

CHARACTERIZATION OF ZINC OXIDE AS DIELECTRIC  
CERAMICS FOR REDUCTION OF INTERFACIAL TENSION  
BETWEEN OIL AND WATER

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Tension Between Oil and Water**

by

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Dissertation submitted in partial fulfilment of  
the requirement for the  
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**(Mechanical)**

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CERTIFICATION OF APPROVAL

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January 2017

## 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.

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AHMAD WAFIY ADLI BIN YAHAYA

## ABSTRACT

Generally, the application of nanotechnology is widely used in daily life. Nanotechnology helps to improve the characteristic and properties of the material. Most of the application of the nanoparticles is to enhance the ability of the material which improve their performance during its operation. Nanoparticles also would help to increase the wettability of the particles. The increasing wettability of the material or substances will lead to the decreasing of the interfacial tension between the surfaces. In the oil and gas industry, oil that is extracted is not optimum due to high interfacial tension between oil and water. Thus, this research was conducted to study the effect of different concentration of zinc oxide nanoparticles to the reduction of interfacial tension between oil and water. This project is mainly to investigate the changes on the physical and molecular level properties of the oil and water after the zinc oxide nanoparticle is used as additional component in the brine solution to increase mobility of the oil. The characterization of zinc oxide is done by using XRD and FESEM. For this experiment, the IFT is not directly measured by using IFT machine instead, the IFT is analyzed through the contact angle testing and pendant drop testing. The contact angle and pendant drop test were conducted by using the goniometer machine, to justify the changes of interfacial tension between oil and water. Increasing the concentration of the nanofluid, the contact angle and surface tension are decreasing. It is confirmed that the interfacial tension between oil and water is decreasing along with the increasing concentration of zinc oxide nanofluid. The graph of concentration of nanofluid against the contact angle and surface tension will be plotted by the data obtained from the contact angle testing and pendant drop testing which to analyze the interfacial tension of the water to the zinc oxide. In the nutshell, this study can prove that zinc oxide nanoparticles have the wettability properties that possess a high potential to replace the current method of reducing the interfacial tension between oil and water and used in the oil and gas industry as one of the Enhanced Oil Recovery (EOR) alternatives.

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*In the Name of Allah the Most Gracious, the Most Merciful*

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# **CHAPTER 1**

## **INTRODUCTION**

### 1.1 Project Background

Generally, the application of nanotechnology and biomaterial to enhance the oil recovery are started to catch the researchers' attention. As the characteristic of the oil which is more viscous than a water, it makes the interfacial tension between them is high. The other properties of the oil and water that affect the interfacial tension are the PH value of the substances, temperature, pressure and the composition of each phase. The interfacial tension is the force that holds between the two surfaces of a particular phase together. The force that applied between the surfaces are different to one and another.

As of this moment, nanoparticles are widely used in industry. The present of nanoparticle would help to change the properties of the material such as Optical (Give color/Transparent), Chemical (Catalyst), Electronic/Electrical (Conductivity) and Thermal (Heat Transfer/Insulation).

Oil and water are stick together due to the strong interfacial tension between these two mediums which strict the mobility of the oil. From previous research studies, there are many approaches been made to reduce interfacial tension between the oil and water. One of them is injecting the biomaterial and nanoparticles in the oil and water mixtures. Nanoparticles works as a bridge in between bulk material and atomic or molecules. As the size of the nanoparticles are usually in order of below 100nm, they can easily flow into the mixture structure of oil and water. So, this project is mainly to investigate the changes on the physical and molecular level properties of the oil and water after the zinc oxide nanoparticle is used as additional component in the brine solution to increase mobility of the oil. In addition to characterize zinc oxide in the preparation as nano-dielectric materials.

At the end of the experiment, the interfacial tension and the mobility of the oil can be calculated by measuring the contact angle to obtain the wettability of the oil drop in the nanofluid and pendant drop test to have the surface tension of the nanofluid. In a nutshell, this study can prove that the nanoparticle of zinc oxide can help to reduce the interfacial tension between oil and water which lead to the increasing of oil mobility.

## 1.2 Problem Statement

The interfacial tension between the oil and water is caused by the composition of the oil that is more viscous than the water. This property leads the oil to stick to the water surfaces. The problem is likely to be seen at the reservoir where the operation of extracting mineral sources from the well. As the mineral sources are sucked up to the upper ground, the volume of the oil that are managed to be extracted is not in optimum capacity. Thus, it is a waste of a time and the operation itself as every operation and time spent at the reservoir will be included in the operation cost.

To fix the problem, the current technology introduces some methods to enhance the oil production. There are chemical method, physical method, biological method and technical method.

These methods are proven to increase the mobilization of the oil, but there are few challenges coming from them. Most of them are expensive, give a low sweep efficiency and has a possibility to damage the formation inside the well.

Thus, it is suggested to use the nanotechnology especially zinc oxide nanoparticle to replace the existing method and technology to improve the mobility of the oil and at the same time, could reduce the cost of the operation without damaging the formation.

### 1.3 Objectives

The objectives of this research study as below:

- i. To characterize zinc oxide in the preparation as nano-dielectric materials.
- ii. To apply contact angle and pendant drop test to calculate the interfacial tension between oil and water.
- iii. To evaluate the effect of the zinc oxide nanoparticle concentration on the interfacial tension between water and oil.

### 1.4 Scope of Study

For this project, the scope is to emphasize on the preparation of the zinc oxide as a nano-dielectric material and to reduce the interfacial tension between the oil and water. The outcome will be compared to the current technology and method used in the industry. Thus, this project is mainly to investigate the changes on the physical and molecular level properties of the oil and water. The focus is highlighted on the characterization of the zinc oxide nanoparticle, how the zinc oxide nanoparticle help to reduce the interfacial tension, and what concentration of the zinc oxide nanoparticle is needed. We will monitor the how the concentration of the nanoparticle in the brine solution would affect the interfacial tension. Wettability and interfacial tension between two (2) surfaces will be determined by using contact angle measurement and pendant test.



## **CHAPTER 2**

### **LITERATURE REVIEW**

#### 2.1 Overview of nanoparticle

At present, the nanoparticle research has gained momentum in order to improve the current technology to a whole new level. Nanoparticles are part of nanomaterials that are defined as single particles 1-100 nm in diameter, (Negin, Ali et al. 2016). For the past few years, nanoparticles have been one of the common materials used for development purposes in many areas including communication, energy and data storage, environment protection, medicine as well as engineering. To be specific, the uniqueness of nanoparticles is due to their attributes and properties depending on the metal oxide nanoparticles used. Due to the size of the nanoparticles that are small, it allows the particles to dissolve and change the host material at the molecular level which then will enhance its capability to the maximum level.

There are many types of nanoparticles which are metal nanoparticles, metal oxide nanoparticles and polymer nanoparticles. Among these, metal oxide nanoparticles stand out as one of the most flexible materials due to diverse properties and functionalities.

## 2.2 Composition of zinc oxide nanoparticle

The zinc oxide (ZnO) has a broad and versatile range of application in many field such as engineering, medical and cosmetic. Zinc is a block D, period 4 element while oxygen is a block P, period 2 element. As for ZnO nanoparticles, it is available in two forms which are powder and dispersion.

“The crystalline ZnO has a wurtzite (B4) crystal structure at ambient condition. The ZnO wurtzite structure has a hexagonal unit cell with two lattice parameters, a and c, and belongs to the space group of  $C^4_{6v}$  or  $P6_3mc$ .” (Vaseem, Umar et al. 2010)

“Figure 1 clearly shows that the structure is composed of two interpenetrating hexagonal closed packed (hcp) sublattices, in which each consist of one type of atom (Zn or O) displaced with respect to each other along the threefold c-axis. It can be simply explained schematically as a number of alternating planes stacked layer-by-layer along the c-axis direction and composed of tetrahedrally coordinated  $Zn^{2+}$  and  $O^{2-}$ .” (Vaseem, Umar et al. 2010)

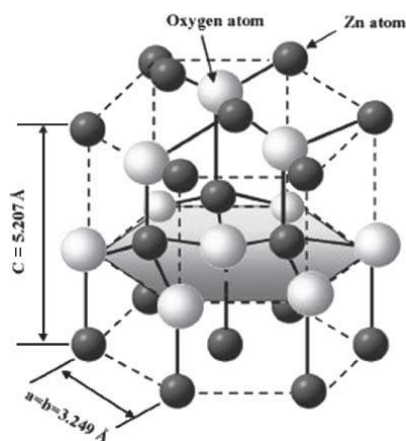


Figure 2.1 : The hexagonal wurtzite model of ZnO.  
(Vaseem, Umar et al. 2010)

## 2.3 Characterization of zinc oxide

Characterization is a fundamental process in the field of material science which measure and identify the material's structure and properties.

