SYNTHESIS AND CHARACTERIZATION
OF
ZnO-CNTs FILLED PVA COMPOSITE AS EM DETECTOR

By

AHMAD FAUZIE BIN MASURI

FINAL PROJECT REPORT

Submitted to the Electrical & Electronics Engineering Programme
in Partial Fulfillment of the Requirements
for the Degree
Bachelor of Engineering (Hons)
(Electrical & Electronics Engineering)

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CERTIFICATION OF APPROVAL

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December 2009

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.

Ahmad Fauzie Bin Masuri

ABSTRACT

An Electromagnetic (EM) composite based detector filled with ZnO and carbon nanotubes (CNTs) nanoparticles was developed. This work premise deals with synthesis of ZnO using self combustion and sol-gel techniques. These two techniques were chosen in order to compare which technique will give a better result of ZnO nano structure. The mixture consists of 10grams of zinc (II) nitrate, $Zn(NO_3)_2.6H_2O$ salt was dissolved in 50mL of nitric acid, HNO_3 and stirred at 250 r.p.m continuously for 24 hours. The mixture was then gradually heated for every 15 minutes until it combusted at 110°C for the self combustion technique. For sol gel technique, the dissolved mixture was heated at 40°C, 50°C, 60°C and 70°C until the gelatin is formed. After drying process, the as-prepared samples were annealed at 150°C and 250 °C for 2 hour for each technique. Morphology and phase characteristic was covered in the characterization part. Characterizations were done by using X-Ray Diffractions (XRD), Energy Dispersion X-ray Spectroscopy (EDX) and Field Emission Scanning Electron Microscope (FESEM). Single phase crystal nano structure of ZnO was successfully obtained. The morphology of ZnO is shown in the nano-size dimension. These samples were used as additive to the PVA composite detector. The PVA composite was prepared using casting method. The result from the testing and validation part shows that the presence of ZnO and CNTs as a filler in the PVA composite detector can enhance the detection up to 70%.

ACKNOWLEDGEMENT

Alhamdulillah, in the name of Allah, Most Gracious and Most Merciful, thank God for His blessing and guidance, at last this Final Year Project successfully completed. The acknowledgement would be tribute towards my Final Year Project supervisor which is Associate Professor Dr. Noorhana Yahya. During the progress of the FYP, Dr. Noorhana provided lots of feedback that is related to the project as well as provided her valuable experience on the field. She also introduced to me the aspects needed to do in my FYP and the methods which are to be used to analyze the project so that the needed result can be obtained. Besides that, Dr. Noorhana always encouraged me to do the best and so that the project can be solved by multiple methods and that it is important that the objective of this project is achieved.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Detecting and assessing hydrocarbon reservoirs without the need to drill test wells is of major importance to the petroleum industry [1]. Seismic methods have traditionally been used in this context, but the results can be ambiguous [1]. Another approach is to use electromagnetic sounding methods that exploit the resistivity differences between a reservoir containing highly resistive hydrocarbons and one saturated with conductive saline fluids [1].

The seabed logging (SBL) method is a remote resistivity sensing method which exploits the facts that hydrocarbons are electric insulators and consequently, the hydrocarbon filled reservoirs normally are more resistive than surrounding water-filled sediments.

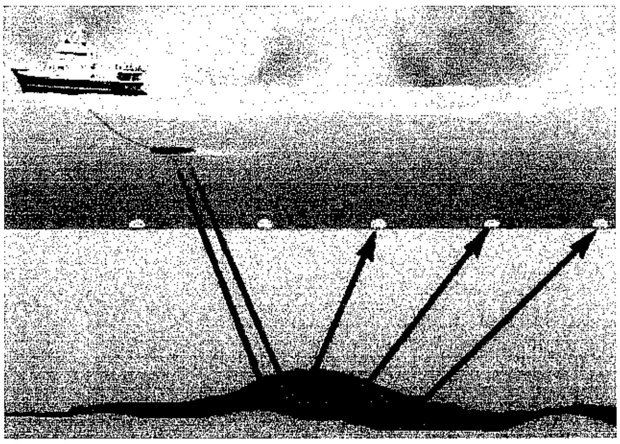


Figure1: Sea Bed Logging Working Principle [2]

The working principles of SBL is, firstly, a ship tows a horizontal electric dipole source which is act as EM transmitter close to the seabed to create a large electric field. As the electric field propagates through the subsurface, it is perturbed by any variations in the subsurface resistivity. The electric and magnetic fields are both measured and recorded by highly sensitive units distributed over the seabed. Then, once sufficient data has been recorded, an acoustic signal is sent to the receivers to trigger a release mechanism, and the recorders return to the surface for data analysis. After that, the recorded data is processed to remove noise, and compensate for environmental variations, such as water depth and background resistivity. In many cases, this data can then be interpreted directly. Increasingly, however, the data is imaged using depth migration or inversion to facilitate easy integration with seismic and other subsurface data.

1.2 Problem Statement

Designing EM detector for very high sensitivity and high accuracy is very important but still remain a challenge.

1.3 Objective and Scope of Study

1.3.1 Objective

This project is to design a working prototype of electromagnetic detector for shallow water for SBL

(1) To synthesis ZnO nano particles by using self-combustion and sol-gel techniques;

(2) To synthesis a composite with ZnO as filler and act as an EM detector.

(3) To characterize the chemical and electrical properties of ZnO and CNTs.

(4) To developed EM detector prototype potentially used for exploration of hydrocarbon in oil & gas industry.

1.3.2 Scope of study

The area of study for this project will be divided to 3 parts: the designing EM detector, antenna designation and the environment to be applied. The target user is for the petroleum industry to detect and assess hydrocarbon reservoirs without the need to drill test wells that is used in SBL.

CHAPTER 2

LITERATURE REVIEW

2.1 Electromagnetic Detector

An electromagnetic (EM) detector is needed to detect the hydrocarbon in a wide range of environments; high quality data from the high sensitivity and low noise detector/receiver are needed for the oil and gas industry.

The EM detector must be capable of measuring field strength that vary greatly in magnitude, from weak naturally occurring magneto telluric signals to strong direct signals from the source, and be capable to measuring the EM phase with respect to the source requires precise measurement of the timing of the signal.

For receiver to be accurately positioned on the seabed, they must be designated to descend rapidly when dropped from the survey vessel, and is large surveys are to be performed, to remain autonomously operational for several weeks.

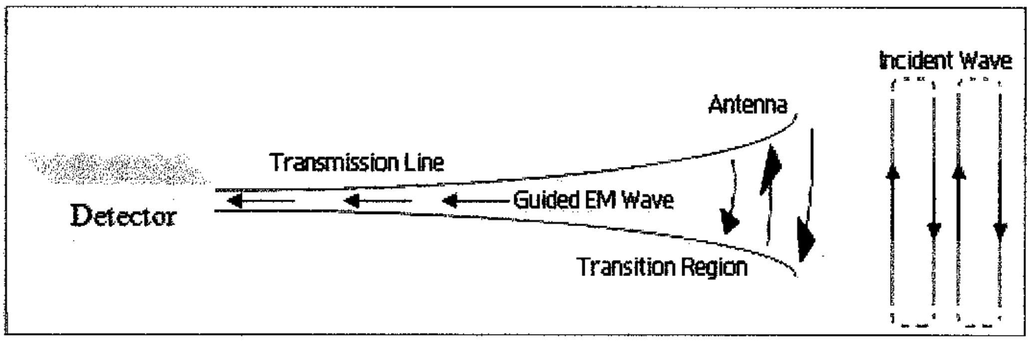


Figure 2: Detecting EM Wave Block Diagram [3]

2.2 Antenna

An antenna is defined as a transducer between guided wave propagating in a transmission line and an unbounded medium or vice versa [3]. Figure 2 show how a wave is launched by an antenna, with the acting as a transition between the waveguide and free space. Any material whether conductor or dielectric to receive EM energy with directional and polarization properties suitable for the intended application. It is important to know the impedance of the antenna and to match it to the transmission line for minimize reflection at the transmission line-antenna juncture purposes. Antenna for EM detector can be made in various shapes and sizes. The radiation and impedance properties of an antenna are governed by its shape and size and the material of which it is made [3]. The dimensions of an antenna are usually measured in units of wavelength of the wave it is launching or receiving [3].

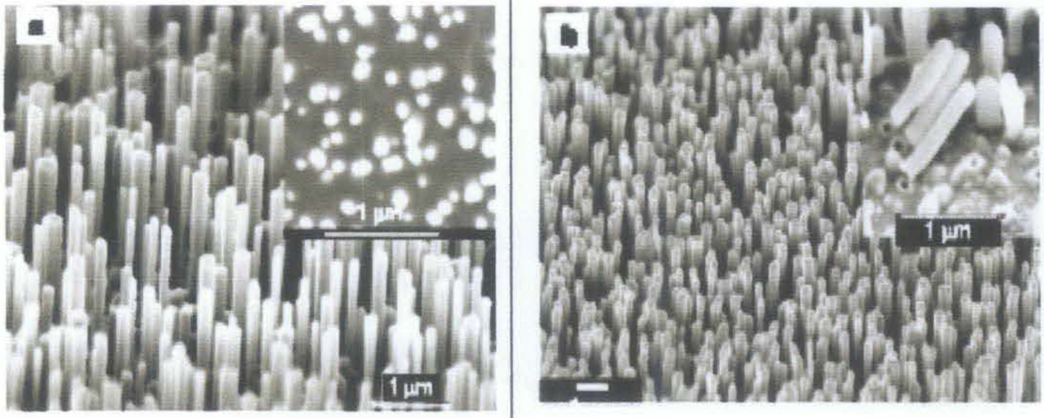
To design good antenna for EM detector, the reciprocity, radiation sources, far field region and the antenna arrays need to be considered. Reciprocity is more discuss about the antenna radiation pattern to have a good performance while designing antenna polarization and antenna impedance. The radiation sources are depends on the types that are used. Besides that, the material is going to be used as an electromagnetic wave detector must have the excellent performance.

