids starting from the first contact in the tubing. The fluid in the tubing (before entering the pores) represents a volume hundred times larger than the pore volume of the model which may cause contact zone time lag when the nano-particles reach the pore space area through the fluid in channel. What is mean by contact zone time lag is the distance for the two fluids travel will be very long before mixing properly and this might cause disturbance in seeing the real effect of nano-particles towards fluid in the pores. Therefore, the development of syringe injection method for desirable injection rate is the most optimum way to overcome radical nano-particles flowrate injection problem with the current limitation. The fluid flowrate will not be a problem here since it is not very rapid that may cause damage to the micromodel if carry out properly.

The fluid and nano-particles will be flown throughout the micromodel and the fluid flow will be observed and the images of it will be taken. This should be done macroscopically. Thus, visualization of nano-particles flow through porous media in transparent micromodel will be achieved. The fluid flow are re-run again under various conditions of model as to simulate the reservoir state as near as possible such as by making it oil-wet (residual oil) and water-wet.

In this stage of experiment, there are two set of experiments were conducted to in achieving the objectives of visualizing the nano-particles effect towards fluids flow in transparent micromodel. Both will use the nano-particles presences as the variable in the experiment. The set of experiments are;

- Comparison of tests with water displacing crude oil at connate water saturation with and without the presence of nano-particles.
- Comparison of tests with diluted surfactant in water solution displacing crude oil at connate water saturation with and without the presence of nano-particles.

Therefore, both set of experiments (water and water+surfactant solution) will be experimented with and without the presence of nano-particles condition as the

main purpose of this stage is to observe the nano-particles effect towards fluid flow (although in different type of fluids state).

The procedure of the each set of the experiment is presented below;

- a) Water displacing crude oil at connate water saturation with and without the presence of nano-particles tests.
 - i. The micromodels, chemicals as per requirement, and equipments were prepared as listed below;
 - Micromodels
 - Dyed-water (blue)
 - Crude oil
 - Nano-particles powder
 - Fluid containers/beakers
 - Syringes
 - Optical microscope with camera
 - Computer



Figure 16 : (From the right) Crude oil, dyed-water and dyed-water with nano-particles



Figure 17 : Fluid containers



Figure 18 : Optical microscope with built-in camera

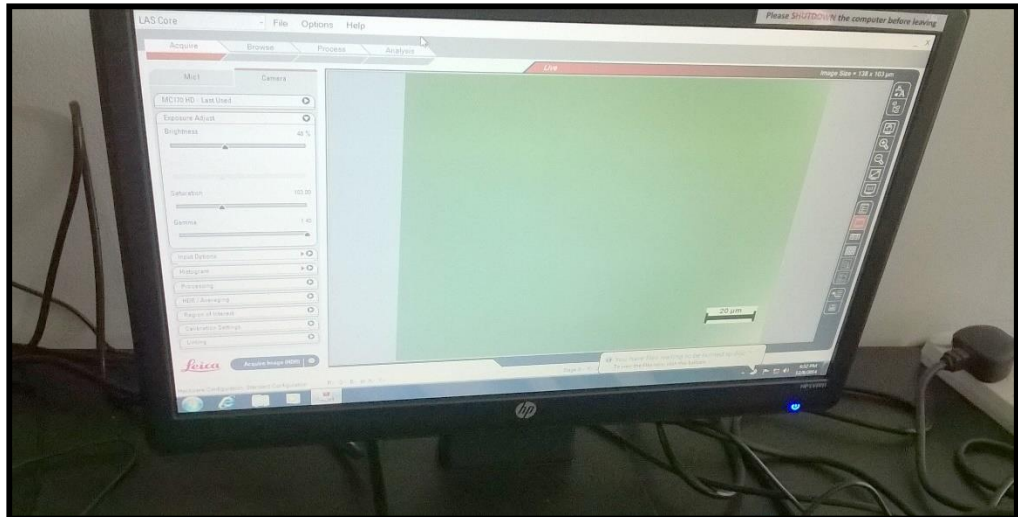


Figure 19 : Computer with microscope software

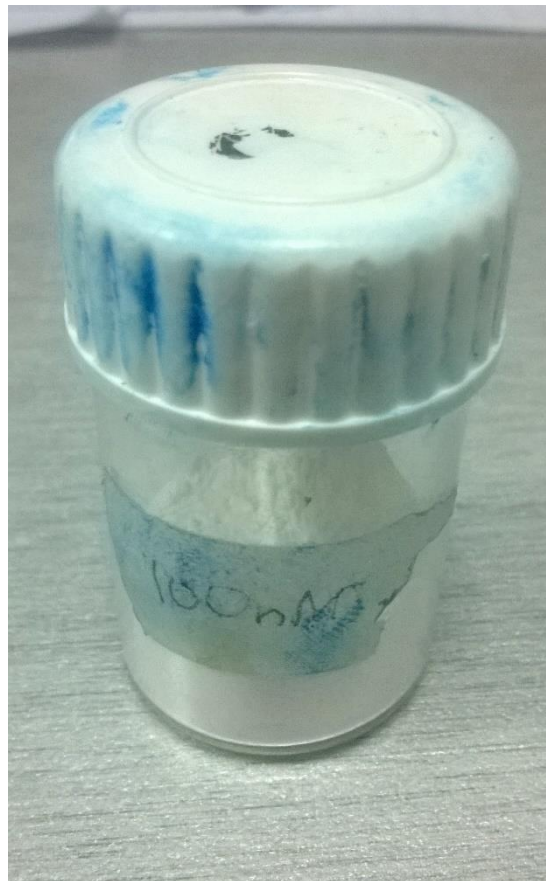


Figure 20 : Nano-particles

- ii. The micromodel was placed under the microscope lens and the part of micromodel that will be observed is chosen. Then, the micromodel was clipped properly.



Figure 21 : Micromodel being placed inside the microscope

- iii. One syringe containing 5 ml of dyed-water is connected in the chosen inlet port of the micromodel. Another syringe tube is connected in outlet port as for outlet flow of the fluid and connected to a fluid contained to receive used fluids. (avoid spillage)



Figure 22 : Syringe containing dyed-water (blue) was connected to the inlet port of micromodel

- iv. The dyed-water was slowly injected into the micromodel. The fluid flow was observed. The image of the flow condition in pore-level was projected in the computer using the microscope camera and being recorded.



Figure 23 : The view of micromodel pores condition using the computer software

- v. The inlet connection was replaced with the syringe containing 5 ml of crude oil. The crude oil was slowly injected into the micromodel and will displace the dyed-water in the micromodel pores as to form a condition of high saturation of crude oil with connate water. The fluids flow was observed and the images were recorded.
- vi. The syringe was again being replaced with the syringe containing 5 ml of dyed-water. The dyed-water was slowly injected into the micromodel and will displace the crude oil saturation in the micromodel pores. The fluids flow was observed and the images were recorded.
- vii. Repeat step (ii) until step (v) again but this time, the micromodel was replaced with the new one.
- viii. The syringe was again being replaced with the syringe containing 5 ml of dyed-water with nano-particles. The dyed water with nano-particles was slowly injected into the micromodel and will displace the crude

oil saturation in the micromodel pores. The fluid flow was observed and the images were recorded.

- b) Diluted surfactant in water solution displacing crude oil at connate water saturation with and without the presence of nano-particles tests.
 - i. The micromodels, chemicals as per requirement, and equipments were prepared as listed below;
 - Micromodels
 - Dyed water (blue)
 - Water with 10% concentration of surfactant
 - Crude oil
 - Nano-particles powder
 - Fluid containers/beakers
 - Syringes
 - Optical microscope with camera
 - Computer
 - ii. The micromodel was place under the microscope lens and the part of micromodel that will be observed is chosen. Then, the micromodel was clipped properly.
 - iii. One syringe containing 5 ml of dyed-water is connected in the chosen inlet port of the micromodel. Another syringe tube is connected in outlet port as for outlet flow of the fluid and connected to a fluid contained to receive used fluids. (avoid spillage)
 - iv. The dyed-water was slowly injected into the micromodel. The fluid flow was observed. The image of the flow condition in pore-level was projected in the computer using the microscope camera and being recorded.
 - v. The inlet connection was replaced with the syringe containing 5 ml of crude oil. The crude oil was slowly injected into the micromodel and will displace the dyed-water in the micromodel pores as to form a condition of high saturation of crude oil with connate water. The fluids flow was observed and the images were recorded.
 - vi. The syringe was again being replaced but this time with the syringe containing 5 ml of dyed-water added with dilution of surfactant. The

dyed-water added with dilution of surfactant was slowly injected into the micromodel and will displaced the crude oil saturation in the micromodel pores. The fluids flow was observed and the images were recorded.

- vii. Repeat step (ii) until step (v) again but this time, the micromodel was replaced with the new one.
- viii. The syringe was again being replaced but this time with the syringe containing 5 ml of dyed-water added with dilution of surfactant combined with nano-particles. The dyed-water added with dilution of surfactant combined with nano-particles was slowly injected into the micromodel and will displaced the crude oil saturation in the micromodel pores. The fluids flow was observed and the images were recorded.

3.3 Gantt Chart

Table below show the targeted achievable timeline for Final Year Project (FYP) course of the project entitled, Visualizing Nano-Particles flow in Porous Media by using Transparent Micromodel. The week 1 is starting from 9 June 2014 until the end of week 24 of 27 December 2014.

Task	Week										
	1	2	3	4	5	6	7	8	9	10	11
FYP 1 briefing	●										
Selection of project	■	■	●								
Literature research			■	■	■	■	■	■	■	■	
Gathering data about the nano-particles and micromodel				■	■						
Derive the methodology for the research					■						
IRC briefing					●						
Submission of extended proposal						■	●				
Proposal defence						■	●				
Continue the research about methodology								■	■	■	
Searching the materials and equipments for research experiment in FYP 2										■	■
Interim report											■

- - Deliverable
- - Progress

Task	Week												
	12	13	14	15	16	17	18	19	20	21	22	23	24
FYP II Briefing	●												
Continue research													
Preparing the materials and equipments and constructing the micromodels													
Interim Report						●							
Conducting the experiments													
Pre-SEDEX/poster presentation													
Completing and submission of the Final Draft													
Viva													
Hardbound copies submission													

- - Deliverable
- - Progress

Chapter 4 : Result and Discussion

4.1 Result

The result that has been generated after the experiment will be thoroughly discussed here. It will be presented with respect to the each objectives.

4.1.1 The Selection of the Best Porous Media

As had been discussed in previous methodology part, few micromodel has been constructed with each having different structure in terms of their material, grain/pore structure and process of construction. The micromodel created must resemble as much as possible like the rock in the reservoir to be selected as the porous media for the next stage of the experiment. The two important parameters involved in the consideration the porosity and the visibility; porosity must be as low as possible while visibility is still maintained under the lens of microscope.



Figure 24 : Micromodel with small size grains (above), micromodel with medium size grains (middle), and micromodel with large size grains (bottom)

The comparison was made qualitatively with the interpretation from visual observation. As seen above, the small size grains micromodel (sand) has the lowest porosity compare to medium size grains micromodel (sand) and medium size but regular-in-shape grain micromodel (bead). When testing the visibility under microscope, the micromodel with the lowest porosity still show a good visibility and good porous images. This match the criteria needed for the selection of micromodel. Hence, the micromodel with small size sand grains has been chosen as the prefer micromodel.