### 2.3.1 Characterization by using X-Ray Diffraction (XRD)

For this stage, X-Ray Diffraction (XRD) pattern will be used to evaluate and justify the type of the particles. XRD is a non-destructive analytical technique for identification and quantitative determination of the various crystalline forms. It is important to identify the composition of the material before proceeding with further investigation as the composition of the material would affect the outcome of the experiment. Typically, the process is performed by using X-Ray diffractometers which is consist of X-Ray tube, a sample holder and an X-Ray detector. The procedure of identifying the component is basically the X-Ray are collimated and directed onto the sample. As the sample and/or detector are rotated, the intensity of the reflected X-Rays is recorded.

As shown in the figure 2.2 (a), (b) and (c) the XRD pattern shows the different data obtained from the XRD characterization due to different composition mixed in the material.

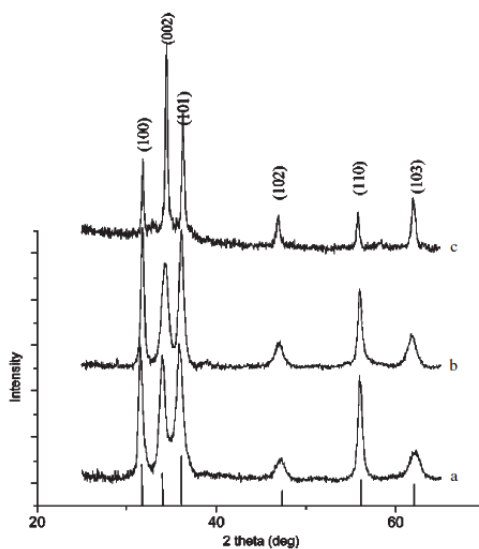


Figure 2.2 : Example of XRD spectra of zinc oxide with different particle formation (a) nanotriangles, (b) spherical nanoparticles, and (c) nanorods. (Andelman, Gong et al. 2005)

### 2.3.2 Characterization by using Field Emission Scanning Electron Micrographs (FESEM)

The Field Emission Scanning Electron Micrographs (FESEM) also is used to differentiate between two crystalline structures. It is imaged via electrons from upper most layer of the material which yields a topographical-type image. Generally, FESEM is operating by focusing the electron beam (2-10keV) scans on the surface. There are several types of signals are produced and detected as a function of position on the surface. FESEM imaging is typically obtained by using secondary (SE) or backscattered (BSE) electrons.

For zinc oxide, the formation of the particles is depending on the type of the synthesis. It is varying from one to another. Based on (Andelman, Gong et al. 2005) further explained regarding the different effect by using the different solvent. For example, as shown in figure 2.3 and figure 2.4, when synthesizing by using trioctylamine (TOA) solvent yield nanorods. Then, if use 1-hexadecanol (HD), solvent yields nanotriangles, and 1-octadecene (OD) solvent yields spherical nanoparticles.

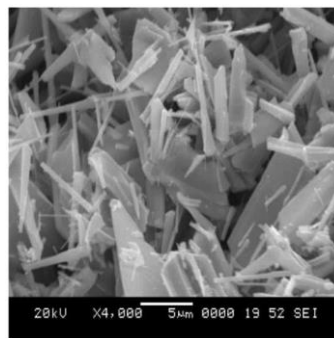


Figure 2.3 : Example of FESEM micrograph of ZnO particles at 4K magnification shows cylindrical shape particle. (Sonage and Mohanan 2014)

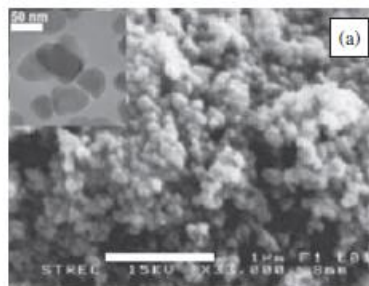


Figure 2.4 : Example of FESEM micrograph of ZnO particles at 33K magnification shows spherical-shaped particles. (Vaseem, Umar et al. 2010)

## 2.4 Surface and Interfacial Tension

Surface and Interfacial tension force (IFT) is a property of the interface which occur between two surfaces. When both phases are liquid, then term that is used is interfacial tension meanwhile if the phases are different from one to another, the surface tension term is used. Interfacial tension is the tendency of a liquid to expose a minimum free surface when it is in contact with an immiscible fluid, and interfacial tension acts perpendicular to the interface, (Birkeland 2013).

The force of IFT is different from one surface to another due to several factors which are the viscosity of the fluid, the hydrophilic and hydrophobic of the fluid, and wettability of the fluid to the surfaces. There are few methods and applications can be done to calculate can determine the IFT of the material. There are by calculating the contact angle of the fluid, pendant drop test, spinning drop and capillary rise, (Yuan and Lee 2013).

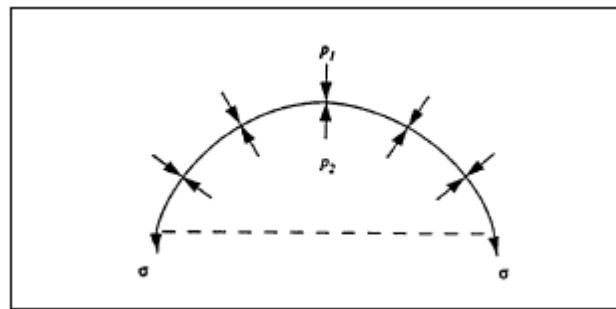


Figure 2.5 : The IFT acts perpendicular to the interface between the two immiscible fluids. (Birkeland 2013)

### 2.4.1 Contact Angle

Contact angle can be defined as the angle formed by the intersection of the liquid-solid interface and liquid-vapor interface, (Yuan and Lee 2013). Specifically, contact angle obtained indicates the condition of the wet-surface as shown in figure 2.7. If the wetting conditions are neither strongly oil-wet nor strongly water-wet, the balance of forces in the system will result in a contact angle,  $\theta$ , (Enge 2014). As shown in the figure 2.6, on the most left shows the oil drop produce the contact angle approximately zero degree, indicating water wet surface. The center oil drop shows intermediate-wet surface due to the force balance between the interfacial tension term.

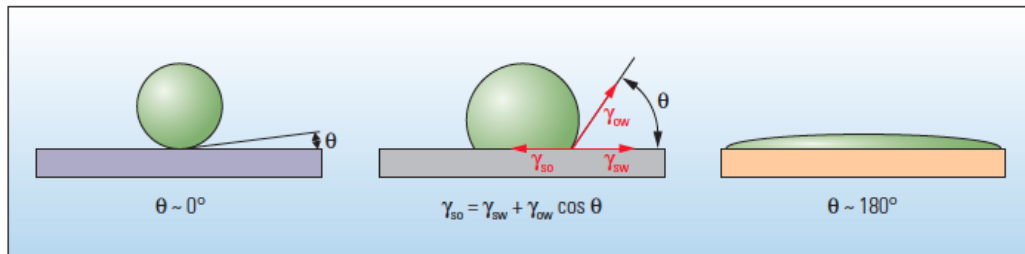


Figure 2.6 : Contact angle between two surfaces. (Abdallah 2007)

Besides, according to the Young's equation (Equation 2.1), there is a relationship between the contact angle,  $\theta$ , the surface tension of the liquid,  $\sigma_{ow}$ , the interfacial tension between liquid,  $\sigma_{ws}$ , and solid and the surface free energy of the solid.

$$\sigma_{os} = \sigma_{ow} \cdot \cos \theta + \sigma_{ws} \quad (2.1)$$

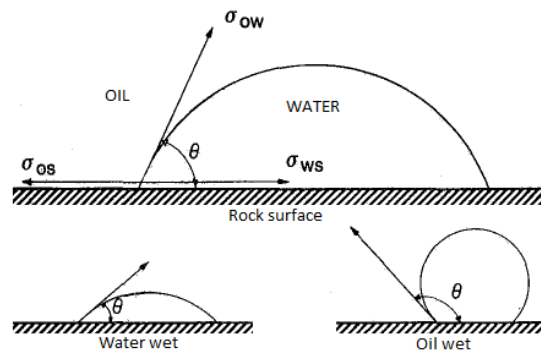


Figure 2.7 : Illustration of interfacial tension for a two-phase system with water and oil on a rock surface. (Birkeland 2013)

### 2.4.2 Pendant drop test

Pendant drop test is one of the experimental techniques to measure the interfacial tension between two surfaces, (Arashiro and Demarquette 1999). The test has an ability to determine the interfacial tension from the shape of pendant liquid drop deform by gravity, (Berry, Neeson et al. 2015).