2.3 Zinc Oxide(ZnO)

Nanostructures ZnO materials have received considerable interest from scientists due to their remarkable performance in electronics, optics and photonics. As early as the 1960s, synthesis of ZnO thin films was an active field because of applications in sensors, transducers and as photo catalysts. In the last few decades, study of one-dimensional material has gained importance in nano science and nanotechnology. With reduction in size, novel electrical, mechanical, chemical and optical properties are introduced resulting from surface and quantum confinement effects. ZnO is a significant technological material. The absence of a centre of symmetry in its wurtzite structure, along with large electromechanical coupling, results in strong piezoelectric and pyroelectric properties. ZnO is therefore widely used in mechanical actuators and piezoelectric sensors. [4]

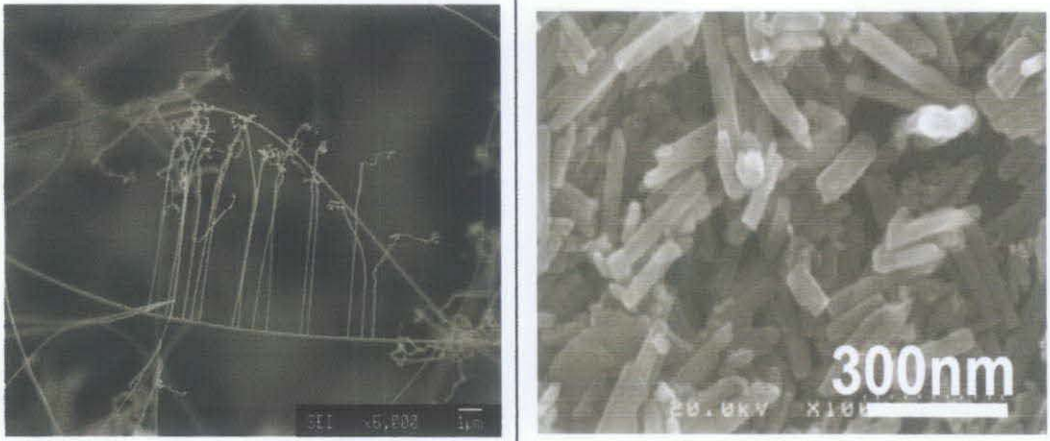
In addition, ZnO is a wide band-gap (3.37 eV) compound semiconductor that is appropriate for short wavelength optoelectronic applications. The high exciton binding energy (60 meV) in ZnO crystal allows efficient excitonic emission at room temperature. ZnO is transparent to visible light and its conductivity can be increased through doping. ZnO nanostructures have a wide range of high technology applications like surface acoustic wave filters, photonic crystals, photodetectors, light emitting diodes, photodiodes, gas sensors, optical modulator waveguides, solar cells, and varistors[4]. ZnO is also receiving a lot of attention because of its antibacterial property and its bactericidal efficacy has been reported to increase as the particle size decreases [18]. The discovery of carbon nanotubes by Iijima [4] in 1991 has initiated active research leading to the growth and characterization of one-dimensional nanowires of elemental and compound semiconductors such as Si, Ge, InP, GaAs, and ZnO[4]. Different nanostructures of ZnO have been reported such as nanowires and nanorods, nanocombs, nanorings, nanoloops and nanohelices, nanobows, nanobelts, and nanocages[4].

ZnO nanostructures can be grown either in solution or from gaseous phase. The gas phase synthesis methods are expensive and complicated [4]. The solution phase synthesis is usually done in water [4]. The hydrothermal process of growing ZnO nanostructures has gained immense popularity due to its simplicity and tolerable growth conditions [4]. As synthesis is carried out in aqueous solution, the growth temperatures are less than the boiling point of water [4]. Figure 3 show the various type of ZnO nanostructure [5, 6, 7, 8].



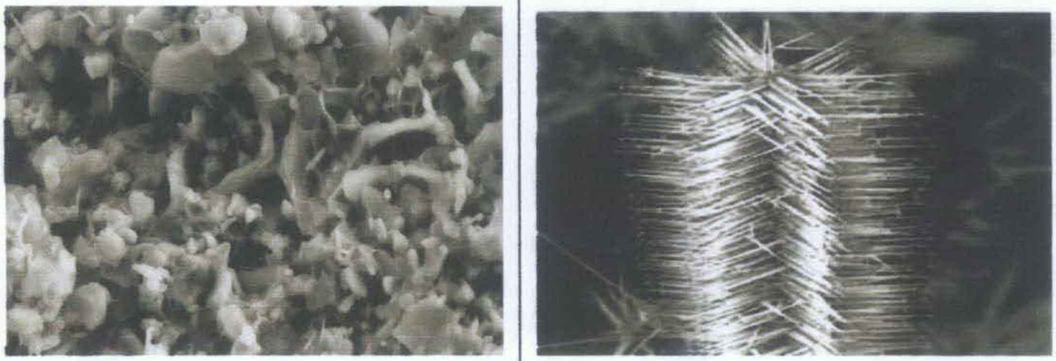
a) ZnO Nano-rods Structure

b) ZnO Nano-tubes Structure



c) ZnO Nano-wire Structure

d) ZnO Nano-rod Structure



e) ZnO Nano-flake Structure

f) ZnO Nano-needle Structure

Figure 3: Various Type of ZnO Nano Structure [5, 6, 7, 8]

2.4 Polyvinyl Alcohol (PVA)

Polyvinyl alcohol has excellent film forming, emulsifying, and adhesive properties. It is also resistant to oil, grease and solvent. It is odorless and nontoxic. It has high tensile strength and flexibility, as well as high oxygen and aroma barrier properties. However these properties are dependent on humidity, in other words, with higher humidity more water is absorbed. The water, which acts as a plasticiser, will then reduce its tensile strength, but increase its elongation and tear strength. PVA is fully degradable and is a quick dissolver. PVA has a melting point of 230°C and 180–190°C for the fully hydrolysed and partially hydrolysed grades. It decomposes rapidly above 200°C as it can undergo pyrolysis at high temperatures. PVA is an atactic material but exhibits crystallinity as the hydroxyl groups are small enough to fit into the lattice without disrupting it. [9] Figure 4 shows the PVA atomic molecules bonding structure.

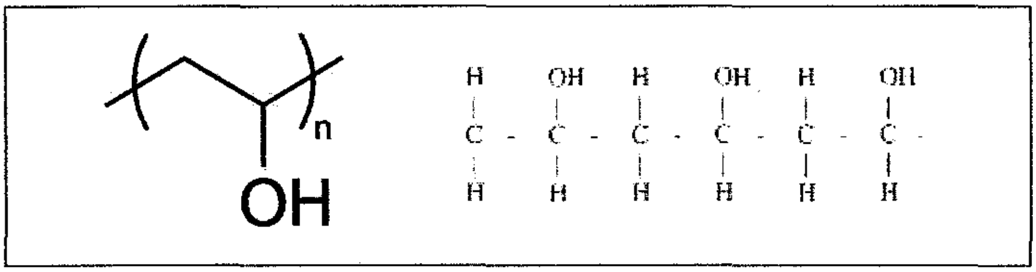


Figure 4: PVA molecules bonding structure [9]

2.5 Carbon Nanotubes (CNTs)

Carbon nanotubes are related to graphite. The molecular structure of graphite resembles stacked, one-atom-thick sheets of chicken wire - a planar network of interconnected hexagonal rings of carbon atoms. In conventional graphite, the sheets of carbon are stacked on top of one another, allowing them to easily slide *over each other. That is* why graphite is not hard, but *it* feels greasy, and can be used as a lubricant. When graphene sheets are rolled into a cylinder and their edges joined, they form CNTs. Only the tangents of the graphitic planes come into contact with each other, and hence their properties are more like those of a *molecule.* [10]

CNTs come in a variety of diameters, lengths, and functional group content. CNTs today are available for industrial applications in bulk quantities up metric ton quantities from Cheap Tubes. Several CNT manufacturers have >100 ton per year production capacity for multi walled nanotubes. [10]

A nanotube may consist of one tube of graphite, a one-atom thick single-wall nanotube, or a number of concentric tubes called multiwalled nanotubes. When viewed with a transmission electron microscope these tubes appear as planes. Whereas single walled nanotubes appear as two planes, in multi walled nanotubes more than two planes are observed, and can be seen as a series of parallel lines. There are different types of CNTs, because the graphitic sheets can be rolled in different ways. The three types of CNTs are Zigzag, Armchair, and Chiral. It is possible to recognize zigzag, armchair, and chiral CNTs just by following the pattern across the diameter of the tubes, and analyzing their cross-sectional structure. [10]

Multi walled nanotubes can come in an even more complex array of forms, because each concentric single-walled nanotube can have different structures, and hence there are a variety of sequential arrangements. The simplest sequence is when concentric layers are identical but different in diameter. However, mixed variants are possible, consisting of two or more types of concentric CNTs arranged in different orders. These can have either regular layering or random layering. The structure of the nanotube influences its properties - including electrical and thermal conductivity, density, and lattice structure. Both type and diameter are important. The wider the diameter of the nanotube, the more it behaves like graphite. The narrower the diameter of the nanotube, the more its intrinsic properties depends upon its specific type[10]. Figure 5 shows the structure of CNTs; [11] Figure 6 shows the morphology of the CNTs.