4.1.2 The Effect of Nano-particles towards Fluids Flow

This section is divided into two parts;

- i. Comparison of tests with water displacing crude oil at connate water saturation with and without the presence of nano-particles.
- ii. Comparison of tests with diluted surfactant solution displacing crude oil at connate water saturation with and without the presence of nano-particles.

After carried out the experiment, each set of the test has shown few encouraging result in the experiment. Before going to the elaboration of the test results, the results data are shown first in the next section.

1. Water displacing crude oil at connate water saturation with and without the presence of nano-particles tests

The displacing fluids chosen in this part is water. At first, the micromodel has been flown with a dyed-water (blue) without nano-particles. The fluid flow has been observed in few different parts in the micromodel. Then, the micromodel will be flown by the crude oil to observe on how the fluid flow should happening in the pores inside micromodel with the crude oil displacing the water, thus creating connate water saturation. Finally, the crude oil saturation inside the micromodel will be displaced back by the dyed-water to see on how the fluids flow happening this time without the presence of nano-particles.

After obtaining the result of water displacement flow, then the experiment is proceeding to the next objective; observing the effect of nano-particles towards fluids flow. Repeating again the steps above with the new same type micromodel. But, the crude oil saturation will be displaced by the dyed-water with nano-particles concentration in the end. Upon completing this sets of experiments, comparison with the results obtained. The observation images that have been captured are shown below;

(a) Before the flow of fluid

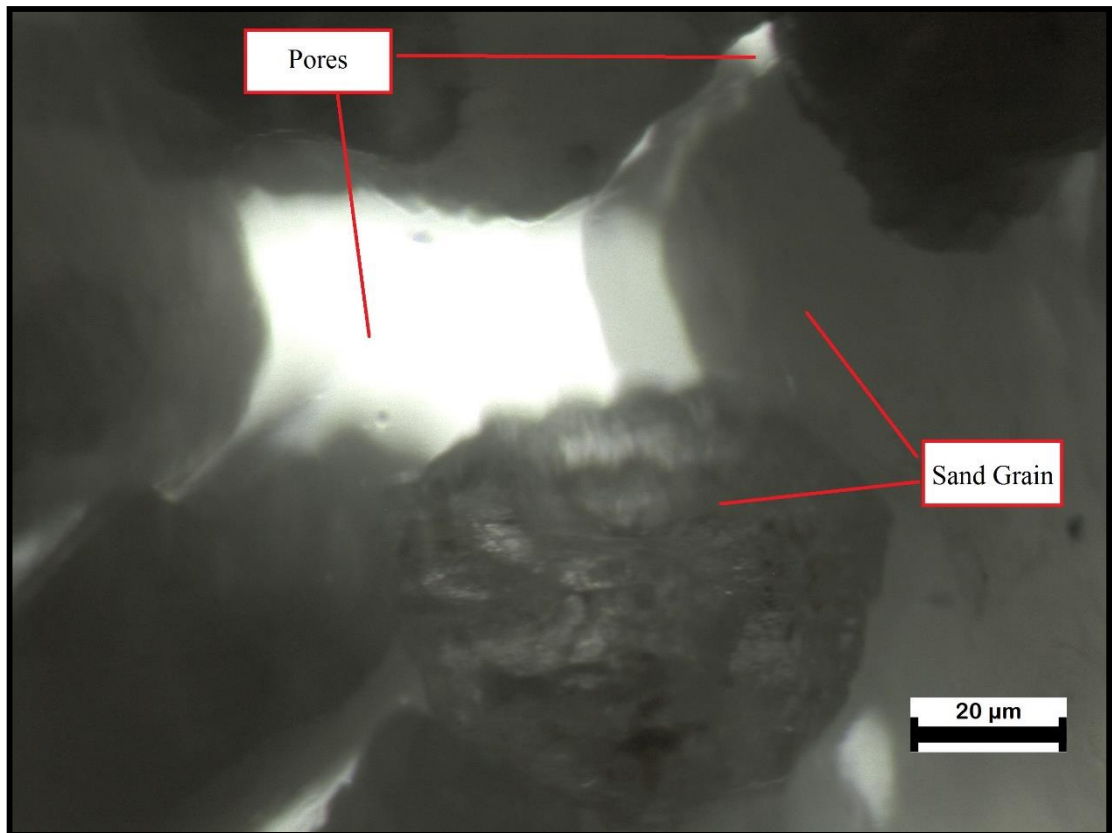


Figure 25 : The picture of pores inside the micromodel without any fluids at grain scale (using microscope)

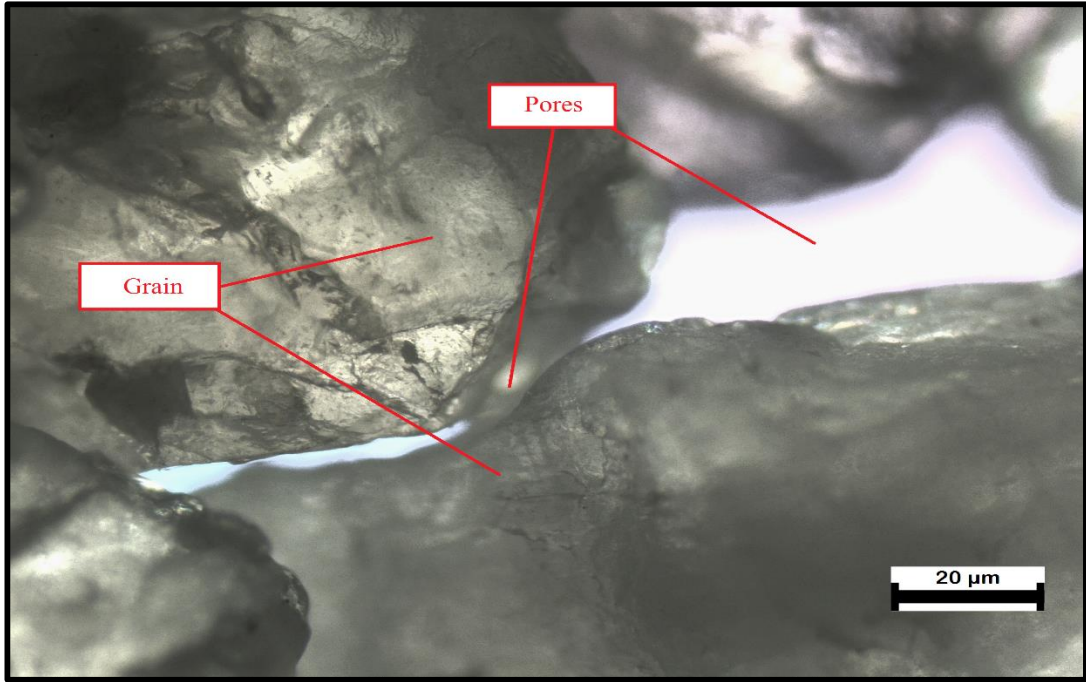


Figure 26 : Other part of the micromodel view with microscope at grain scale (no fluids)

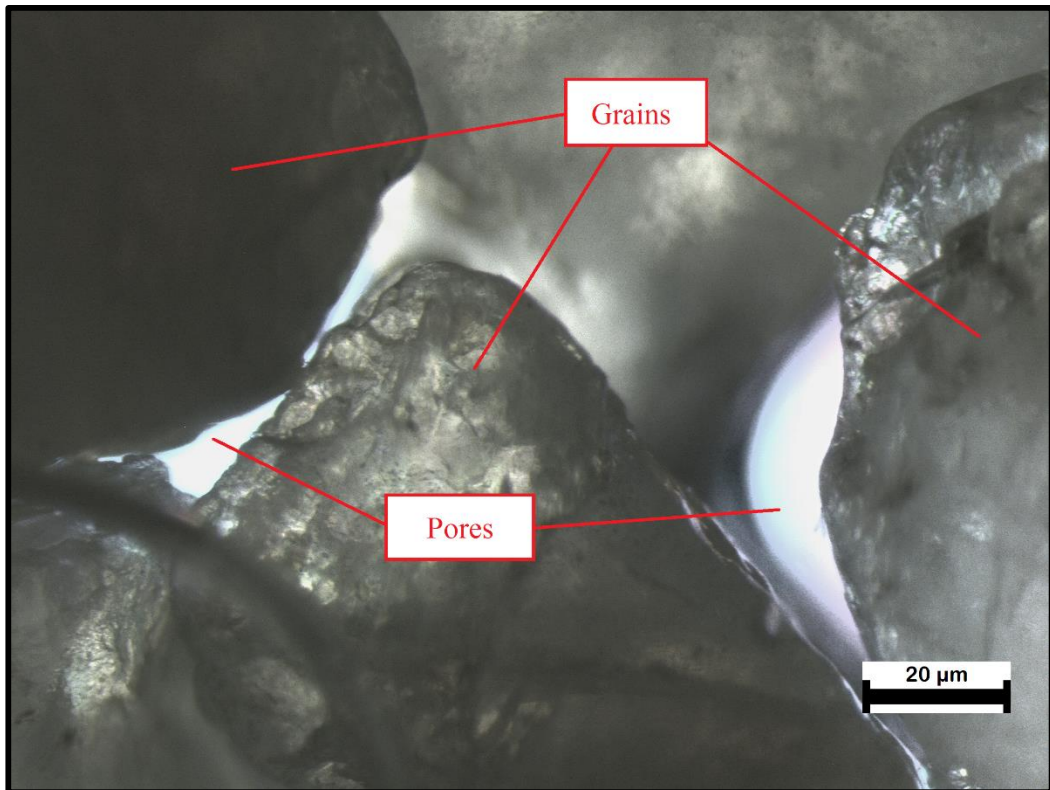


Figure 27 : Other part of the micromodel view with microscope

(b) The first flow of fluid (by dyed-water)

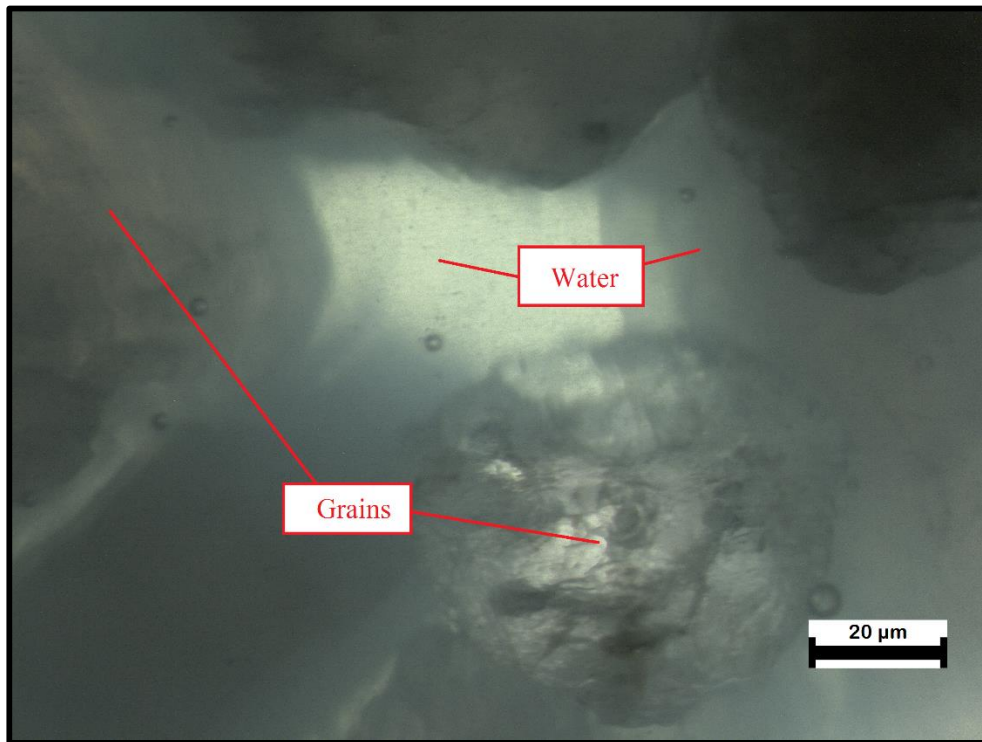


Figure 28 : The picture of dyed-water flow through the pores between the grain (with respect to the image of figure 25)