A pendant drop method involves the determination of the profile of a drop of one liquid suspended in another liquid at mechanical equilibrium, (Arashiro and Demarquette 1999). A pendant drop at equilibrium obeys the Young–Laplace equation (Equation 2.2), which relates the Laplace pressure across an interface with the curvature of the interface and the interfacial tension,  $\gamma$ .

The setup for the experiment is shown in figure 2.8 (a) and the example of the pendant drop shape shown in figure 2.8 (b).

$$\Delta P = \gamma \left( \frac{1}{R_1} + \frac{1}{R_2} \right) \quad (2.2)$$

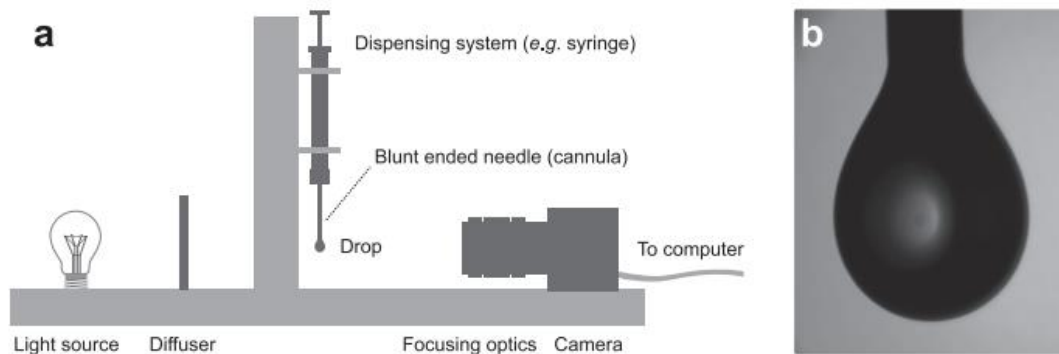


Figure 2.8 : a) Basic experimental setup for pendant drop testing. b) An example of drop image. (Berry, Neeson et al. 2015)

## CHAPTER 3

### RESEARCH METHODOLOGY

3.1 Research Flow Diagram This is the work flow of the process starting from the beginning of the research to the execution in the lab.

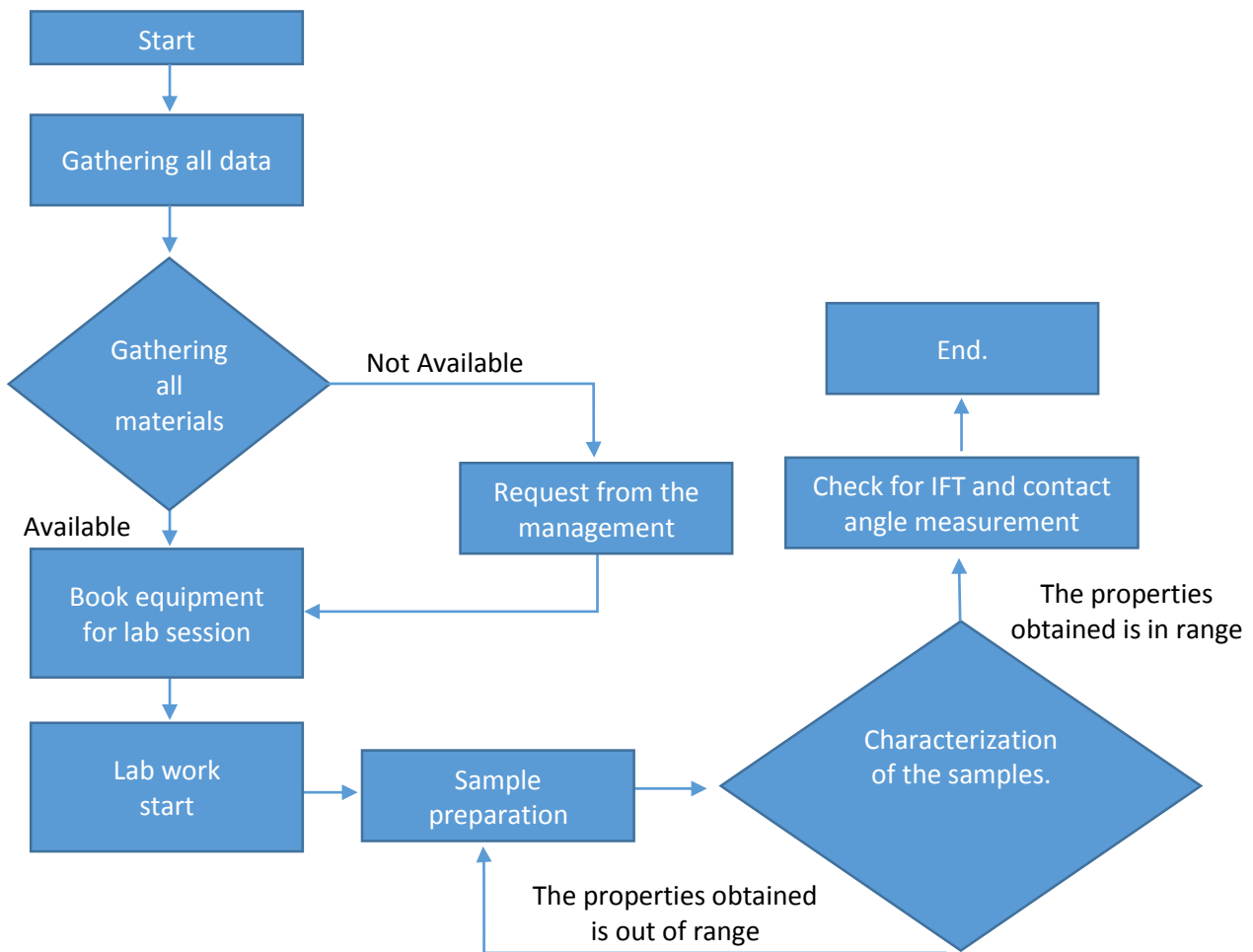


Figure 3.1 : Block diagram of the process flow of the research.