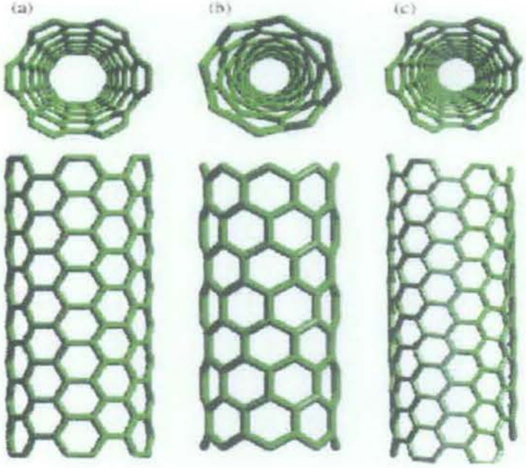


Figure 5: CNTs structure; (a) armchair, (b) zigzag, (c) chiral [11]

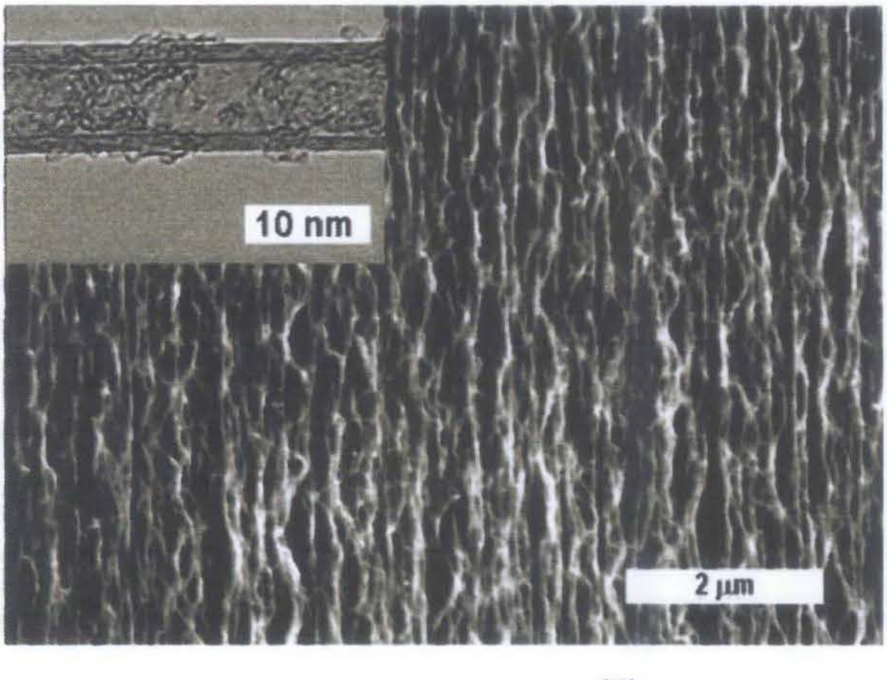


Figure 6: CNTs morphology [12]

CHAPTER 3

METHODOLOGY

3.1 Project Identification

3.1.1 Project flow

The project started with choosing the title and sketching the expected outcome based on current knowledge. This project has been divided to two parts; the synthesis and the characterization of zinc oxide.

However, characterization of zinc oxide was done. After confirming the expected result, the detector system of the expected design will be developed and it will be tested through experimental work. The whole project flow can be seen as shown in Figure 7.

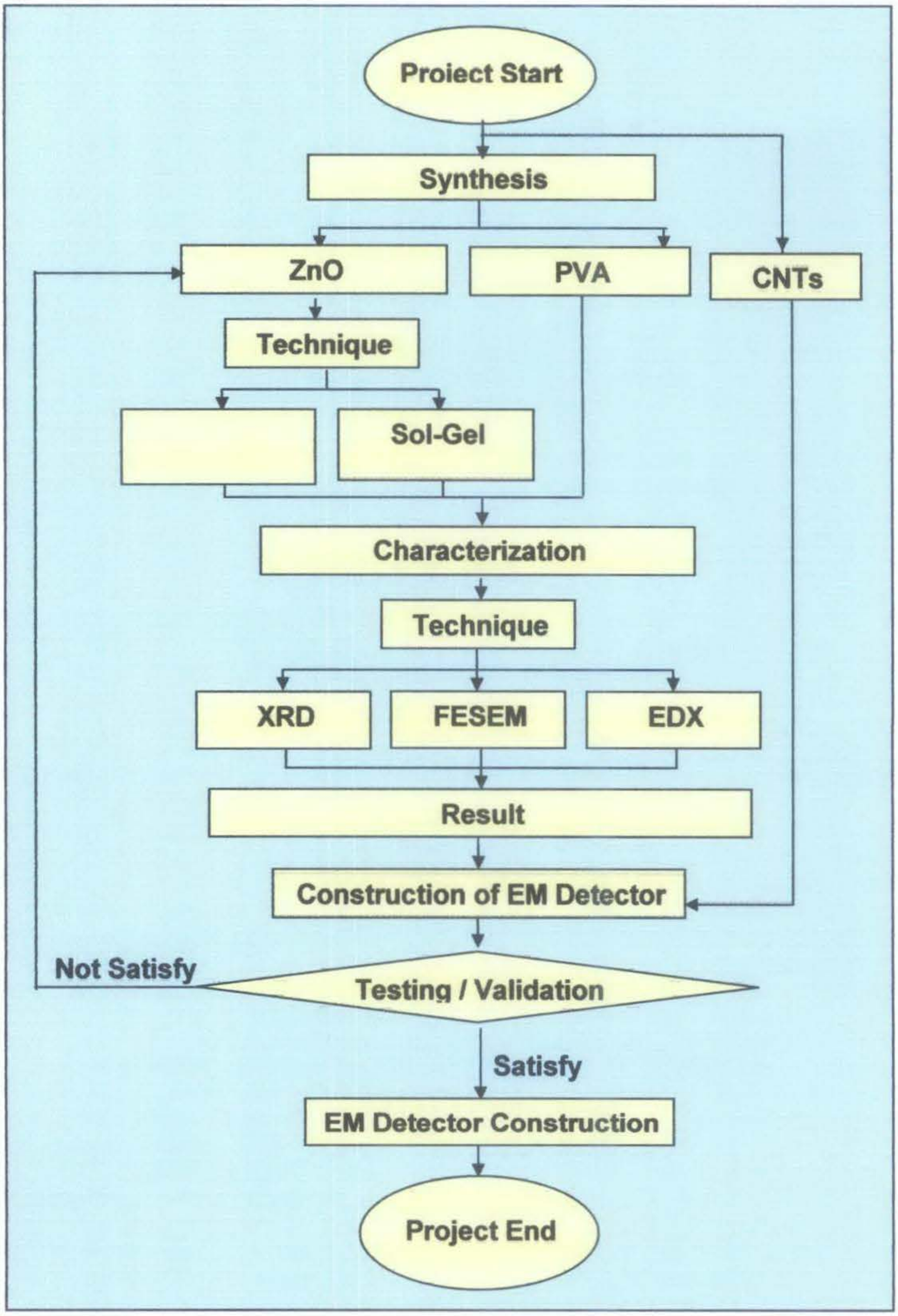


Figure 7: Project methodology and identification

3.2 Tools Required

Tools required for this project consists of software and hardware. The tools are listed as below:

3.2.1 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. [13]

3.2.2 *Field Emission Scanning Electron Microscope (FESEM)*

A field-emission cathode in the electron gun of a scanning electron microscope provides narrower probing beams at low as well as high electron energy, resulting in both improved spatial resolution and minimized sample charging and damage. The application of FESEM include Semiconductor device cross section analyses for gate widths, gate oxides, film thicknesses, and construction details, advanced coating thickness and structure uniformity determination and small contamination feature geometry and elemental composition measurement. FESEM is needed because of; [14]

i. FESEM produces clearer, less electrostatically distorted images with spatial resolution down to 1 1/2 nm. That's 3 to 6 times better than conventional SEM.
ii. Smaller-area contamination spots can be examined at electron accelerating voltages compatible with Energy Dispersive X-ray Spectroscopy.
iii. Reduced penetration of low kinetic energy electrons probes closer to the immediate material surface.

iv. High quality, low voltage images are obtained with negligible electrical charging of samples. (Accelerating voltages range from 0.5 to 30 kV.)
v. Need for placing conducting coatings on insulating materials is virtually eliminated.

3.2.3 *X-Ray Diffraction (XRD)*

X-ray scattering techniques are a family of non-destructive analytical techniques which reveal information about the crystallographic structure, chemical composition, and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy. [15]

X-ray diffraction finds the geometry or shape of a molecule using X-rays. X-ray diffraction techniques are based on the elastic scattering of X-rays from structures that have long range order. The most comprehensive description of scattering from crystals is given by the dynamical theory of diffraction.[15]

3.2.4 Raman Spectroscopy

Raman spectroscopy is a spectroscopic technique used in condensed matter physics and chemistry to study vibration, rotational, and other low-frequency modes in a system. It relies on inelastic scattering, or Raman scattering, of monochromatic light, usually from a laser in the visible, near infrared, or near ultraviolet range. The laser light interacts with phonons or other excitations in the system, resulting in the energy of the laser photons being shifted up or down. The shift in energy gives information about the phonon modes in the system. Infrared spectroscopy yields similar, but complementary, information. [16]

3.2.5 Energy Dispersive X-ray spectroscopy (EDX)

Energy dispersive X-ray spectroscopy (EDX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It is one of the variants of XRF. As a type of spectroscopy, it relies on the investigation of a sample through interactions between electromagnetic radiation and matter, analyzing x-rays emitted by the matter in response to being hit with charged particles. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing x-rays that are characteristic of an element's atomic structure to be identified uniquely from each other [17].

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Synthesis of Zinc Oxide

ZnO nano particles were prepared by employing two techniques, which are sol-gel and self-combustion techniques.