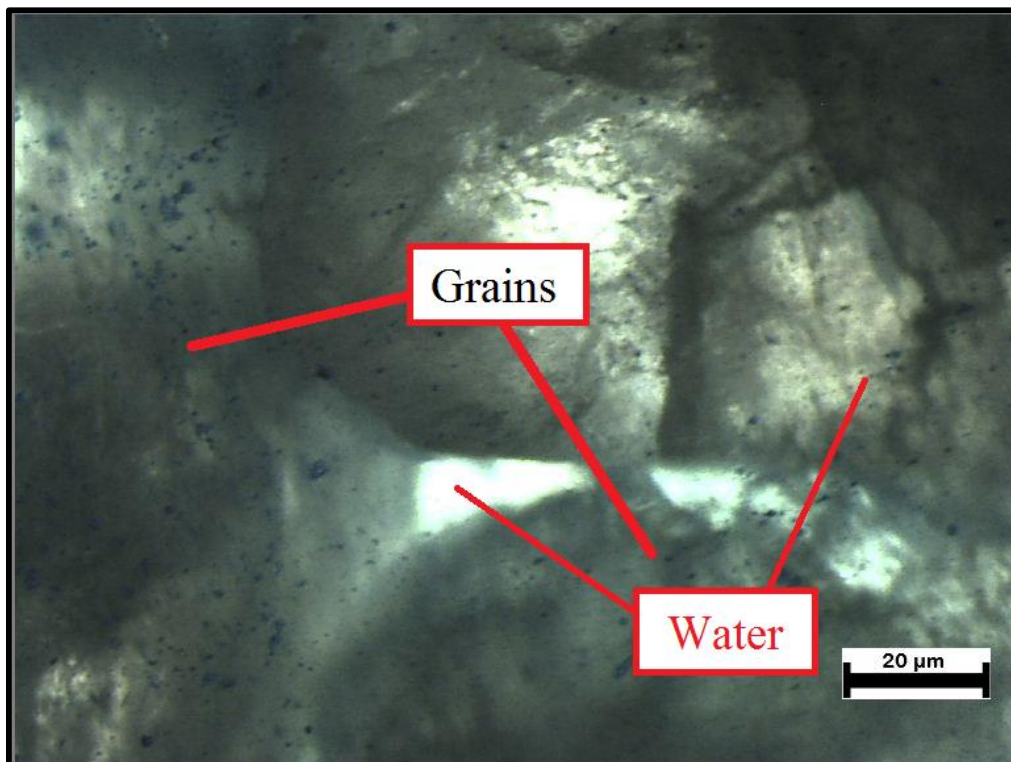


Figure 29 : The flow of dyed-water in other part of the micromodel

(c) Displacement of crude oil with dyed-water without nano-particles

Before

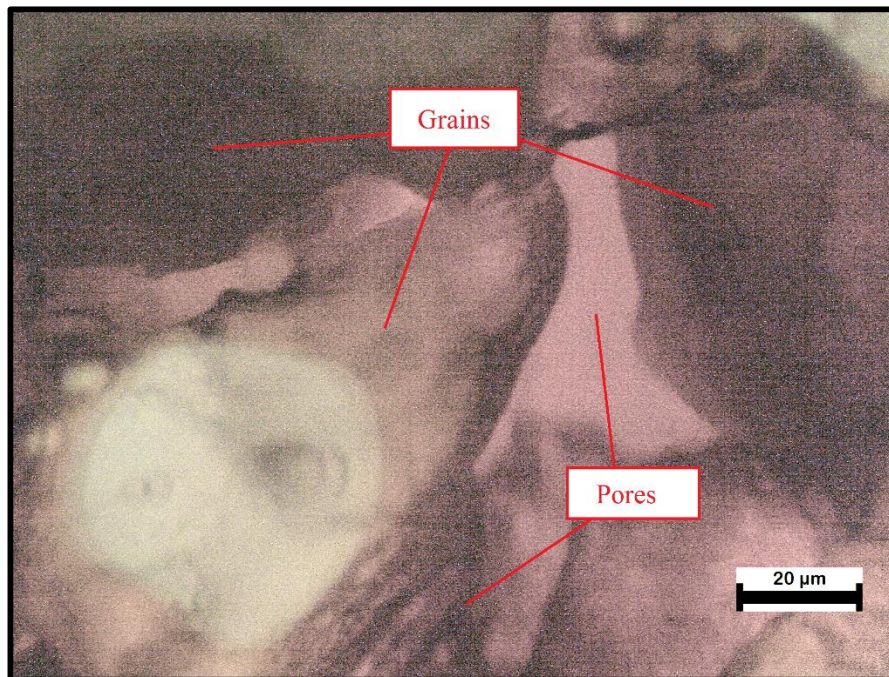


Figure 30 : The image of crude oil occupying the micromodel pores (using microscope)

After

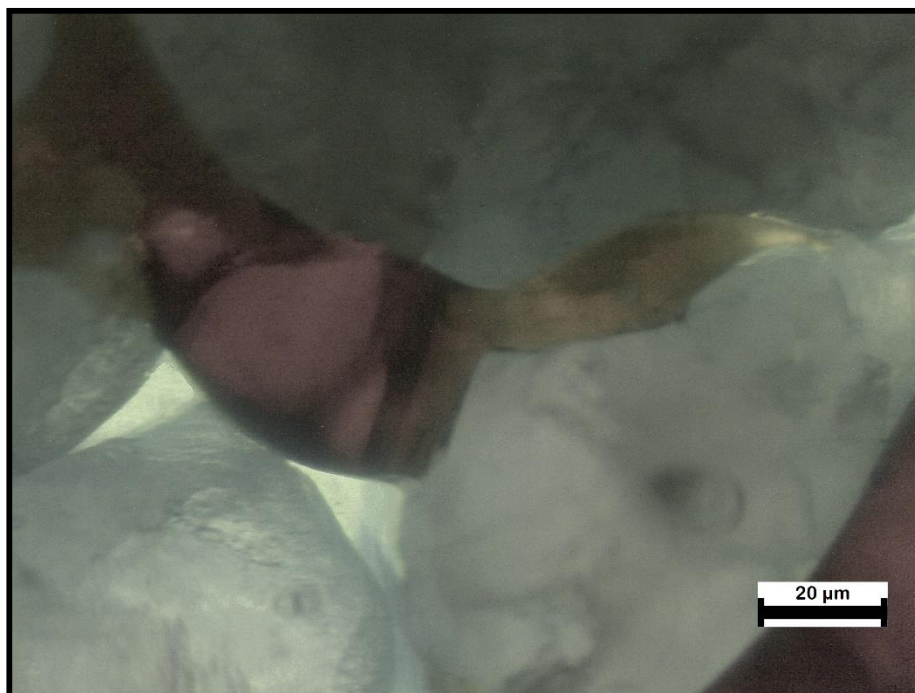


Figure 31 : The displacement of crude oil with dyed-water (without nano-particles)

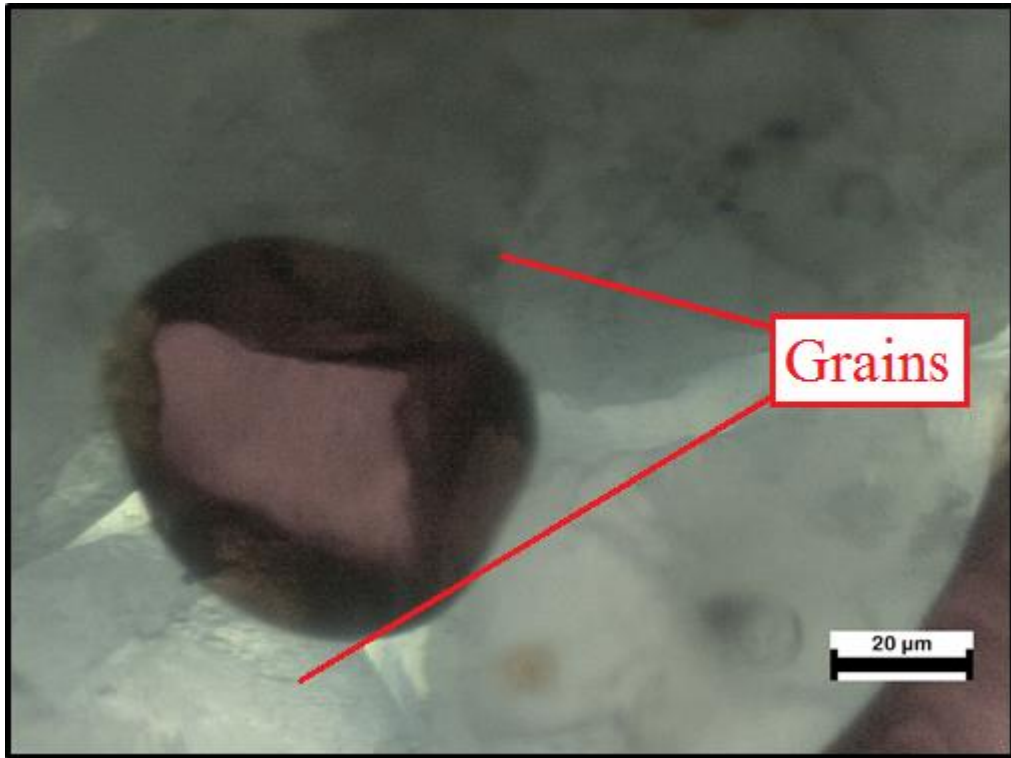


Figure 32 : The displacement of crude oil with dyed water (without nano-particle)