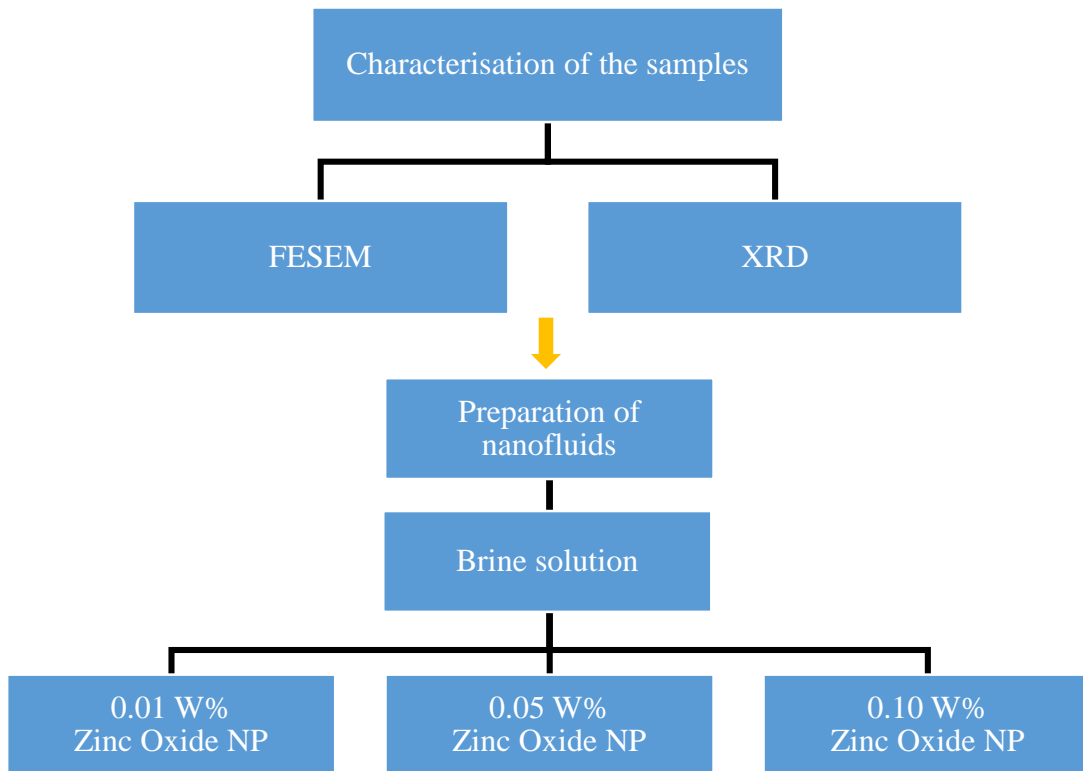


Figure 3.2 : Preparation of nanofluid block diagram.

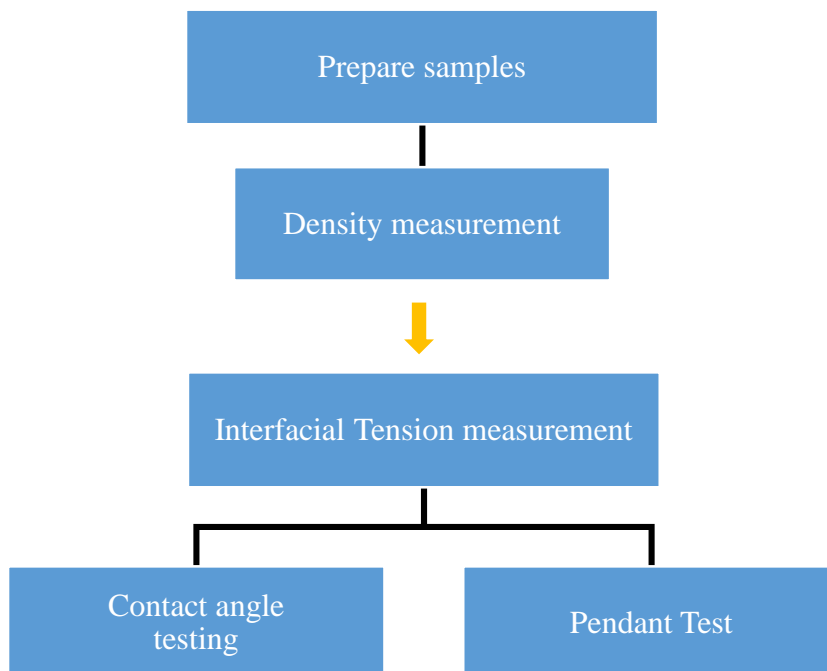


Figure 3.3 : Interfacial tension testing block diagram.

## 3.2 Sample characterization.

### 3.2.1 X-Ray Diffraction (XRD)

XRD machine that used in this experiment is manufactured by Bruker, model D2 Phaser as shown in the appendix section (Appendix A). The procedure of XRD characterization as following :-

- i. The sample used was zinc oxide NP powder.
- ii. The sample was placed on the sample holder as shown in appendix section
- iii. The step size was set to  $20^\circ$  to  $90^\circ$
- iv. The data obtained was compared with the data taken from the library of the Highscore software.

### 3.2.2 Field Emission Scanning Electron Microscopy (FESEM)

For this project, FESEM model SUPRA 55VP that manufactured by ZEISS as shown in appendix section (Appendix A) is used. FESEM characterization required the sample to be in the dry condition. The procedure of FESEM characterization as following :-

- i. The samples were prepared as in table 3.1.
- ii. Drops of the sample solutions on the aluminum stud.
- iii. The sample was magnified to 200,000 times.
- iv. The size of the particles was measured from the images obtained from the characterization process by using the ImageJ software.

### 3.3 Preparation of nanofluid

The nanofluid preparation is started by preparing the brine solution before the weighted zinc oxide nanoparticles mix with the brine solution to form the nanofluid.

#### 3.3.1 Brine solution preparation

Brine solution is used in this experiment act as a carrier of the nanoparticle which is to replace the seawater that exist in the real reservoir. In average the salinity of the seawater is 35000 ppm.

The procedure of the preparation of the brine solution as follow: -

- i. Sodium Chloride (NaCl) was weighted 35g using the weight scale.
- ii. 1L of distilled water was poured in a beaker.
- iii. The weighted NaCl was mixed in the distilled water.
- iv. The mixture was mixed and stirred for 10 minutes by using stirring device at room temperature.

#### 3.3.2 Zinc oxide nanofluid

Three samples of nanofluid were prepared with different weight percent as depicted in the table 1 below.

Table 3.1 : Nanofluids preparation

No.	Sample Name	Brine solution (mL)	Zinc Oxide NP (g)
1.	0.01 wt% Zinc Oxide NP	300	0.03
2.	0.05 wt% Zinc Oxide NP	300	0.15
3.	0.10 wt% Zinc Oxide NP	300	0.30

All the nanofluid were prepared by the following procedure.

- i. The weight of the zinc oxide was measured by using the weight scale.
- ii. The zinc oxide NP was added in the brine solution.
- iii. The mixture was mix and stirred for 10 minutes by using stirring device at room temperature.
- iv. The ZnO nanoparticle were characterized by FESEM and XRD.

### 3.4 Interfacial tension testing

#### 3.4.1 Contact angle testing

The three samples were prepared for contact angle testing using the Goniometer.

- i. The density of the nanofluid were measured.
- ii. Setup the Goniometer machine. The temperature set for the beaker and syringe was 40°C.
- iii. The plate of the goniometer was immersed in the nanofluid.
- iv. The oil was drop at the bottom side of the plate surface by using syringe.
- v. The image of the drop was taken.
- vi. Measure the angle of the drop by using the built-in goniometer software.
- vii. Repeat the experiment by using the different concentration of nanofluid.

The reference for the setup of the experiment taken from Birkeland (2013).

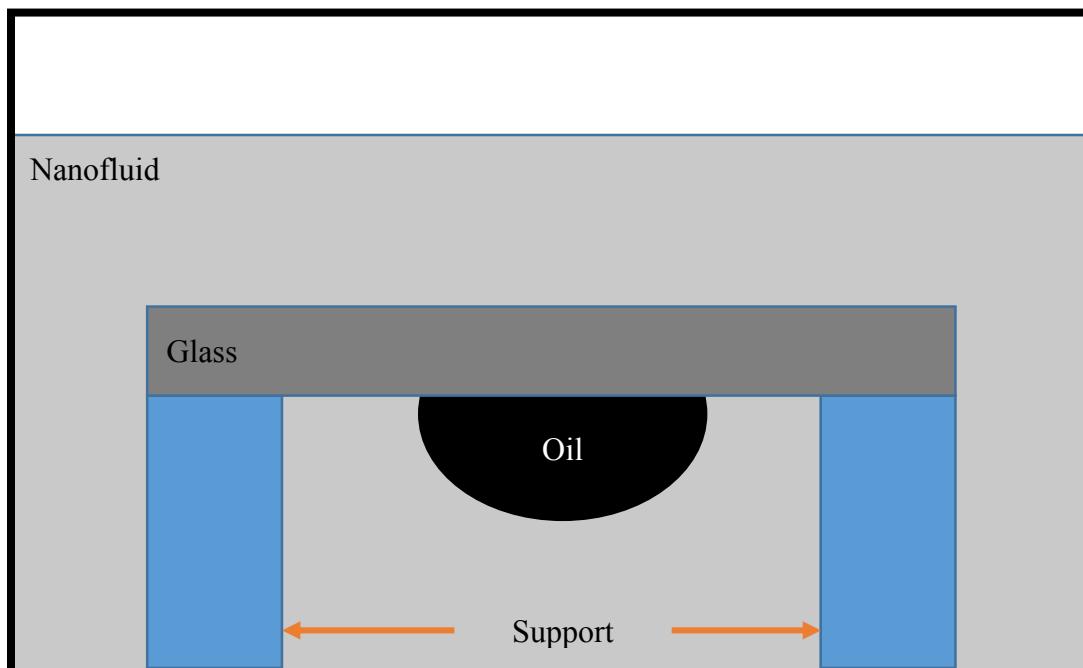


Figure 3.4 : Illustration of the setup for the contact angle test.