4.1.1 Self Combustion Technique

In the self combustion technique, nanoparticles sample were obtained by heating the materials until the mixture combusts at 110°C. The combustion is continually until the samples were in gold-brown colors.

4.1.1.2 Experimental Procedure

This experiment was started by dissolving zinc nitrate, $Zn(NO_3)_2.6H_2O$ salt into 65% concentrated nitric acid, HNO_3 solution for this self combustion technique. Refer to Figure 8.

Figure 8: Chemicals used for ZnO synthesis.

10mg of $Zn(NO_3)_2.6H_2O$ salt and 50ml of HNO_3 is measured by using the balancer. The accurate reading in the balancing process must be taking into consideration to make sure the solvent produced is at the right amount. The chemicals needed were weighed by using the electronics balancer, ROSS Model MK11, which can provide measurement until 4 decimals points. Refer to Figure 9.

Figure 9: Electronics balancer to weight the amount of chemicals accurately.

After both chemicals being mix up, both solutions were stirred for 24 hours to form a homogeneous sol. Refer to Figure 10 and Figure 11.

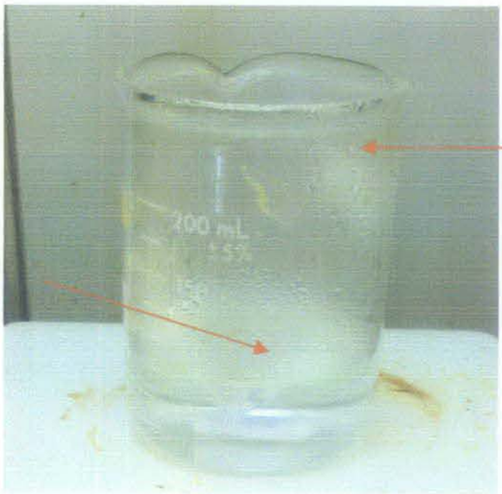


Figure 10: Solvent for self-combustion technique

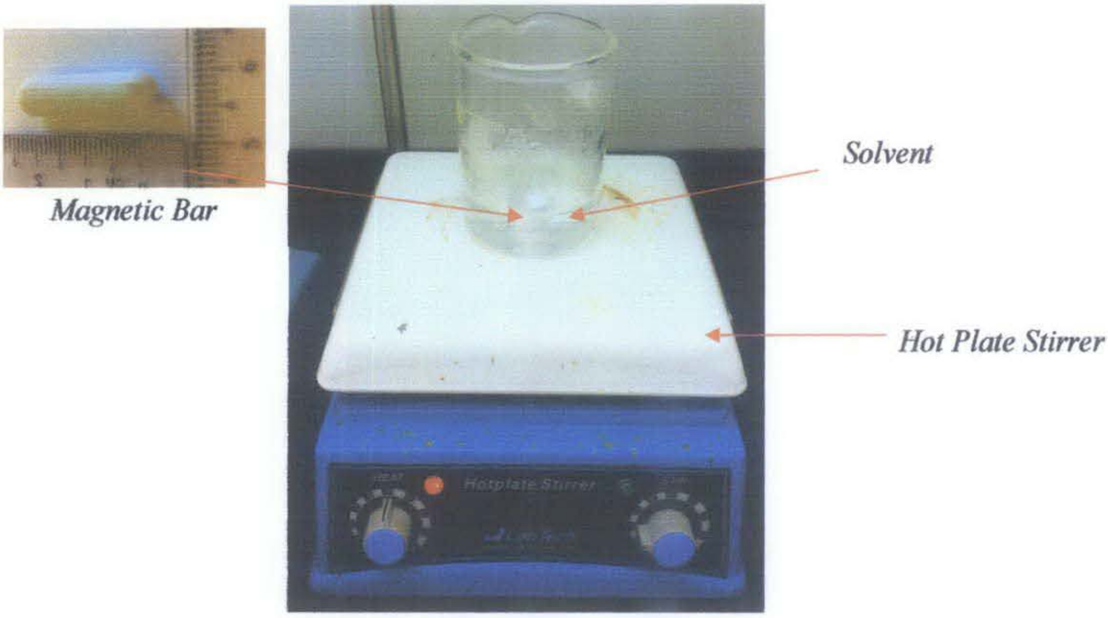


Figure 11: Stirring and heating using hotplate stirrer.

Meanwhile, the homogenized sol solution was also heated until it combusted for at 110°C for this self combustion technique. Refer to Figure 12.

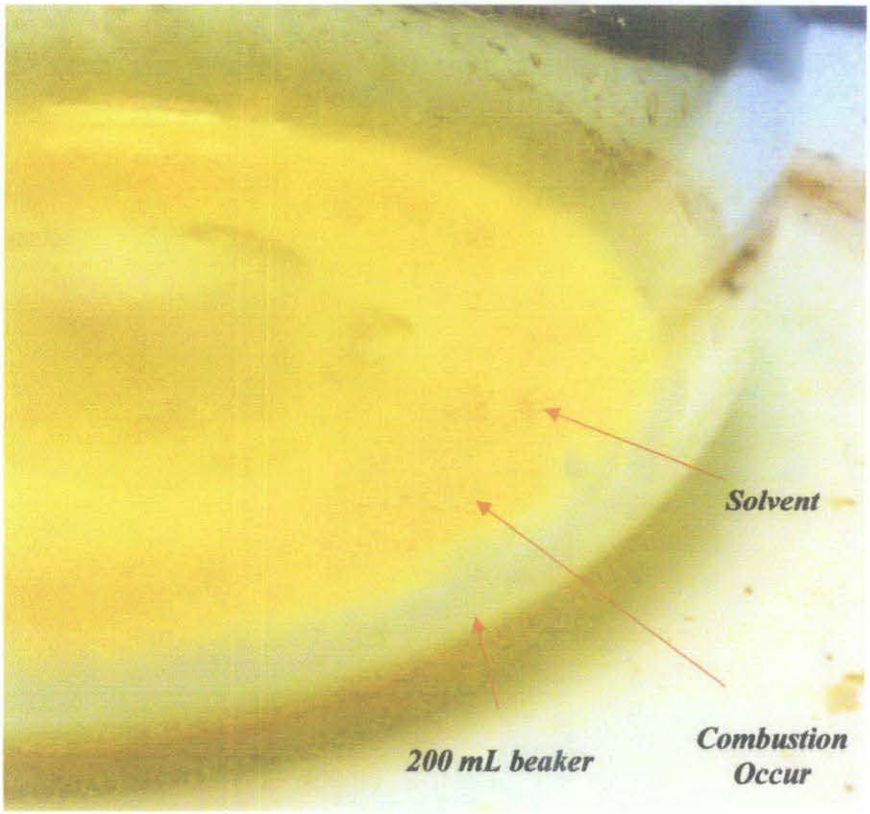


Figure 12: Combustion occurs

Then, the samples were dried in an oven at 110°C for 24 hours for dehydration purpose.

Samples were put in oven for 48 hours at 110 °C

Figure 13: Dehydration process in oven at 110 °C.

After that, the dried samples were crushed for 2 hour to obtain the fine particles.

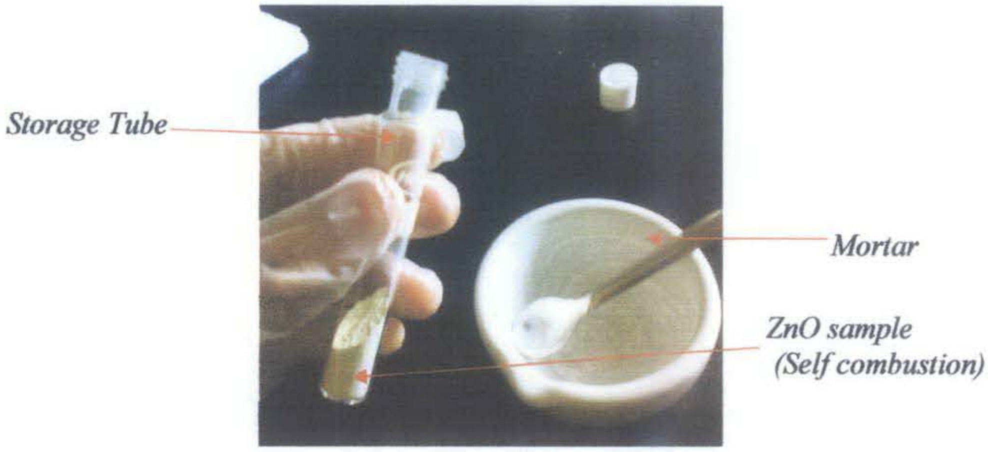


Figure 14: Storing the sample in storage tube after crushing.

After crushing process, the as-prepared samples were annealed at 250°C and 350°C for 2 hour. The samples were divided into two for the use of pre-sinter process at these 2 temperatures. The purpose is to compare which annealing temperature will give the good result for this ZnO synthesis. Refer figure 15.