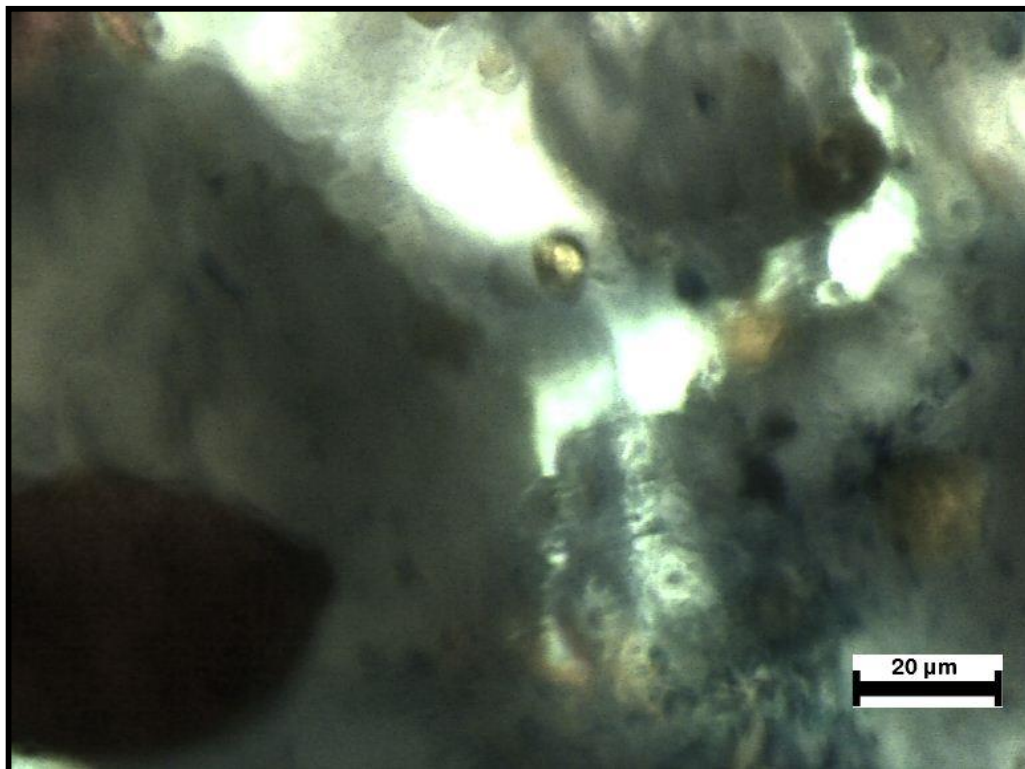


Figure 33 : The displacement of dyed-water without nano-particles (at other part of micromodel)

(d) Displacement of crude oil with dyed-water with nano-particles

Before

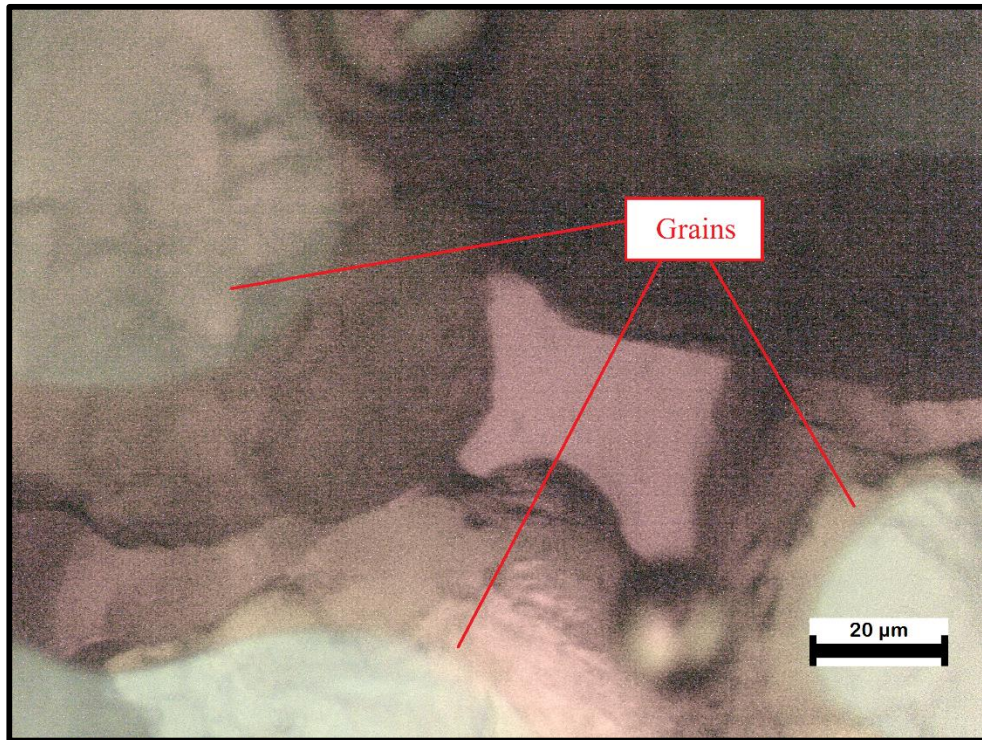


Figure 34 : The image of crude oil occupying the micromodel pores

After

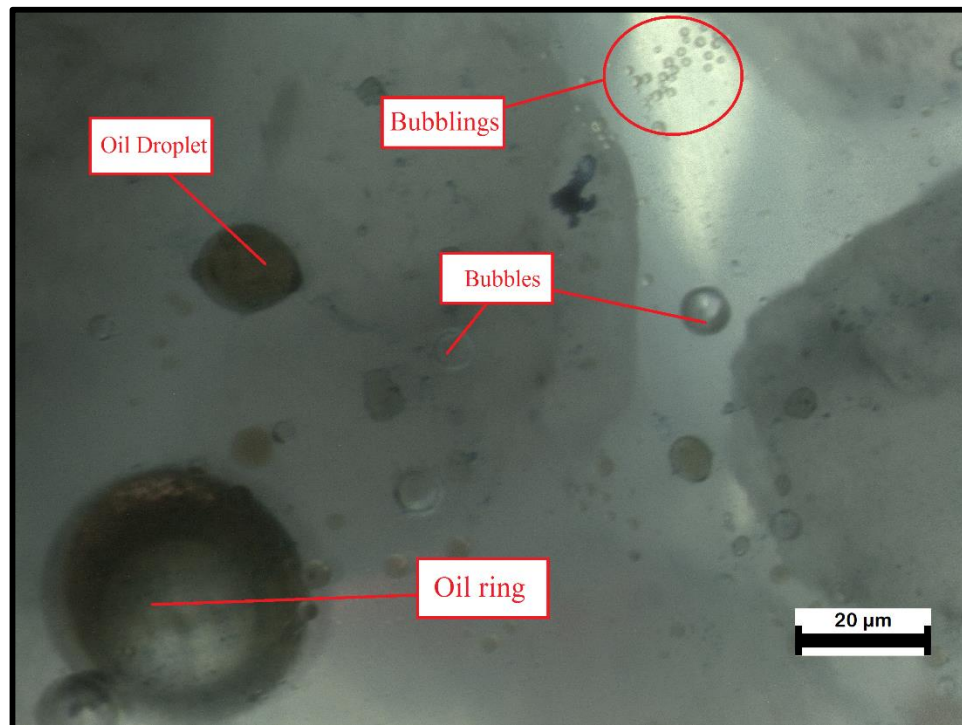


Figure 35 : The displacement of crude oil with dyed water + nano-particle

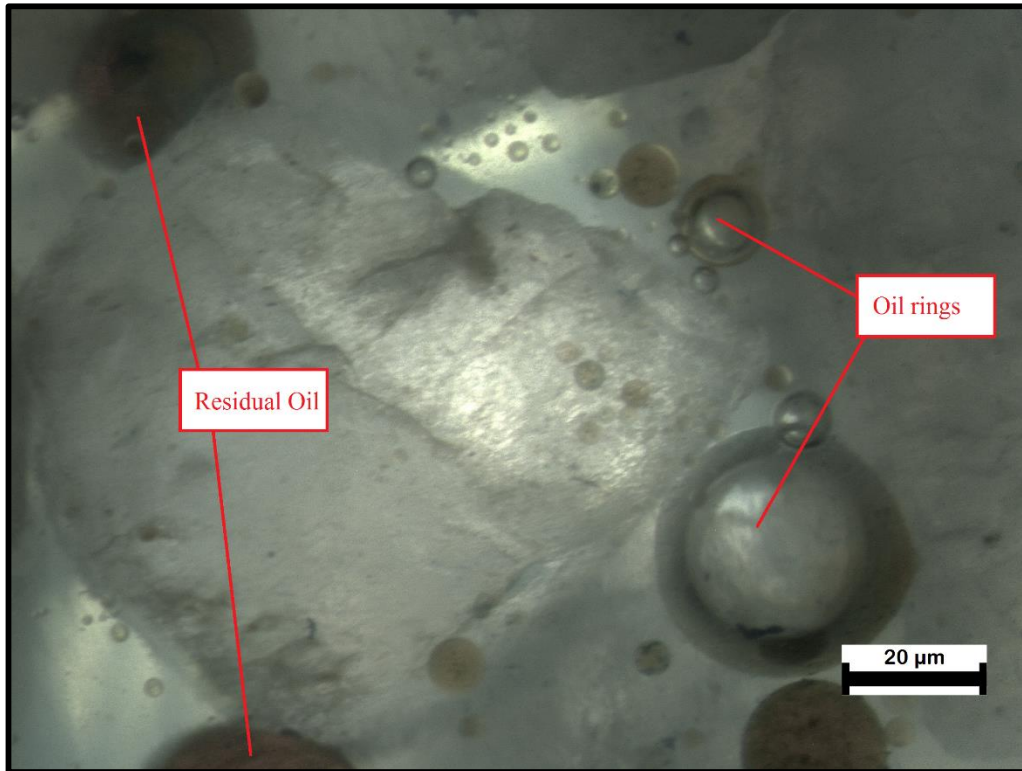


Figure 36 : The displacement of crude oil with dyed water + nano-particle (at other part of micromodel)

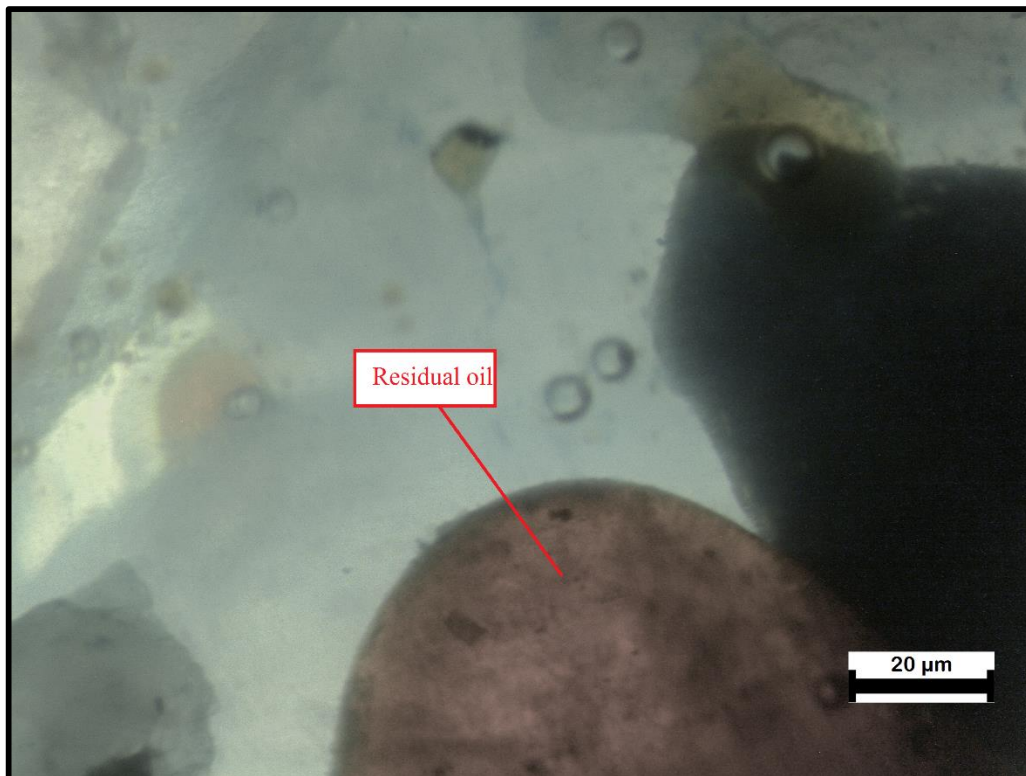


Figure 37 : The displacement of crude oil with dyed water + nano-particle (at other part of micromodel)