### 3.4.2 Pendant test

The three samples were prepared for pendant test using the Goniometer.

- i. Setup the Goniometer machine.
- ii. Prepare the sample. (For angsi oil sample, it is heated at 40°C).
- iii. The sample was then suck in the syringe.
- iv. Slowly inject the sample.
- v. The images of the samples at the tip of the syringe were captured and analyzed by using the software.
- vi. Repeat the experiment with the different concentration of the nanofluid.

Table 3.2 shows the Gantt chart of the project.

Table 3.2 : Gantt Chart

No	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of project topic														
2	Preliminary research work														
4	Submission of Extended Proposal					X									
5	Proposal defense							X							
6	Project work - More literature review - Purchase any material involved - Lab booking														
8	Submission of Interim Report (Draft)													X	
9	Submission of Interim Report														X
<b>Semester break (3 Jan 2017 – 15 Jan 2017)</b>															
1	Continue research work - Collect the material - Nanoparticles preparation														
2	Characterization of the nanoparticles - Analyze data from characterization														
3	Progress Report submission							X							
4	Interfacial tension testing - Contact angle - Pendant test														
5	Pre-SEDEX										X				
6	Documentation of project - Final report submission - Soft-bound dissertation submission - Technical paper submission														
9	Viva													X	
10	Hard-bound dissertation submission														X

### Key Milestone

1. Submission of Progress Report: 17<sup>th</sup> March 2017
2. Pre-SEDEX: 21<sup>st</sup> March 2017
3. Submission of Dissertation Report (Softbound) & Technical paper: 14<sup>th</sup> April 2017
4. Viva: 21<sup>st</sup> April 2017
5. Submission of Dissertation Report (Hardbound): 28<sup>th</sup> April 2017

## CHAPTER 4

### RESULTS AND DISCUSSIONS

#### 4.1 Density measurement of zinc oxide NP

The density of different concentration of the ZnO NP is measured and the data obtained is shown in the table 4.1 and illustrated in a graph shown in figure 4.1.

Table 4.1 : Density of zinc oxide nanoparticles

No.	Concentration of ZnO NP (wt%)	Density (kg/m <sup>3</sup> )
1.	0.01	1.01816
2.	0.05	1.01856
3.	0.10	1.01853

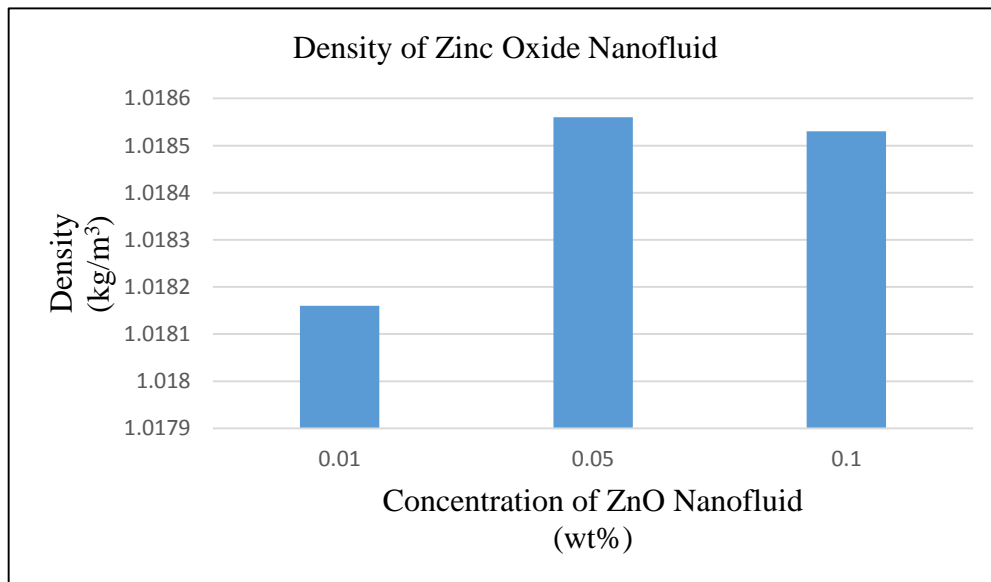


Figure 4.1 : The graph of density of zinc oxide nanofluid.

Based on the data obtained, the density of the ZnO nanofluid is increasing as the concentration of nanofluid increase. The density for the ZnO is decreasing when the concentration is 0.10wt% compared to 0.05wt% is believe due to the solution is already saturated.

## 4.2 Characterization of zinc oxide

The characterization of the zinc oxide was done as stated in section 3.4.

### 4.2.1 Characterization by using XRD

The data obtained is shown in the figure below.

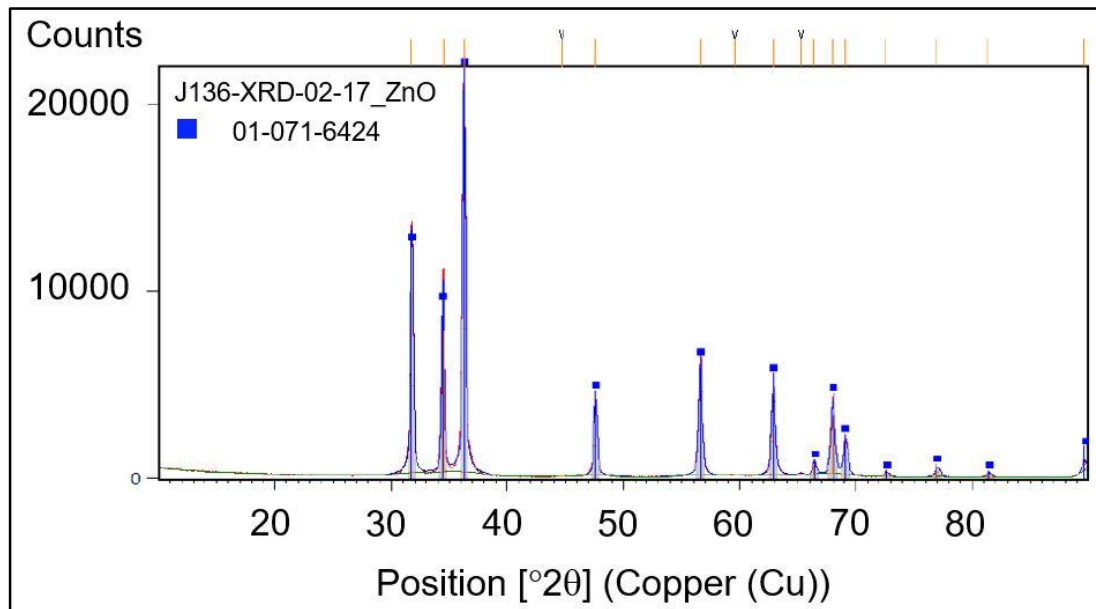


Figure 4.2 : XRD characterization of zinc oxide

The data obtained shown in figure 4.2 shows that the peak is in between 30° to 80°. As shown in the figure 4.3, the peak of the sample which is in orange color is matched with the selected component using the library.