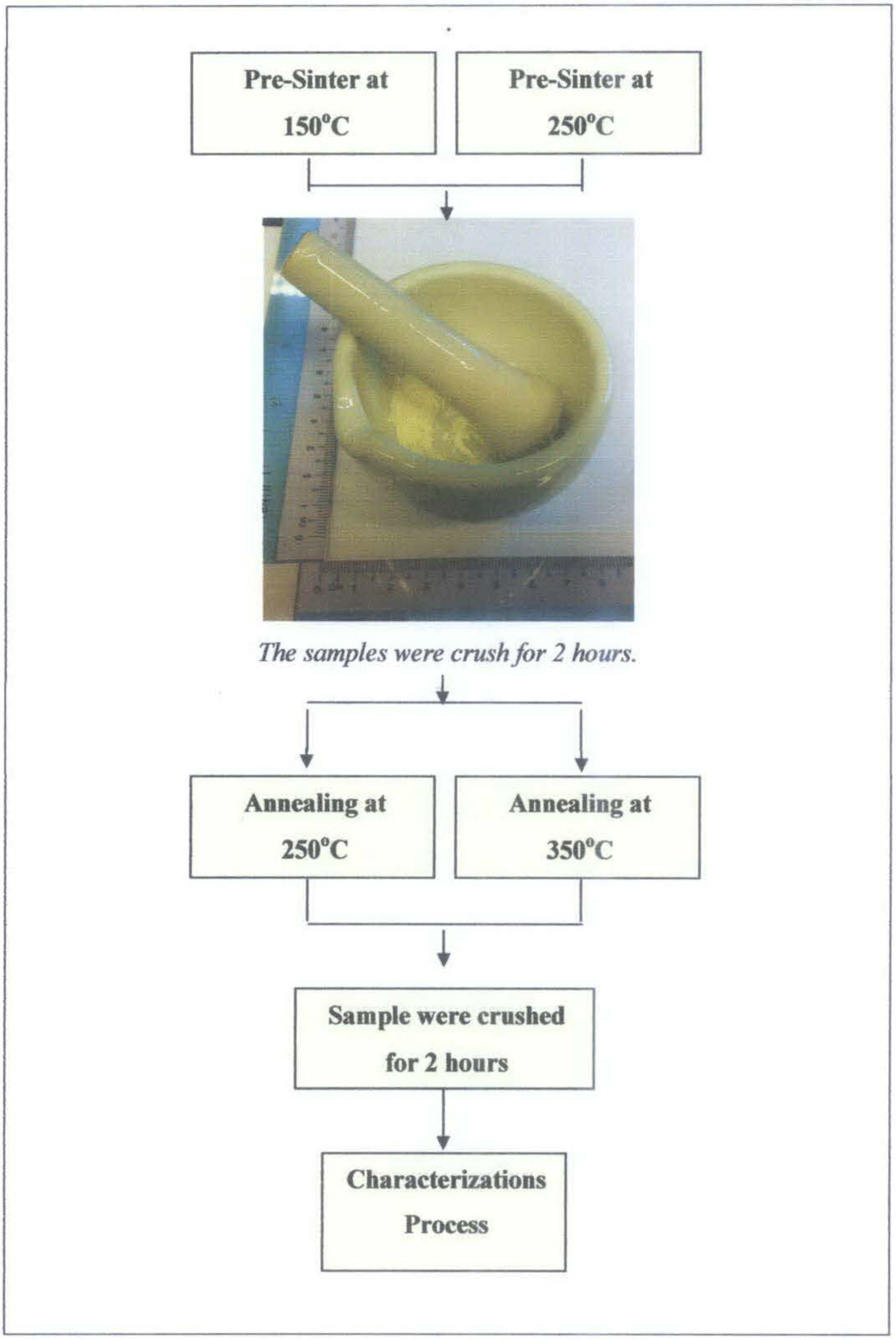


Figure 15: Planning for annealing flow chart

4.1.2 Sol-Gel Technique

In the sol gel technique, nanoparticles sample were obtained by heating the materials until the mixture combusts at 110°C. After the early combustion occurred, heat applied to the samples was stop immediately.

4.1.2.2 Experimental Procedure

This experiment was started by dissolving zinc nitrate, $Zn(NO_3)_2.6H_2O$ salt into 65% concentrated nitric acid, HNO_3 solution for this self combustion technique. Refer to Figure 8.

10mg of $Zn(NO_3)_2.6H_2O$ salt and 50ml of HNO_3 is measured by using the balancer. The accurate reading in the balancing process must be taking into consideration to make sure the solvent produced is at the right amount. The chemicals needed were weighed using the electronics balancer, ROSS Model MK11, which can provide measurement until 4 decimals points. Refer to Figure 9.

After both chemicals being mix up, both solutions were stirred for 24 hours to form a homogeneous sol. Refer to Figure 11 and Figure 16.

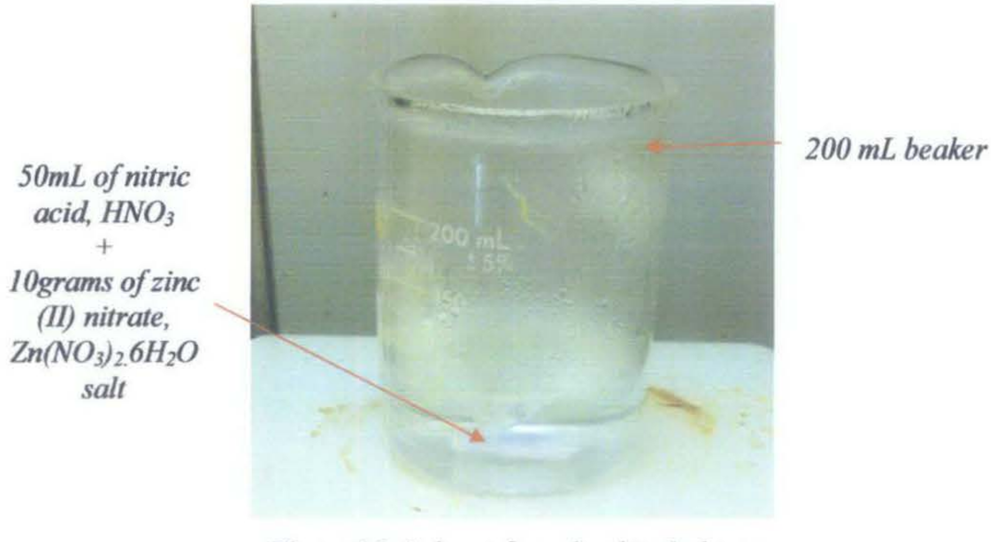


Figure 16: Solvent for sol-gel technique

This solution was stirred for 24 hours to form a homogeneous sol. For this sol gel technique, the homogenized sol solution was then heated up gradually at 40°C, 50°C, 60°C and 70°C until the gelatin formed. Refer to Figure11.

Meanwhile, the homogenized sol solution was also heated until it combusted for at 110°C for sol gel technique. The heating process is stopped immediately when the combustion start occur. This is the difference of the preparation using self combustion technique and sol gel technique. Refer to Figure17.

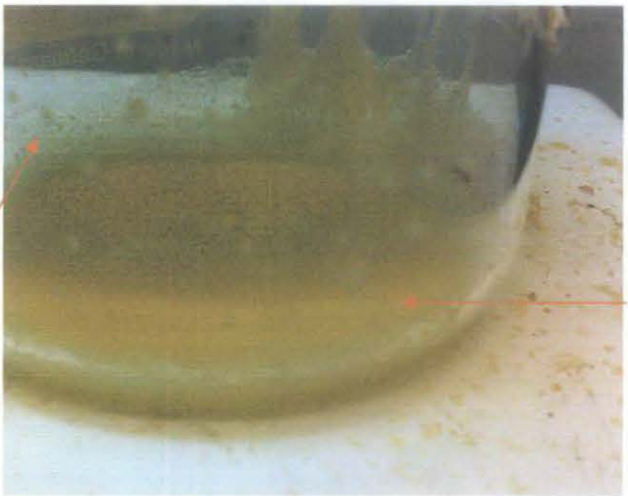


Figure 17: Sol-gel sample

The samples were dried in an oven at 110 °C for 24 hours. Refer to Figure 9. After that, the sample was crushed for 2 hours and stored in the storage tube. Refer to Figure 14.

After crushing process, the as-prepared samples were annealed at 250°C and 350°C for 2 hour. The samples were divided into three for the use of pre-sinter process at these 2 temperatures. The purpose is to compare which annealing temperature will give the good result for this ZnO synthesis. Refer to Figure 15.

4.2 Characterization of Zinc Oxide

This section aims to have the result of ZnO synthesis based on these 3 methods of characterization process; XRD, FESEM and EDX.

4.2.1 X-Ray Diffraction (XRD) Results

4.2.1.1 Standard card of zinc oxide

The result gathered from the XRD is matched with the standard card of zinc oxide. Refer to Table 1.

Table 1: Standard Card of Zinc Oxide

SAMPLE	STANDARD CARD
ZnO SC(250^{0}C)	SS-NNNN 89-0511
ZnO SC(350^{0}C)	SS-NNNN 89-1397
ZnO SG(250^{0}C)	SS-NNNN 36-1451
ZnO SG(350^{0}C)	SS-NNNN 89-1397

By applying Scherer equation [18, 19] which is:

$$D = 0.89\lambda /(\beta \cos \theta)$$

where: λ is the X-ray wavelength, θ is the Bragg diffraction angle, and β is the peak width at half-maximum. The average crystallite size of the particles can be calculated.

4.2.1.2 Self Combustion technique samples

4.2.1.2.1 ZnOSC250 sample

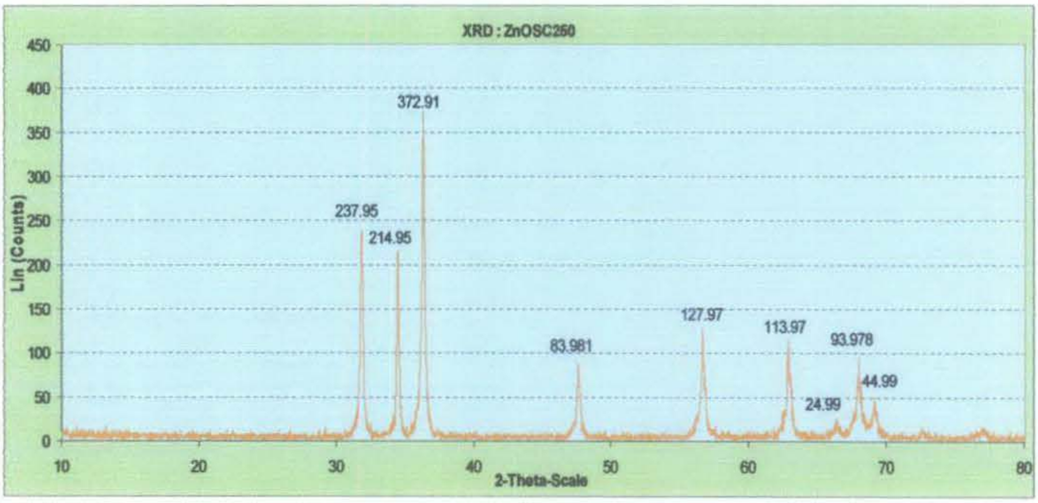


Figure 18: XRD result for ZnOSC250 sample.