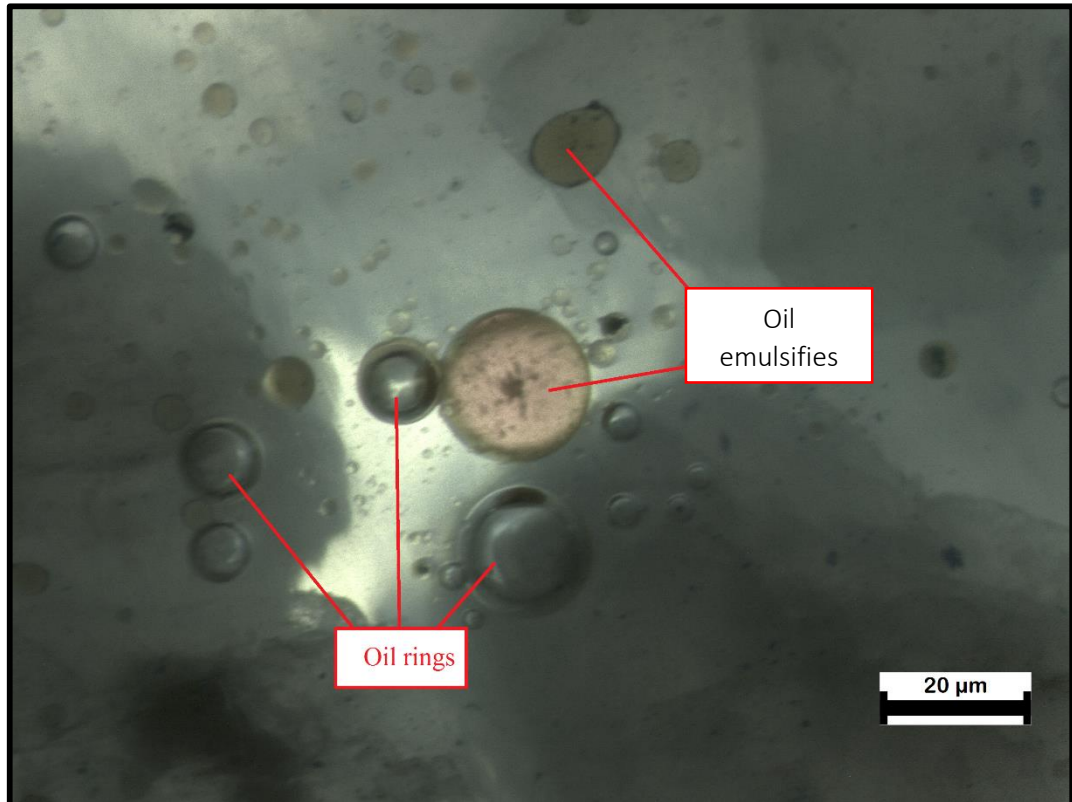


Figure 39 : The displacement of crude oil with dyed water + nano-particle (at other part of micromodel)

2. Diluted surfactant solution displacing crude oil at connate water saturation with and without the presence of nano-particles tests

Basically, the concept of the tests applied in this part is the same as the previous one. The difference is the parameter involved which in previous part does not include the dilution of surfactant as the parameter of study. In this part, the effects of nano-particles towards fluids flow that comprise of surfactant too was observed. The behavior of fluids flow as stated below;

- (a) Before the flow of fluid
- (b) The first flow of fluid (by dyed-water)

were showing the similar behavior of the fluid flows as the being shown in the images recorded on the computer. The simplification is being made that the similar process of fluids flow happening in theses stages are the same with the previous part of study due to the same parameters, properties and variables involved.

(c) Displacement of crude oil with 10% of diluted surfactant in dyed-water without nano-particles

Before

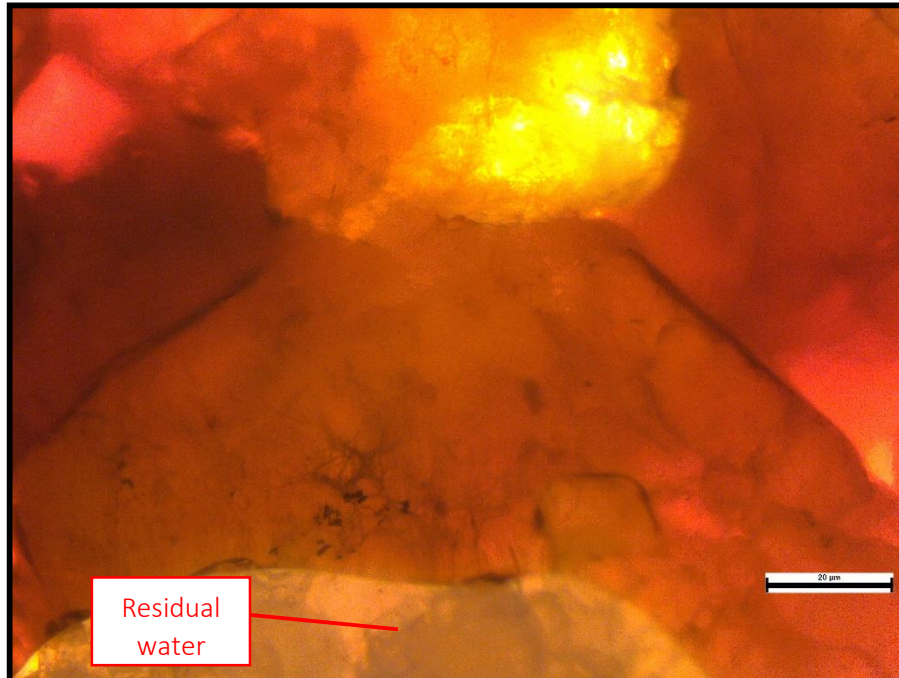


Figure 39 : The image of crude oil occupying the micromodel pores

After

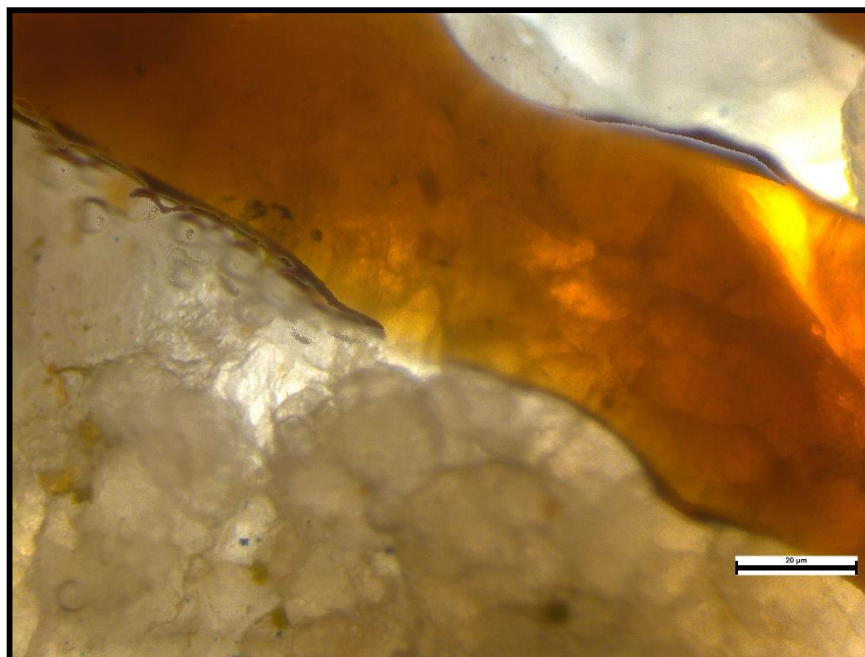


Figure 40 : The early phase of crude oil displacement by diluted surfactant in dyed-water solution

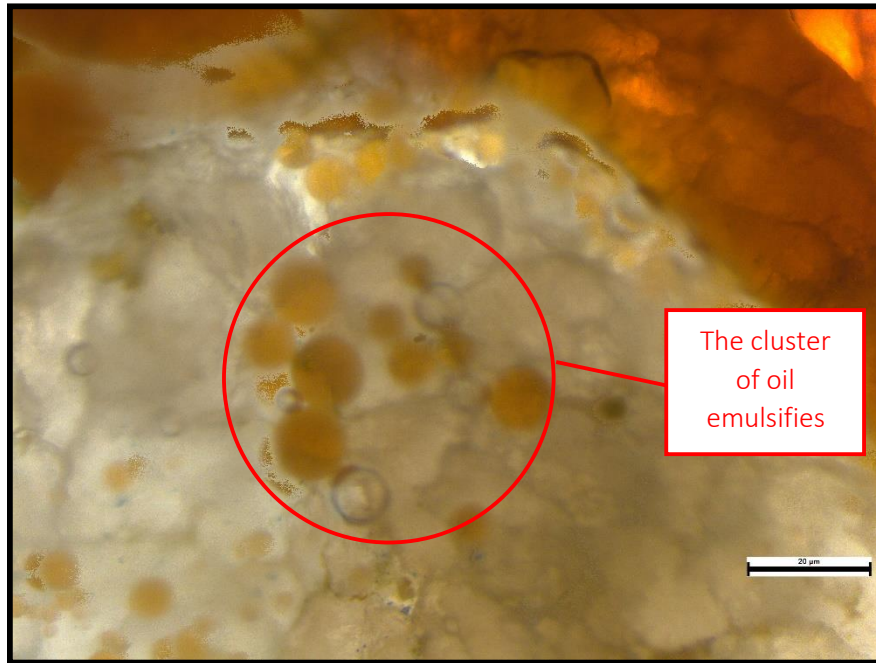


Figure 41 : The crude oil that had been displaced by diluted surfactant in dyed-water solution