The XRD pattern is matching with International Center of Diffraction Data (ICDD) database file number 01-071-6424 which confirm that the powder is zinc oxide. The pattern obtained is also matched with the result obtained from Sonage and Mohanan (2014).



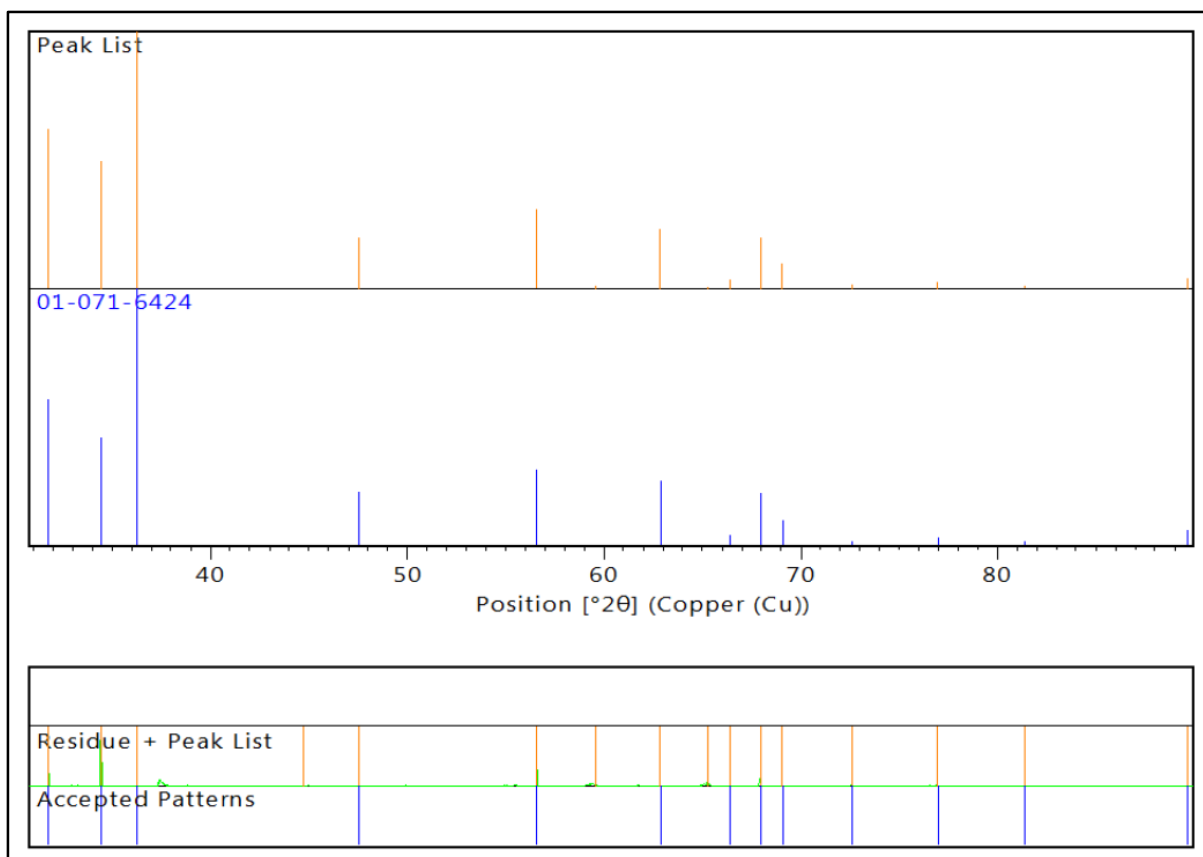


Figure 4.3 : The sample peak (orange line) is compared with selected sample in database (blue color).

#### 4.2.2 Characterization by using FESEM

The samples were magnified to 200,000 magnification. The FESEM images obtained is shown below.

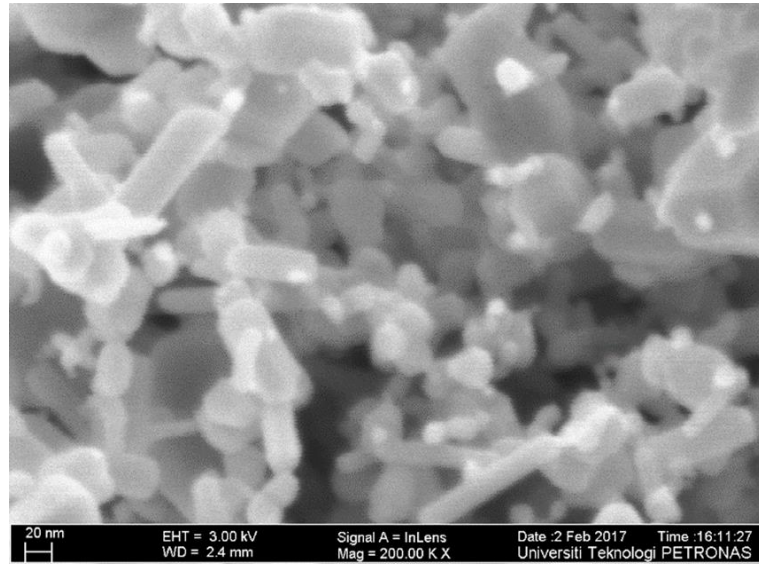


Figure 4.4 : FESEM micrograph of ZnO NP at 200,000 magnification.

From the image shown in figure 4.4, the zinc oxide nanoparticles are in cylindrical shape. The shape of the ZnO nanoparticles obtained is similar to the result obtained by kumar (2013) The size of the particles are not uniform but they are below than 100nm. To justify the size of the particles, ImageJ software is used.

#### 4.1.2.1 FESEM data evaluation by using ImageJ software

The FESEM image is evaluated by using ImageJ software to identify the diameter and length of the zinc oxide nanoparticles.

Table 4.2 shows the diameter of the cross section of zinc oxide NP with the average of 24.31nm. Meanwhile, in table 4.3 shows the length of the cylindrical shape of the zinc oxide NP. The average length of the zinc oxide NP is 58.21nm

Table 4.2 : The diameter of the cross section of the zinc oxide NP.

No of reading	Area (nm <sup>2</sup> )	Mean (nm)	StdDev (nm)	Min	Max	Perim. (nm)	Angle	Diameter (nm)
1	15.87	197.42	6.90	175.00	210.37	25.63	-58.57	25.63
2	15.87	196.59	8.41	175.00	208.98	25.53	-38.23	25.53
3	14.40	231.00	14.38	174.00	251.00	23.09	-90.00	23.09
4	14.40	187.49	11.81	148.00	203.68	23.14	-29.93	23.14
5	14.77	230.16	14.07	183.00	250.18	23.89	-82.69	23.89
6	15.14	199.73	11.94	160.00	217.12	24.61	-69.78	24.61
<b>Average</b>	<b>15.07</b>	<b>207.06</b>	<b>11.25</b>	<b>169.17</b>	<b>223.56</b>	<b>24.31</b>	<b>-61.53</b>	<b>24.31</b>

Table 4.3 : The length of the cylindrical shape of the zinc oxide NP.

No of reading	Area (nm <sup>2</sup> )	Mean (nm)	StdDev (nm)	Min	Max	Perim. (nm)	Angle	Length (nm)
1	37.29	204.05	10.42	183.05	229.57	60.75	-8.05	60.75
2	45.04	203.63	16.96	178.91	237.76	73.48	-60.26	73.48
3	32.12	175.73	9.51	158.63	197.78	52.26	-156.71	52.26
4	32.86	208.84	13.77	178.00	247.40	53.35	-52.87	53.35
5	31.38	200.32	11.54	181.58	230.00	51.22	-131.63	51.22
<b>Average</b>	<b>35.74</b>	<b>198.51</b>	<b>12.44</b>	<b>176.03</b>	<b>228.50</b>	<b>58.21</b>	<b>-81.90</b>	<b>58.21</b>

## 4.2 Pendant Drop test.

A pendant drop test was carried to find the interfacial tension (IFT) and surface tension (ST) of the liquids. IFT is calculated between two liquid surfaces and ST is calculated in between fluid and air surfaces.