4.2.1.2.2 ZnOSC350 sample

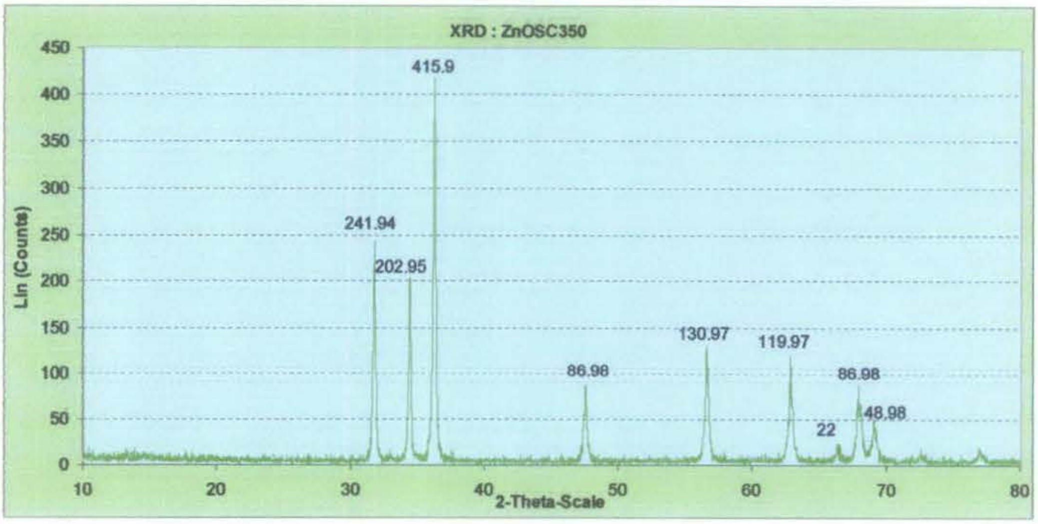


Figure 19: XRD result for ZnOSC350 sample

4.2.1.3 Sol Gel technique samples

4.2.1.3.1 ZnOSG250 sample

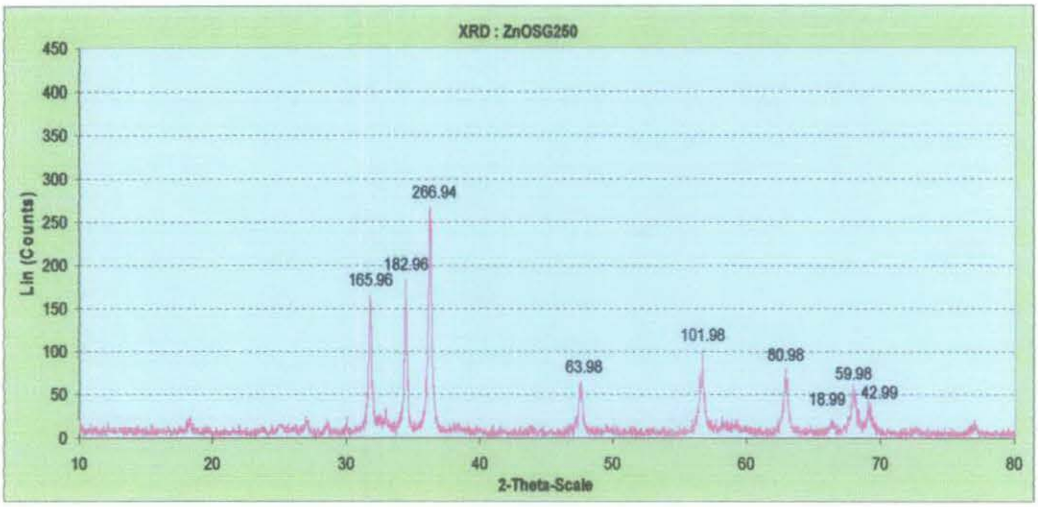


Figure 20: XRD result for ZnOSG250 sample

4.2.1.3.2 ZnOSG350 sample

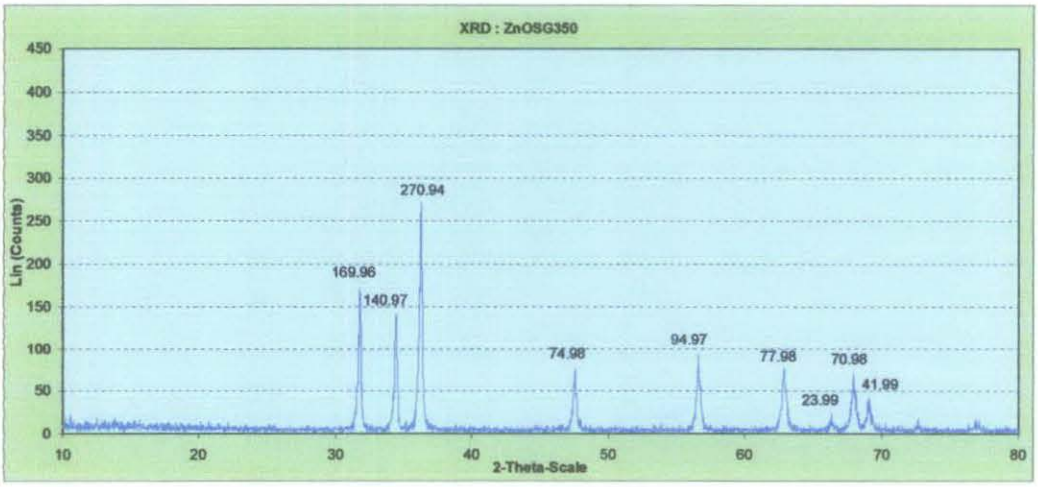


Figure 21: XRD result for ZnOSG350 sample

4.2.1.4 Analysis of XRD results

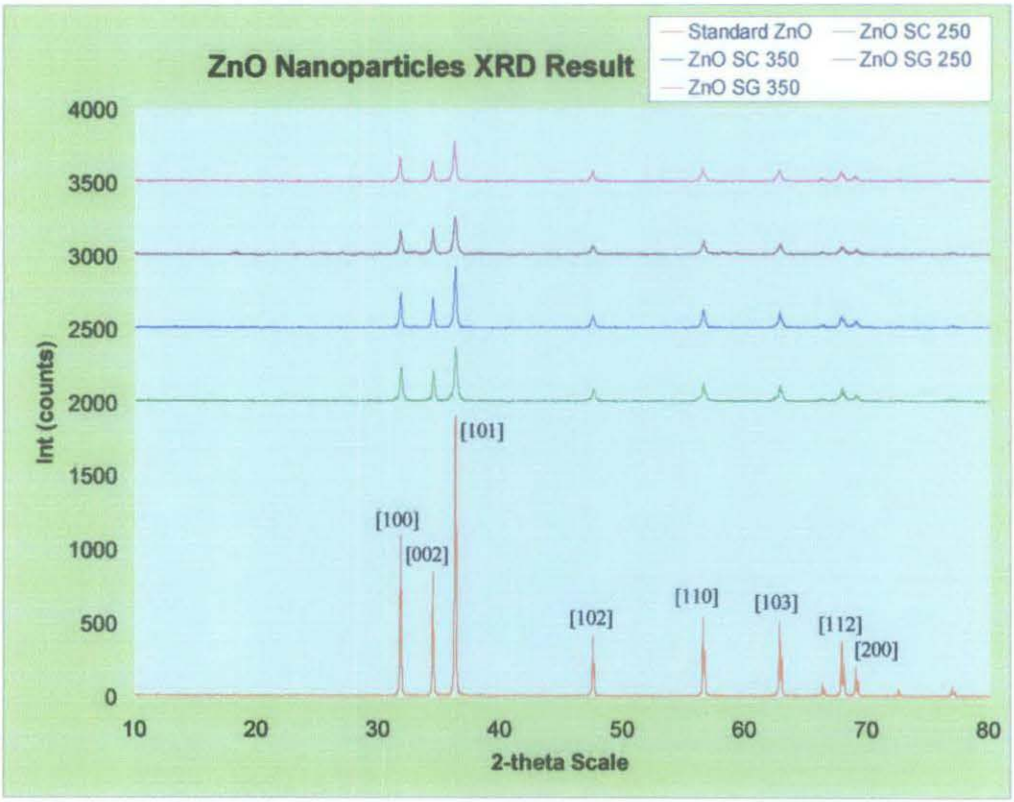


Figure 22: ZnO sample peak is following the plane of ZnO standard card.