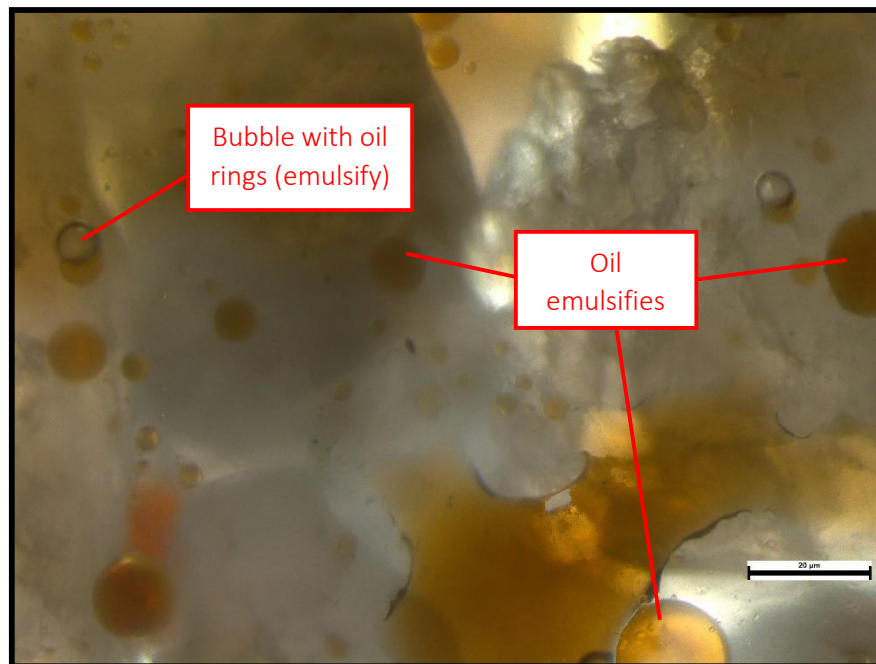


Figure 42 : The crude oil that had been displaced by diluted surfactant in dyed-water solution (other part of micrmodel)



Figure 43: The crude oil that had been displaced by diluted surfactant in dyed-water solution (other part of micromodel)

(d) Displacement of crude oil with 10% of diluted surfactant in dyed-water with nano-particles

Before

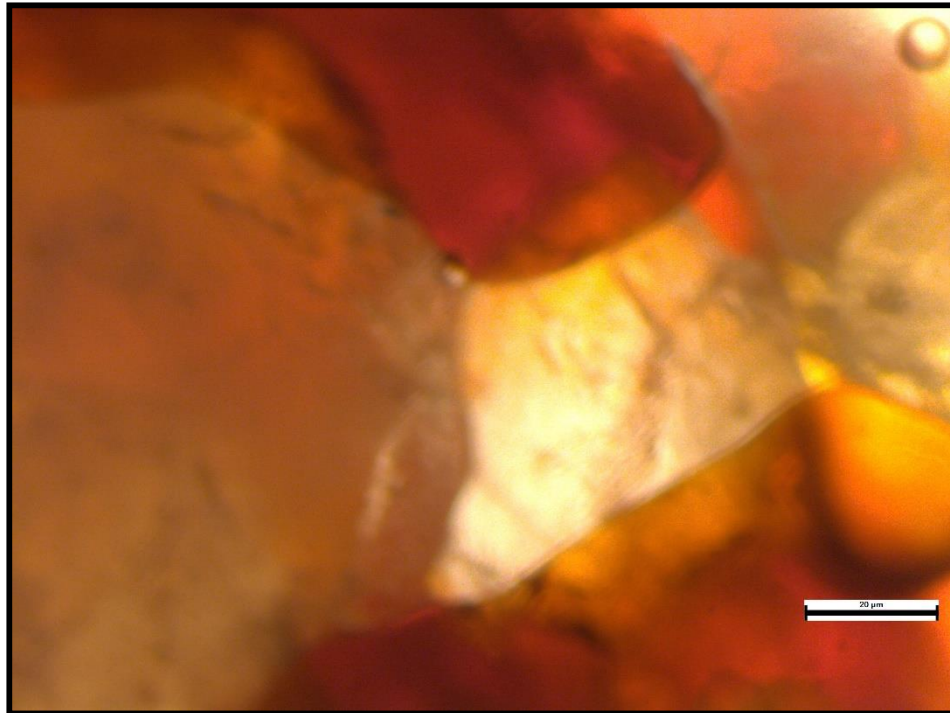


Figure 44 : The crude oil filling the pores of the micromodel

After

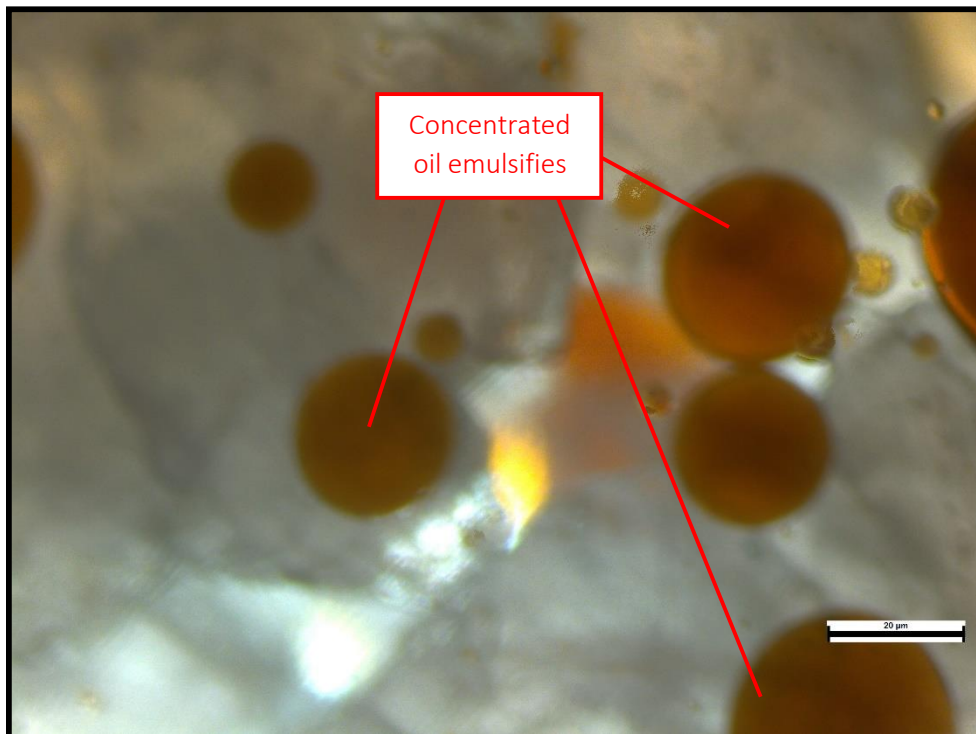


Figure 45 : The crude oil being displaced by diluted surfactant in dyed-water solution (with nano-particles)

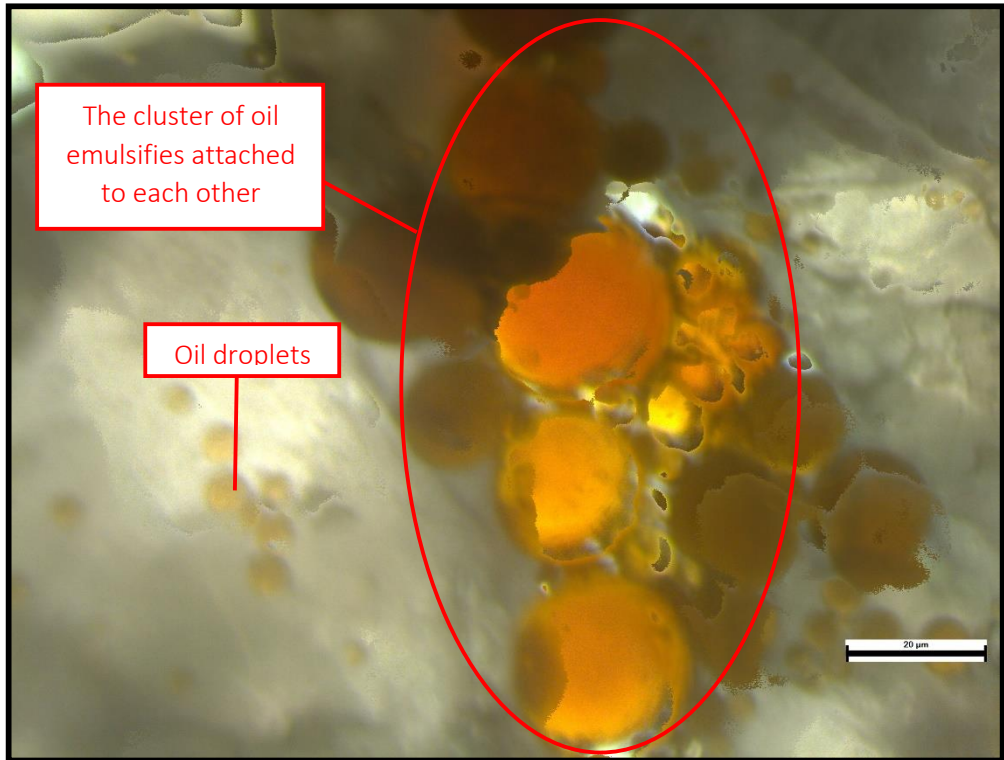


Figure 46 : The crude oil being displaced by diluted surfactant in dyed-water solution (with nano-particles)

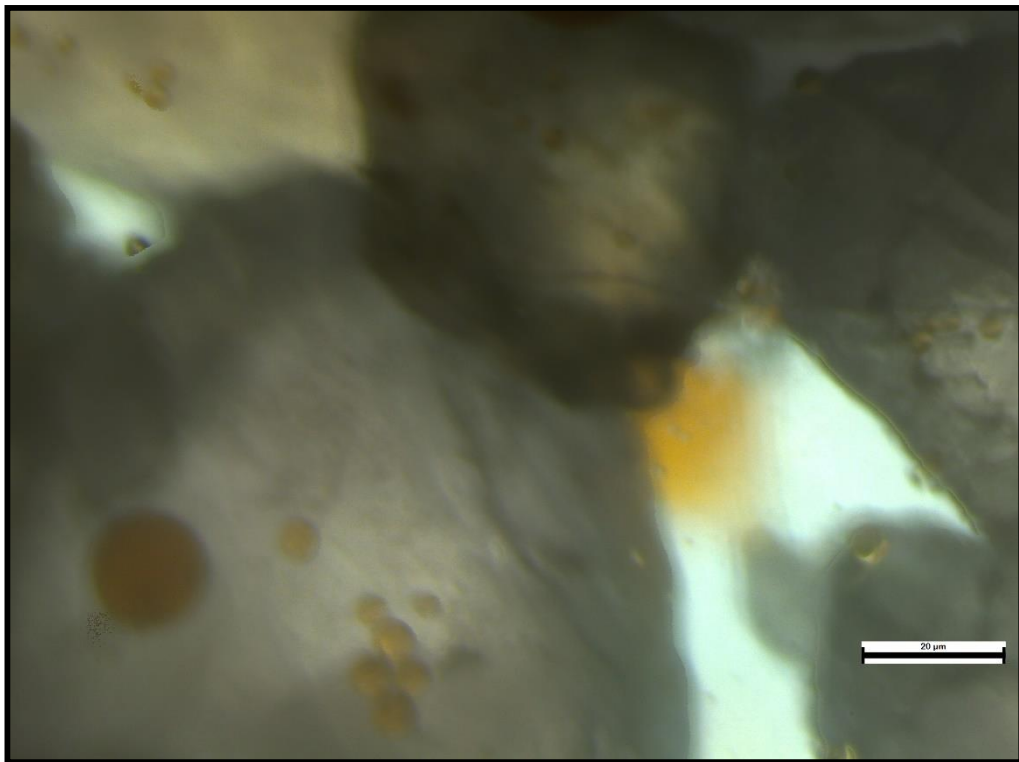


Figure 47 : The crude oil being displaced by diluted surfactant in dyed-water solution (with nano-particles)

4.2 Discussion

The Selection of the Best Porous Media

In this part, the three types of micromodels have been created. The micromodels created are having different characteristic in terms of their structure, type of grain material, and size of grains. The criteria considered in this part are;

3. Porosity
4. Visibility

Comparing between the three types of micromodel,

- a) Sand grains (medium sized) as the inner structure
- b) Sand grains (small sized) as the inner structure
- c) Bead grains as the inner structure

The comparison between the micromodels in the selection is carried out qualitatively. The visual observation is conducted and the results were interpreted and compared. Before going into the selection process, the reason behind the criteria chosen are explain first.