### 4.2.1 Pendant drop test for Angsi crude oil.

Firstly, the angsi crude oil is tested for IFT with air. Figure 4.5 below shows the pendant drop test result between angsi crude oil and air. According to the (Kelechukwu 2011), the Wax Appearance Temperature (WAT) of Angsi crude oil is around 40°C. Thus, the angsi crude oil was heated to 40°C due to the WAT. If the temperature below 40°C is used, then the oil will become too sticky which cause the oil to clog in the syringe. The surface tension between angsi oil with air as the second phase is 25.06 mN/m.

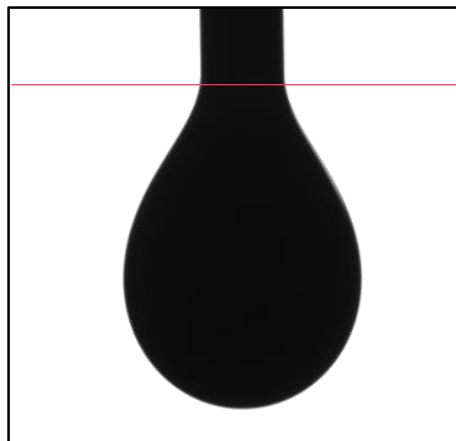


Figure 4.5 : Pendant drop test between angsi crude oil with air as the second phase.

The interfacial tension between angsi oil and ZnO NP nanofluid cannot be proceed due to the nature of the ZnO nanofluid that is 'cloudy'. The goniometer machine requires clear solution as a medium in order to calculate interfacial tension. Due to this reason, instead of IFT, the ST is calculated for the ZnO with different concentration.

#### 4.2.1 Pendant drop test for ZnO nanofluid.

Pendant drop test for the ZnO NP was carried out by using air as the second phase. This method was conducted due to the drop phase cannot be identified when the crude oil is used. The light from the source cannot penetrate the oil which lead the camera cannot capture the image formed from the pendant drop test.

The angsi crude oil was heated to 40°C due to the WAT. If the temperature below it is used, then the oil will become too sticky which cause the oil to clog in the syringe. On the other hand, the temperature cannot be more than 40°C because it will cause the nanofluid to boil and cause bubble trap below the glass surface.

The result obtained from the pendant drop test is shown in the table 4.4 below.

Table 4.4 : The surface tension of ZnO nanofluid with different concentration.

ZnO NP concentration (wt%)	Surface Tension (mN/m)
0.01 wt%	71.88
0.05 wt%	70.38
0.10 wt%	69.24

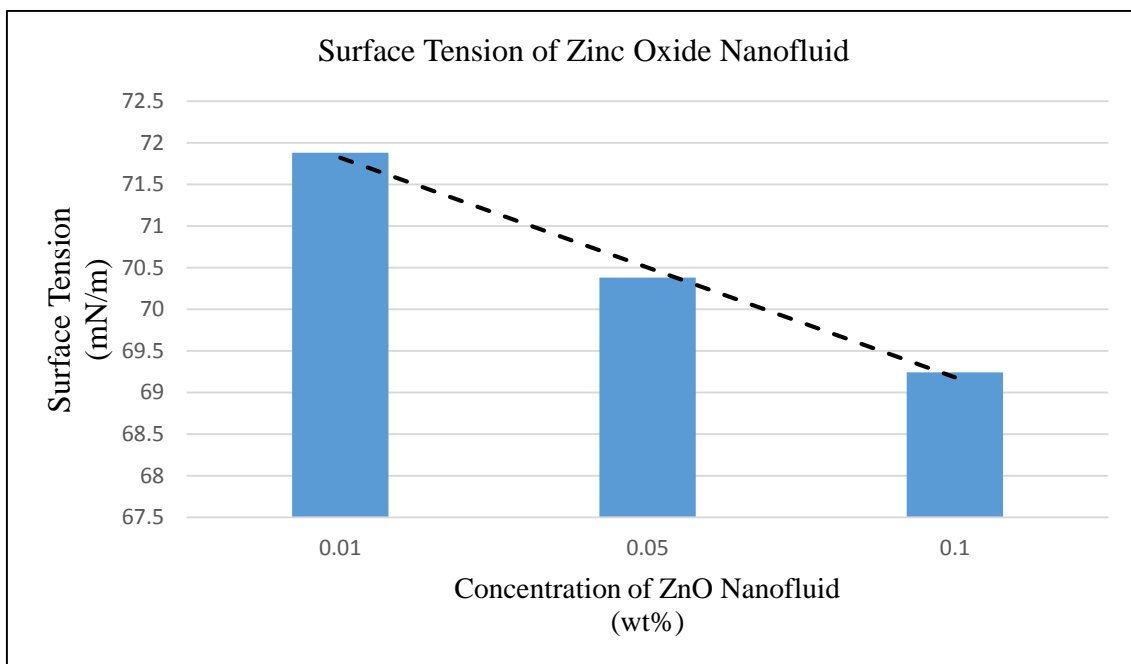


Figure 4.6 : The graph of surface tension for the different concentration of ZnO nanofluid.

Based on the data obtained as shown in figure 4.6, the ST of the ZnO nanofluid is decreasing as the concentration of nanofluid increase. The lower the surface tension indicates that the intermolecular force within the fluid drop to contract the surface is low which make it attach stronger to the surface.

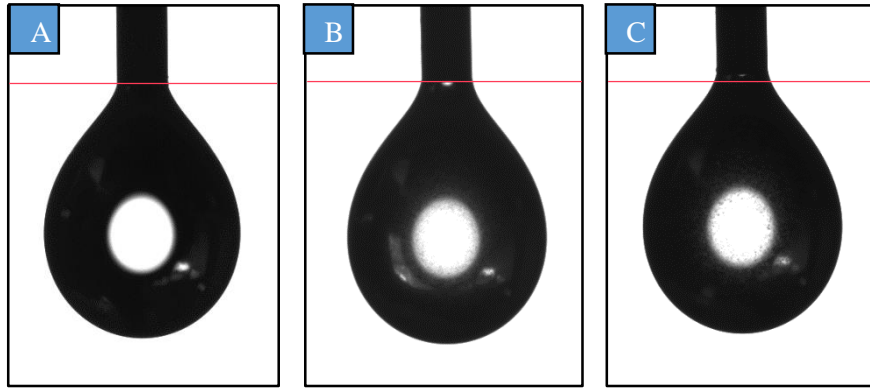


Figure 4.7 : The pendant drop test of ZnO nanofluid with the concentration of a) 0.01wt% b) 0.05wt% c) 0.10wt%

As shown in the figure 4.7, the ZnO particles can clearly be seen in the pendant drop test for the ZnO nanofluid with the concentration of 0.05wt% and 0.10wt%.

### 4.3 Contact angle test.

Contact angle test was carried to determine the wettability of the angsi crude oil in the zinc oxide nanofluid which work as a second phase or medium. In this experiment, the inverted contact angle method is used instead of standard contact angle technique because it is to eliminate the effect of air to the droplet of oil. The setup of the experiment is illustration as shown in figure 4.10

Due to the nature of the ZnO nanofluid which is 'cloudy', the droplet of angsi oil is let to stabilized for 30 minutes. At the same time give space to the nanoparticles to slowly drop and gives a clear image of droplet so the precise calculation can be done. The result obtained from the pendant drop test is shown in the table 4.5 below.

Table 4.5 : The contact angle of Angsi crude oil in the ZnO nanofluid with different concentration.