The calculations of the crystallite size of all samples were done by using Scherer's equation, **$D = 0.9\lambda / (\beta \cos \theta)$**

For ZnO SC250

$\lambda = 1.5406$ m

$\beta = 0.004014$ rad

$\theta = 18.146$ rad

$D = (0.9(1.5406) / (0.004014 \times \cos (18.146)))/10 =$ *45.296 nm*

For ZnO SC350

$\lambda = 1.5406$ m

$\beta = 0.003752$ rad

$\theta = 18.1225$ rad

$D = (0.9(1.5406) / (0.003752 \times \cos (18.1225)))/10 =$ *45.456 nm*

For ZnO SG250

$\lambda = 1.5406$ m

$\beta = 0.004852$ rad

$\theta = 18.124$ rad

$D = (0.9(1.5406) / (0.004852 \times \cos(18.124)))/10 =$ *38.197 nm*

For ZnO SG350

$\lambda = 1.5406$ m

$\beta = 0.004276$ rad

$\theta = 18.146$ rad

$D = (0.9(1.5406) / (0.004276 \times \cos(18.146)))/10 =$ *44.216 nm*

For ZnO Standard

$\lambda = 1.5406$ m

$\beta = 0.001972$ rad

$\theta = 18.1255$rad

$D = (0.9(1.5406) / (0.001972 \times \cos(18.1255)))/10 =$ *93.847 nm*

The calculated crystallite size is shown in Table 2 and result on the nano size. The side measurement of crystallite shows that the samples were wurtzite structure. Increasing on intensity will decrease the FWHM but increasing on crystallite size and d-spacing.

Table 2: XRD results details.

X-Ray Diffraction								
Technique	Samples ID	Intensity (Counts)	FWHM	Crystallite Size (nm)	d-spacing (Å)	a	b	c
SC	ZnO SC 250	364.000	0.230	45.296	2.473	3.249	3.249	5.205
	ZnO SC 350	405.000	0.215	49.456	2.476	3.253	3.253	5.213
SG	ZnO SG 250	262.000	0.278	38.197	2.476	3.250	3.250	5.207
	ZnO SG 350	265.000	0.245	44.216	2.479	3.253	3.253	5.213
	Standard	1890.000	0.113	93.847	2.476	3.250	3.250	5.207