It is important to have minimum porosity value because of the micromodel function to resemble the reservoir rock properties as good as possible. Since the rock layer in the reservoir field will have the low value of porosity and permeability (as the most significant properties), therefore the micromodel should be made in the same way. Due to limitations in this project like cost, equipments and tools availability, and time constraint, the micromodels construction only can be made with larger porosity (not comparable with the reservoir rock porosity) and there will be no control on permeability here. Although the porosity of the micromodel constructed will still be large, the aim of achieving the minimum porosity as much as possible for fluid flow studies is still relevant.

However, due to material limitation where the micromodel used the opaque grains due to its being practical with the cost involved and equipments and tools availability, the next concern should be revolved on the visibility under microscope. The current microscope available for the project is the optical microscope, which will only require simple observation directly under the light beam. Since the light will travel on pore spaces of the micromodel and will not passed the opaque objects like

grains. Therefore, although the micromodel required the very least porosity, the micromodel also required the sufficient size of pores in the grains layer for direct observation under microscope lens. In simpler term, the porosity must not be large for the micromodel and at the same time, the pores must not be too small, so it can be optimally viewed under the microscope.

There are few things need to be highlighted here. In the determination of grain sizes, the grains with smaller diameter than 0.5mm are rejected. This is because of the inner needles size which is 0.5mm. The grains-formed-structure can be disturbed as flow happen since the grains with smaller size will be exited through them (needles). Besides that, the grain shape also played its role here. The grain with irregular (sands) and regular (beads) shapes were carefully choose as they can add the factor into the porosity variable.

With the comparison made in the previous part, the transparent micromodel with smaller grains size was chosen as the micromodel intended for the next stage. It has the smallest porosity and still maintained good visibility under optical microscope.

The Effect of Nano-particles towards Fluids Flow

The first experiment started by flowing dyed-water as fluid into the micromodel. The water was fulfilling the pores as can be seen from **figure 30**. The water cover all part of the pores and therefore, completely achieving the full saturation in this micromodel.

The next experiment was conducted by flowing the crude oil into the micromodel pores which was full with water saturation. The oil displacing the water almost completely, and hence cause the formation of residual water saturation or connate water state. By then, the oil with connate water saturation state was formed in this micromodel.

The aim of this project is study the effects of nano-particles in transparent micromodel where the micromodel will resemble the role of reservoir rock in the oil field. This reservoir rock should be completely understood particularly the place where the oil in the reservoir is residing. By creating the oil with connate water saturation, it

is also resembling the fluids condition of this targeted rock in the petroleum field where the study of this project should revolved; EOR. Therefore, this state of fluids will be the control throughout the fluids displacement and fluids flow study. After the condition of oil with connate water saturation was created, the first variable was tested. This variable will be the displacement of this crude oil by dyed-water with and without the presence of the nano-particle. Any significant effects or behaviors would be observed thoroughly for the interpretation.

Firstly, the dominated crude oil saturation as can be seen in **figure 32** will be displaced by injecting new dyed-water saturation. The condition after the displacement was presented in the few images, subsequently. In **figure 33**, the residual oil bulks was remained in the pores with irregular and sharp edges form. The irregularity of residual oil bulk is still presence after further oil displacement as can be seen in **figure 34**. The small oil droplets were very little numbers and not really concentrated. With the water saturation dominate the pores, not many significant behavior of fluids can be observed.

Next, the dominated crude oil saturation (induced again by the same steps earlier in other micromodel) with water connate was displaced again, this time by using new dyed-water saturation with nano-particles. The resultant effects after displacement was conducted can be seen in **figure 37**. The bubbling effects can be seen in large part of the pores. The bubbles were forming in ranging sizes and dispersed unequally in almost all area of the pores. The formation small oil emulsions also can be seen dispersing in the pores. In **figure 38**, the residual oil bulk were still presence but with the smooth edges or surfaces and much circular-in-shape. The oil rings phenomenon was largely presence in this experiment. It was happening due to process of emulsification. As can be seen from the same pictures, the water bubble with oil layer ring at the outer were vastly formed in large portions of the pores as in **figure 39** and **figure 40**. The emulsify residual oil bulk presented in **figure 39** is a good example of residual oil with smooth edges and circular shape. Comparing the emulsify residual oil bulk shape here with the oil bulk shape in the previous experiment (**figure 33** and **figure 34**), the circularity in shape is much significant in the experiment with nano-particles, assuming the nano-particles effect is taking place here. The beautiful representation of oil droplet circularity can be seen in **figure 40** with good translucency and perfect smooth round edges.

After carried out the control experiment previously by using nano-particles in water displacement, the analysis was further enhance with the new control experiment; by adding surfactant into the displacing water - resembling the chemical flooding in EOR during the oil displacement. The control condition was created again, by using the same principle as earlier which is oil with connate water saturation state.

The experiment analysis began with the displacement of crude oil by injecting diluted surfactant in dyed-water saturation. The crude oil dominate condition as can be seen in **figure 41** was changed completely by the displacement of diluted surfactant in dyed-water saturation which can be observed in **figure 43**. Although there is no presence of nano-particles at this time, the behavior of fluids flow shown here is quite interesting. The large oil droplets were clustering in few portions of the pores and there were some attachment in some of them. As can be seen in this image too, the attachment formed between the oil droplets involving only two or three droplets at the same time. The emulsify oil bulk also formed as can be seen at **figure 44**. The oil bulk is quite concentrated and there are some irregularities in the shape characteristic. Some of the oil emulsifies were circular in shape and some are irregular in shape but with smooth edges. Some are even interconnected in each other. In **figure 45**, the oil bulks are circular in shape, grouped in position and interconnected with few other oil bulks at the oil interface. Few water bubbles can also be seen after the displacement. Although not so much and large, it is still significant in few portions of the pores. The fluids flow behavior as shown here is quite resembling the previous experiment of water with nano-particle displacement but with much lesser concentration.

Next, the experiment is conducted again, this time the displacement with the diluted surfactant in dyed-water with nano-particles. As can be seen in **figure 46**, the pores state was dominated by crude oil. After the displacement, the resultant image as in figure 47 show quite significant effects towards fluids behavior. The residual oil bulk emulsifies were formed with much concentration and regularity in shape. The bulks are circular or round-in-shape and have smooth edges. They were existed in majority portion of the area or pores as in **figure 47** and **figure 48**. Some of them are interconnected with each other at the interface. In figure 48, large number of them were accumulated in the pores and bind by the layer of oil at their outer interfaces. This phenomenon was just appear only on this experiment with surfactant and nano-particles and might indicate certain correlation of nano-particles and certain chemicals

with the reason behind it. The residual oil droplets also can be seen and they were dispersing all around the area. There was certain trend in them this time, majority of the droplets was clustering although randomly disperse. The phenomenon of fluid behavior as observed here is almost the same like what happened in the previous experiment with nano-particles but with more significant and concentrated effects.

The fluids behavior phenomenon in this surfactant experiment also show quite resemble characteristic with the previous experiment of water with nano-particles displacement. It is known commonly that the surfactant tend to have few similar characteristic in its effects towards fluids flow as the nano-particle owns too. This explanation can lead to the reason of why the phenomenon of the experiment with surfactant shown some quite similar behavior with the previous experiment with nano-particles. The effects are enhanced further when both of them are combined together.

The emulsification phenomenon that happening due to nano-particles inducing immiscibility between the two fluids particularly concentrated on residual oil saturation. In scientific terms, oil and water are considered "immiscible," or two liquids that don't like to mix. In chemical aspect, when two molecules do not freely mix they will align themselves in such a fashion to touch as little as possible. This alignment is described as "surface tension." It is this surface tension the oil will constantly battle when attempting to make and maintain an emulsification thus, forming the gobules or colloids like shape.

The phenomenon of bonding between the emulsions also significant especially within the inter-oil-rings connection. This clustering and attachment of emulsions is might be due to hydrophilic characteristic of the oil when being added some impurities to induce it.

In conclusion, the fluids flow effects due to nano-particles do exist. As explained previously, the effects ranging from the displacement effects until the trend of the fluids behaviors. The nano-particles effects are enhanced further with the addition of certain chemicals.

Chapter 5 : Conclusion and Recommendation

5.1 Conclusion

From this study, the nano-particles effects towards fluids flow in porous media can be visualized. Since porous media here should be represented the rock of the reservoir, we can be benefitted with the understanding on how the flow in the reservoir is occurring. In this project, the micromodel has been made better with the use of small sand grains which can produced the good porosity in the layer. The use of large sand grains and beams has been proved not good enough and comparable to produced small porosity value which can help for better illustration or simulation of the fluids flow characteristics in the pores. It will be hard to see and understand macroscopic behavior of the pores level interaction between the fluids and solid matrix if the porosity factor is not influential in the experiment. The visibility also must be good enough for direct observation. This is what make the need of transparent micromodel which mean, transparent to be look upon by any eye contact. All the micromodels created has been made as less porous as possible. Fortunately, the porous section is still not too small for the light beam to pass through until it reach Microsoft lens even with the lowest porosity value model. Therefore, the first objective of developing the good transparent micromodel which can represent a good porous media for this project is achieved. The improvisation on the process of constructing the transparent micromodel might be identified throughout the research and can be carried out to produce better micromodel with the good resemble of porous medium as in reservoir rock. This should be helpful for the future project improvisation with regards of producing the better transparent micromodel.

As the transparent micromodel has been constructed, the fluids flow study with nano-particles would be conducted. As the result shown previously, the nano-particles inclusion in the fluids did affecting the fluids flow behavior. The emergence of emulsions, oil rings at water droplets, clustering behavior of oil emulsions which enhance further by the addition of surfactant and the formation of oil residuals with smooth interfaces or shape indicating the present of nano-particles effects towards fluids flow. Although deeper analysis regarding the real factors of these phenomenon could not be carried out due to the researcher competency level, the objective of identifying the effects of nano-particles has been successfully achieved. Although the

effects of nano-particles towards fluid flows especially with the inclusion of surfactant chemical (as EOR chemical) is presence and known, but whether the activity will lead towards better chemical process in EOR application (in this experiment, regarding the better surfactant application) is still an unknown due to the limited scope of this study. Therefore, the study for future of the nano-particles should include the premise of understanding the effects of nano-particles towards chemical efficiency in EOR.