ZnO NP concentration (wt%)	Contact Angle (°)
0.01 wt%	101.56°
0.05 wt%	94.34°
0.10 wt%	86.04°

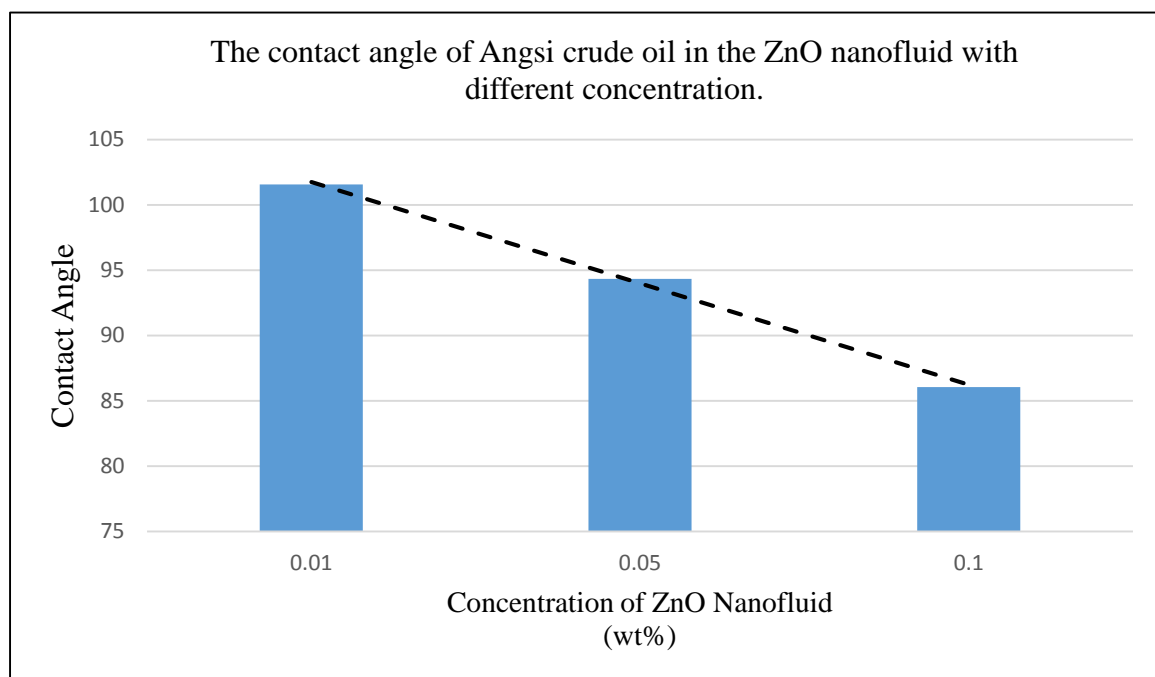


Figure 4.8 : The graph of contact angle of angsi crude oil in the different concentration of ZnO nanofluid.

The result of the contact angle obtained as shown in the table 4.5 and graph in figure 4.8, the higher concentration of ZnO nanofluid, lower the contact angle. Lower the contact angle indicate that it is more water wet. It proves that, the ZnO with the higher concentration has more tendency to adhere to the surface which replace the oil. Thus, oil is easily to be removed and extracted.

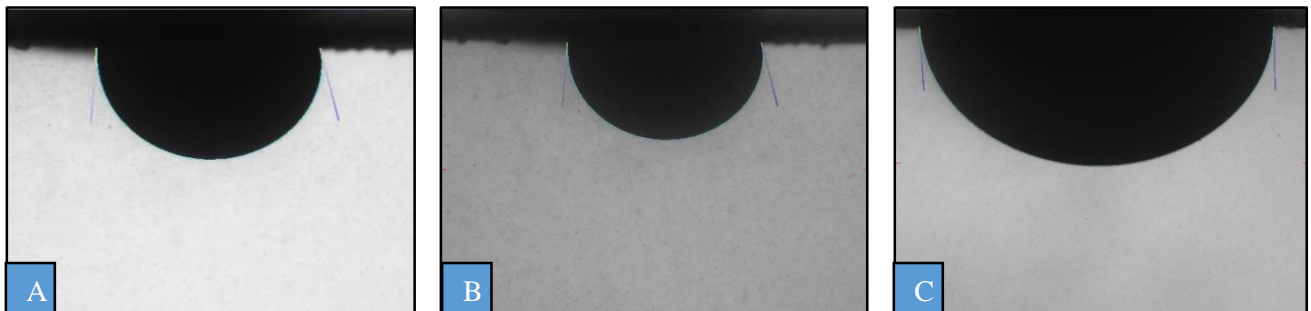


Figure 4.9 : The contact angle of Angsi crude oil in the ZnO nanofluid with concentration of  
a) 0.01wt% b) 0.05wt% and c) 0.10wt%



## **CHAPTER 5**

### **CONCLUSIONS AND RECOMMENDATIONS**

#### 5.1 Conclusion

In conclusion, zinc oxide nanoparticle can reduce the interfacial tension between oil and water. The small size of nanoparticle be able to amplify the properties of the solution. In this case, the nanoparticle would help to attack the surface of attachment between oil and water which reduce its interfacial tension. Thus, the oil is not sticking to the water surface. Due to this reason, the mobility of the oil inside the well can be increased which lead to the extraction of the oil to be at the optimum capacity. Considering the ZnO is cheap and available, it is one of the suitable alternatives to replace the current method to extract the oil. For this experiment, the IFT is not directly measured by using IFT machine instead, the IFT is analyzed through the contact angle testing and pendant drop testing.

By the end of the experiment, all the objectives of this experiment were successfully achieved:

1. Based on the characterization of ZnO by using FESEM and XRD, it was confirmed the present of the ZnO compound. The nanofluid is prepared by dissolving the ZnO nanoparticle in the 35000 ppm brine solution which is to simulate the real condition of reservoir. Through the contact angle test and pendant drop test, the surface tension and wettability of the solution can be calculated.
2. Contact angle test for 0.10wt% concentration of ZnO gives lower contact angle, 86.04° compared to the 0.01wt% and 0.05wt% concentration of ZnO nanoparticle, 94.34° and 86.04° respectively. Lower contact angle of the oil means the surface is more water-wet. Thus, it is easier for the oil to be removed from the surfaces.
3. For surface tension, with higher concentration of ZnO, 0.10wt%, lower surface tension, 69.24 mN/m is obtained compared to 0.01wt% and 0.05wt%. which gives 71.88 mN/m and 70.38 mN/m. Lower the surface tension indicates that the intermolecular force within the fluid drop to contract the surface is low which make it attach stronger to the surface.

In the nutshell, we can conclude that, the presence of nanoparticle can help to reduce the interfacial tension between oil and water which lead to the increasing of mobility of the crude oil. In this case, zinc oxide nanoparticles show a great potential to be used in the operating oil and gas industry. Should be there any extended research continued from this, we might be able to identify more characteristic and factors that would affect the performance of the zinc oxide nanoparticles.

## 5.2 Recommendation

A recommendation that could be suggested is to synthesis the zinc oxide instead of having it directly from the supplier. It is a lengthy process but by synthesizing the material by our own, we can expect the result according to the synthesis process that the material has gone through.

Next, for the contact angle test, it is recommended to investigate the effect of the high temperature towards the angle of the droplet produced. This is because the higher the temperature, lower the viscosity which lead to the lower interfacial tension between the two surfaces. For this experiment, contact angle test with temperature more than 40°C is not possible due to the present of the bubble which may affect the reading of the oil droplet produced.

Next it is also recommended to do spinning drop test that can operate in high temperature and high pressure to check for the interfacial tension. This is due to the lack of the equipment specification that allow high temperature and high pressure testing.

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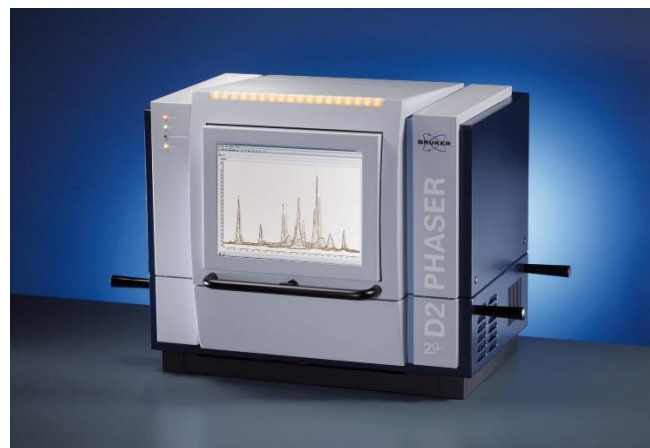
## APPENDICES

### APPENDIX A : Characterization machine



Field Emission Scanning Electron Microscope (FESEM).

SUPRA 55VP (ZEISS)



X-Ray Diffraction. Model D2 Phaser (Bruker)



XRD sample holder.