In conclusion, the fluids flow effects due to nano-particles do exist. As explained previously, the effects ranging from the displacement effects until the trend of the fluids behaviors. The nano-particles effects are enhanced further with the addition of certain chemicals

5.2 Key Milestones

- Producing a good and successful transparent micromodel with the designated procedures.
- Visualization of the fluid flow occur inside the micromodel which is affected by the nano-particles.
- Optional: Identification of flow pattern or behavior of nano-particles fluid flow during the simulation.

5.3 Recommendation

1. The study of the nano-particles flow can be carried out in pilot scale at suitable reservoir condition by utilizing the current latest technologies available for better fluids flow study program.
2. The micromodel used for the experiment can be made better with the help of on-site rock or cores which still possessed the initial condition of grain and fluid properties such as swelling, fluid volumetric and others desired properties which should enhanced better result of almost-true fluids flow in certain reservoir conditions.

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Appendices

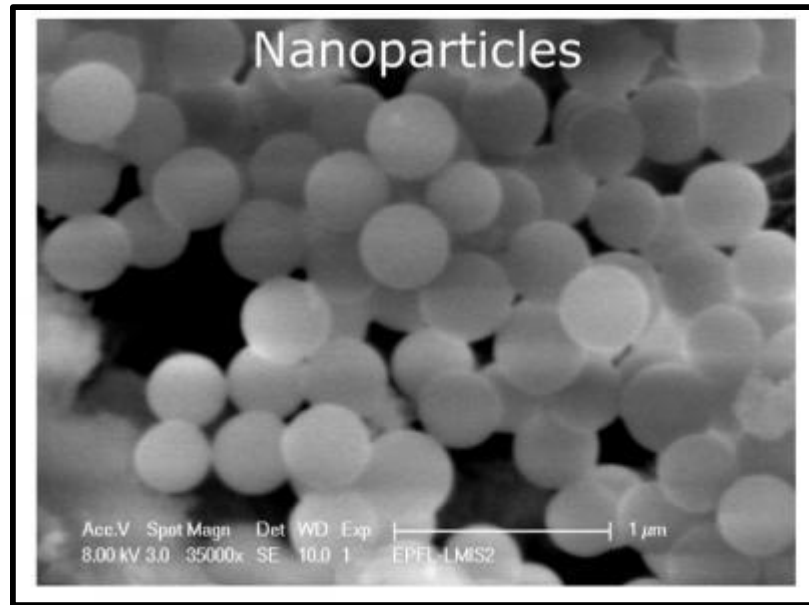


Figure 48 : Nano particles view from SEM Microscope in the scale of 1 μ m

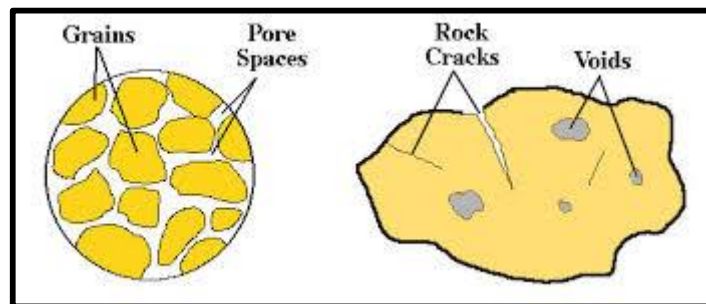


Figure 49 : Rock grain and its pores

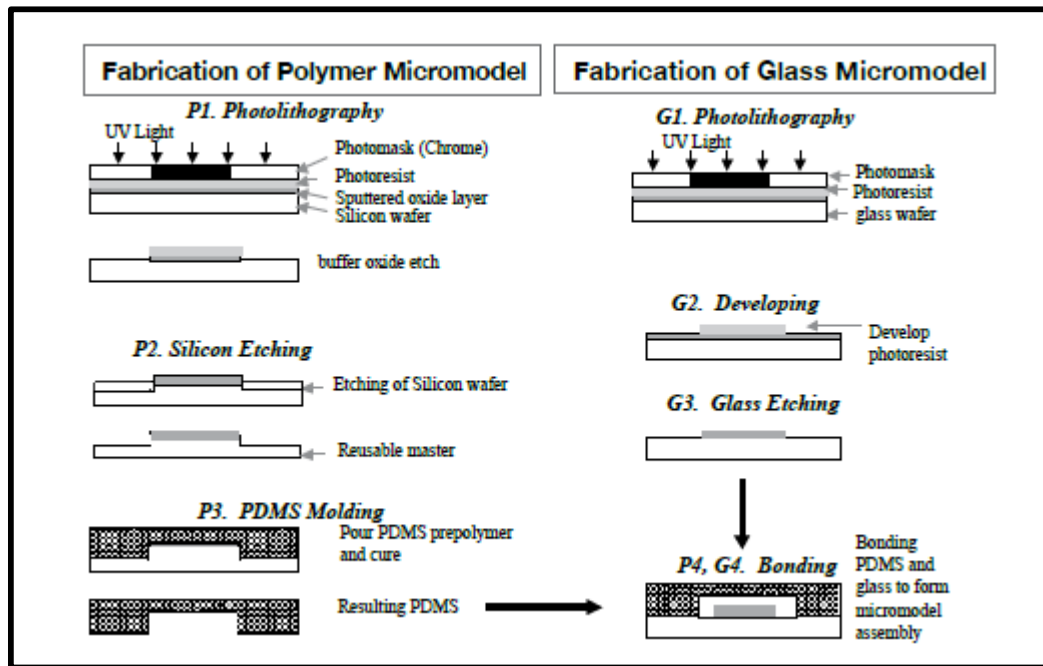


Figure 50 : Fabrication steps of polymer (left) and glass (right) using conventional method with Javadpour method



Figure 51 : Fracture network and matrix micromodel with ceramic boundaries sample constructed in Zhang research

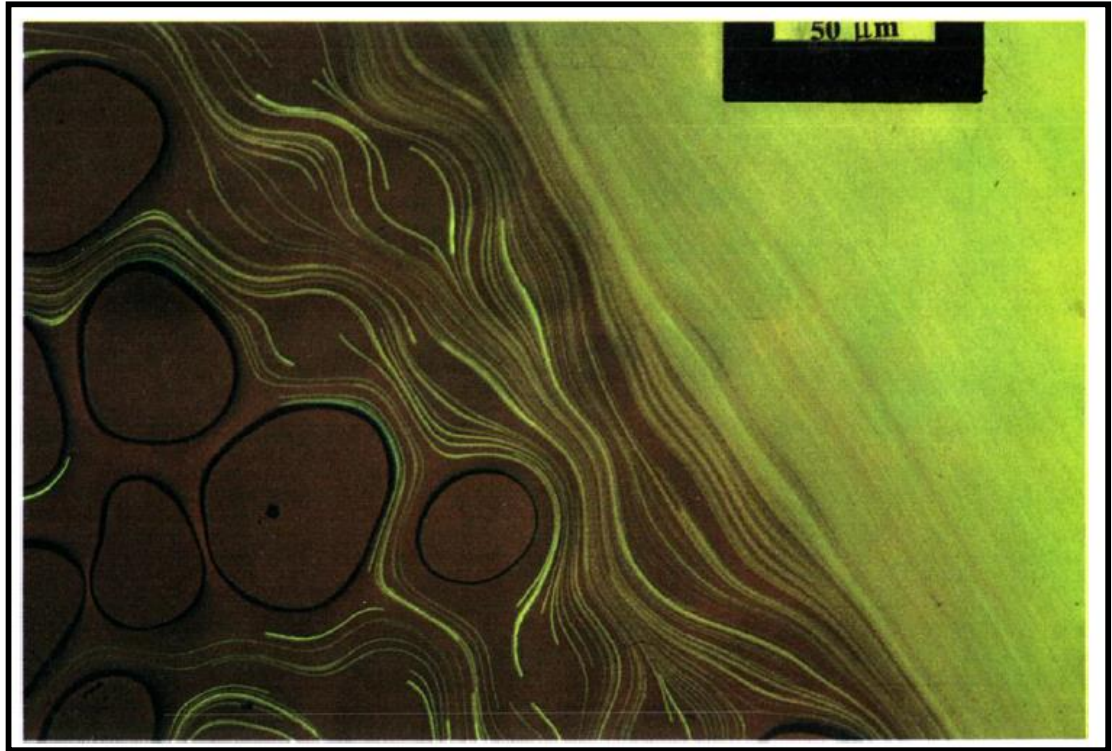


Figure 52 : Example of microphotograph study sample of steady state fluorescent particle tracks in a saturated fracture (upper right) and adjacent matrix (lower left) in micro scale

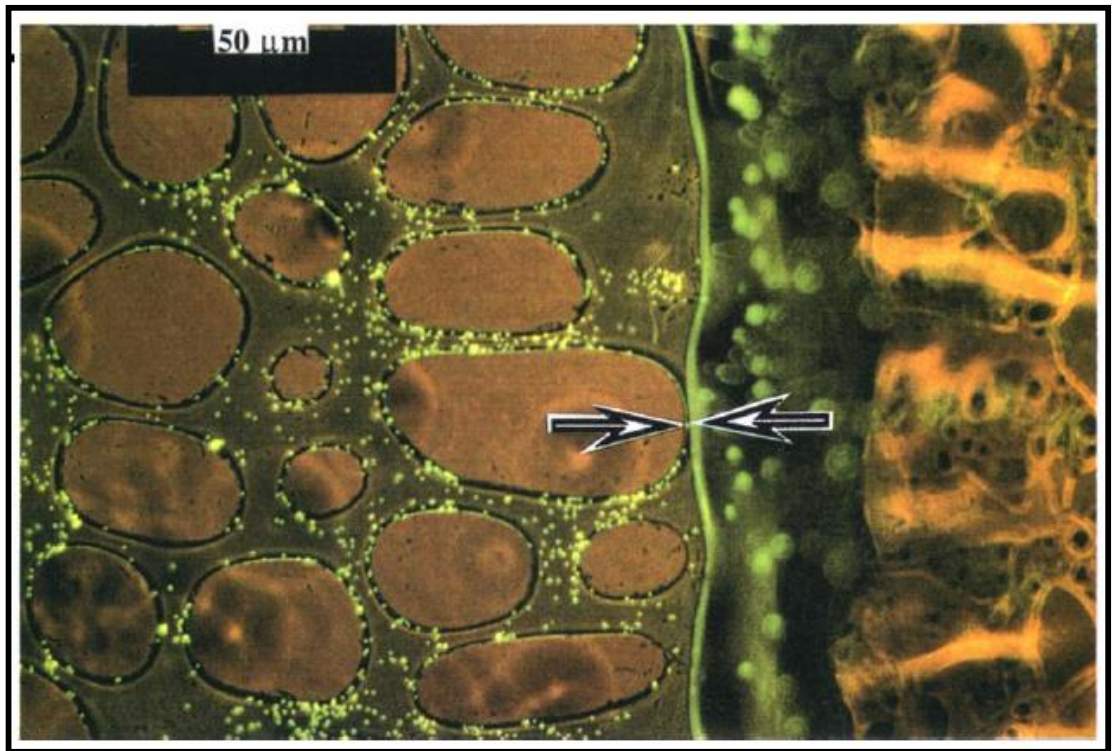


Figure 53 : Sample of microphotographs of fracture-matrix interface taken during transmissivities experiment.