4.2.2 Field Emission Scanning Electron Microscope (SEM) results

4.2.2.1 Self Combustion technique sample

4.2.2.1.1 ZnO SC250 morphology

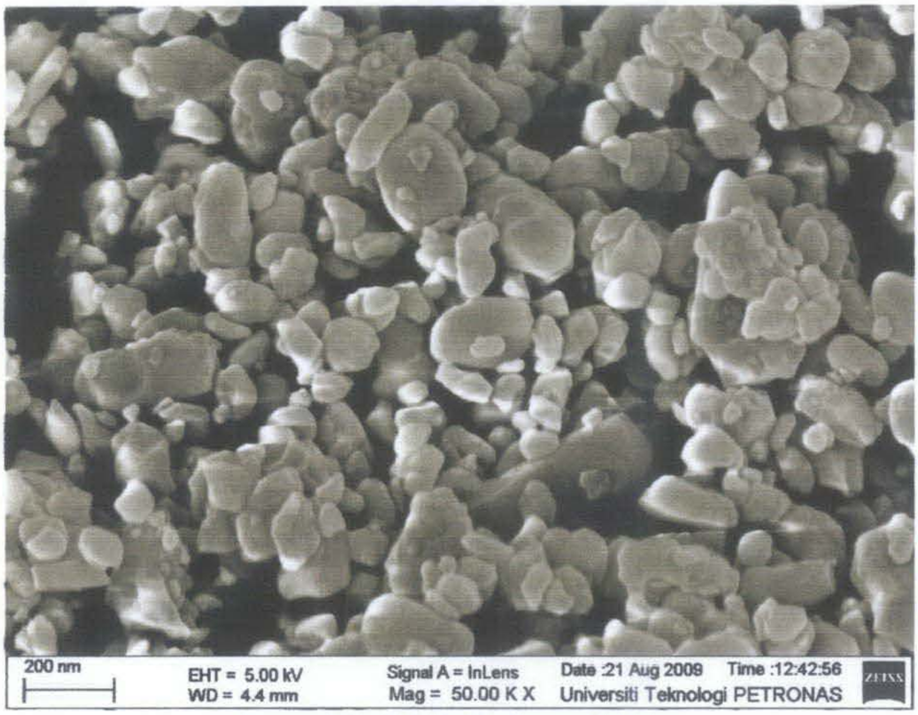


Figure 23: ZnO SC250 morphology

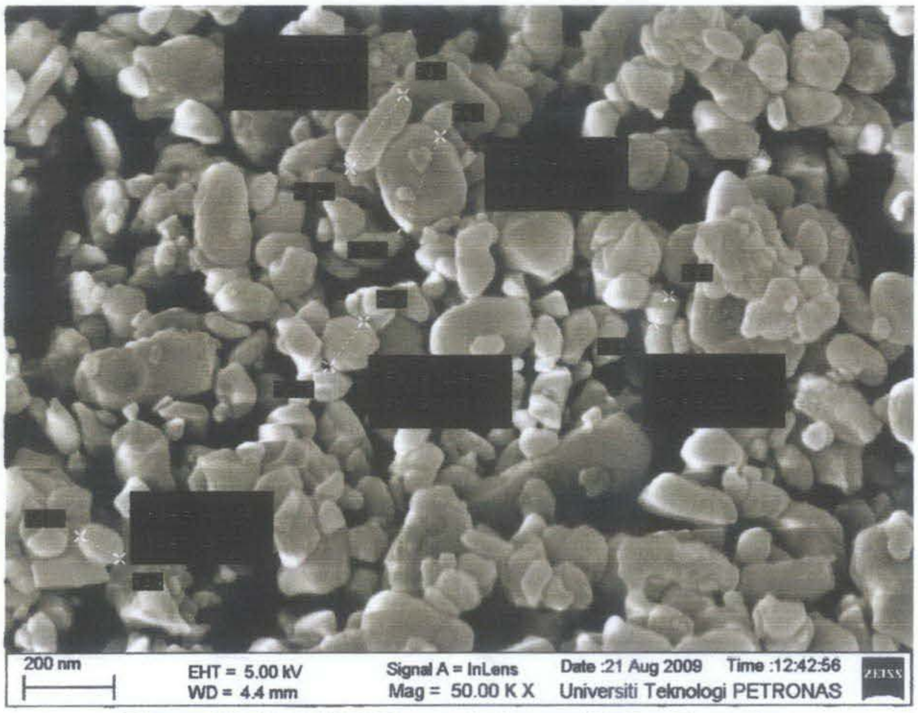


Figure 24: ZnO SC250 morphology with the dimension

4.2.2.1.2 ZnO SC250 morphology

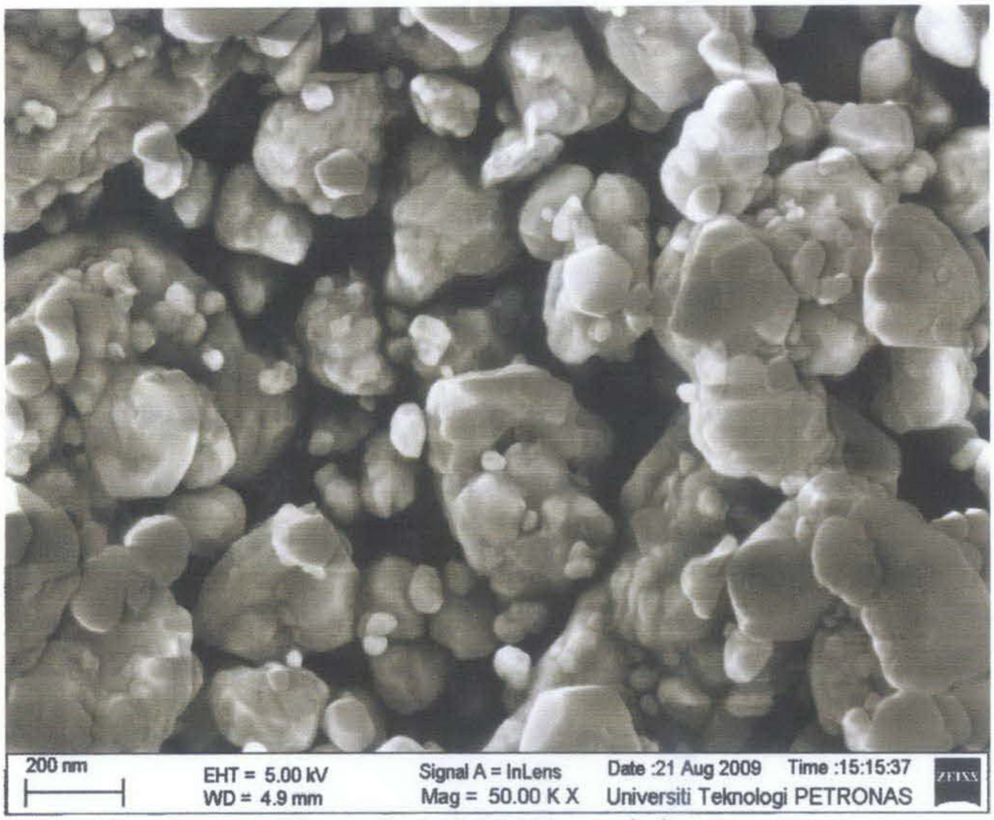


Figure 25: ZnO SC350 morphology

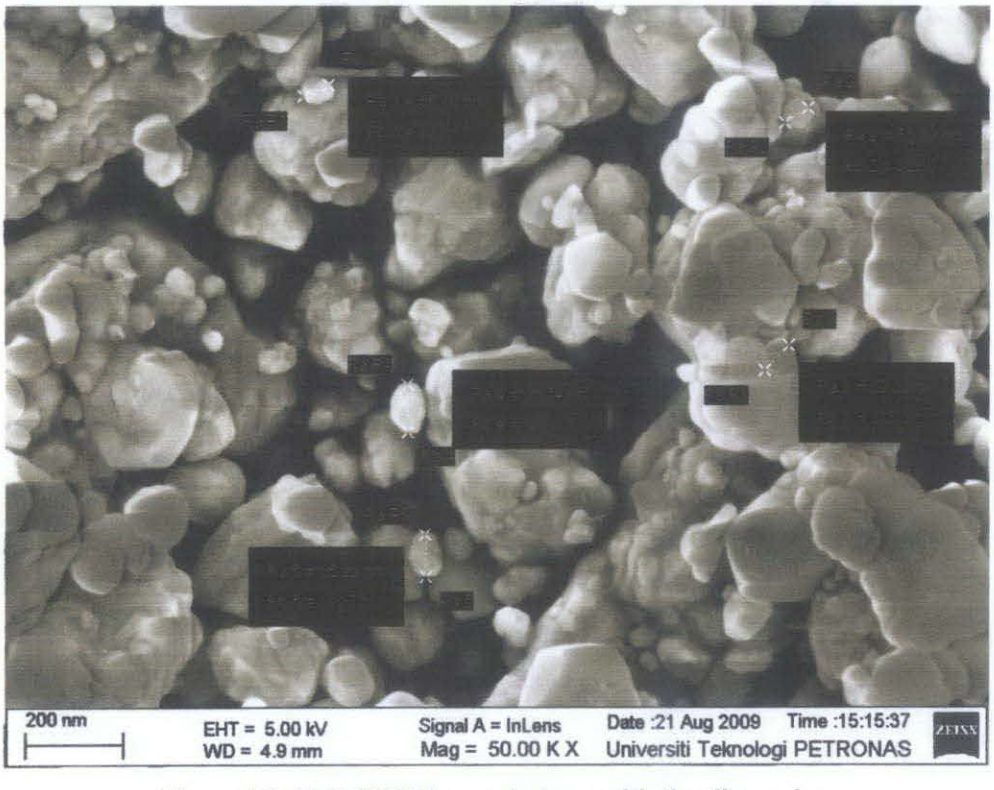


Figure 26: ZnO SC350 morphology with the dimension

4.2.2.2 Sol Gel technique sample

4.2.2.2.1 ZnO SG250 morphology

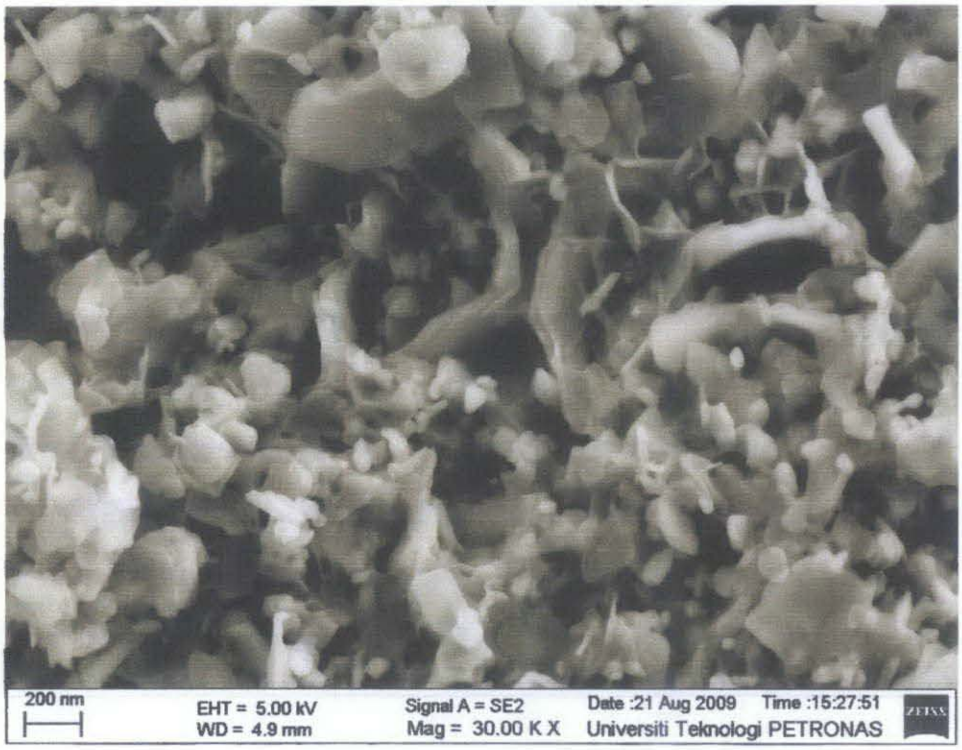


Figure 27: ZnO SG250 morphology

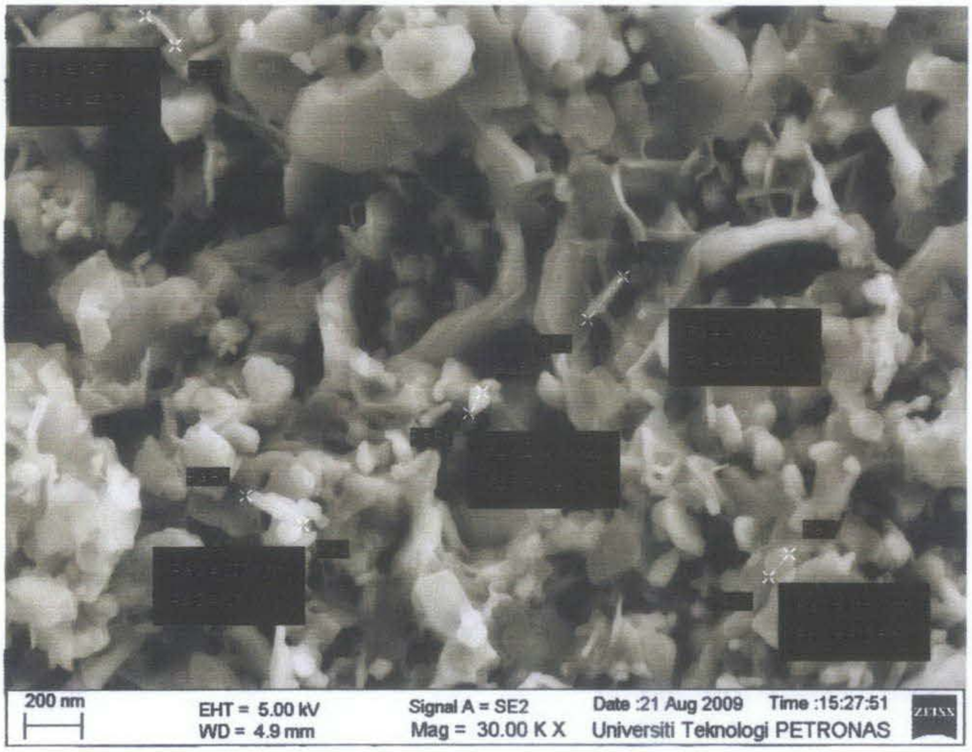


Figure 28: ZnO SG250 morphology with the dimension

4.2.2.2.2 ZnO SG350 morphology

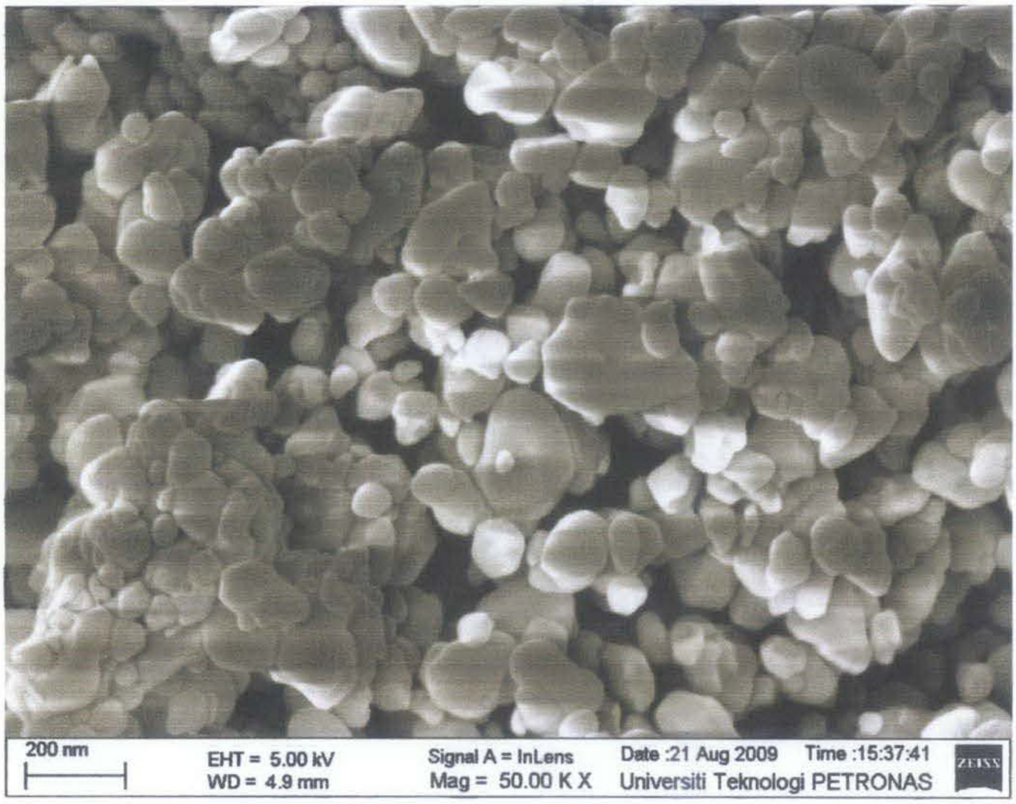


Figure 29: ZnO SG350 morphology

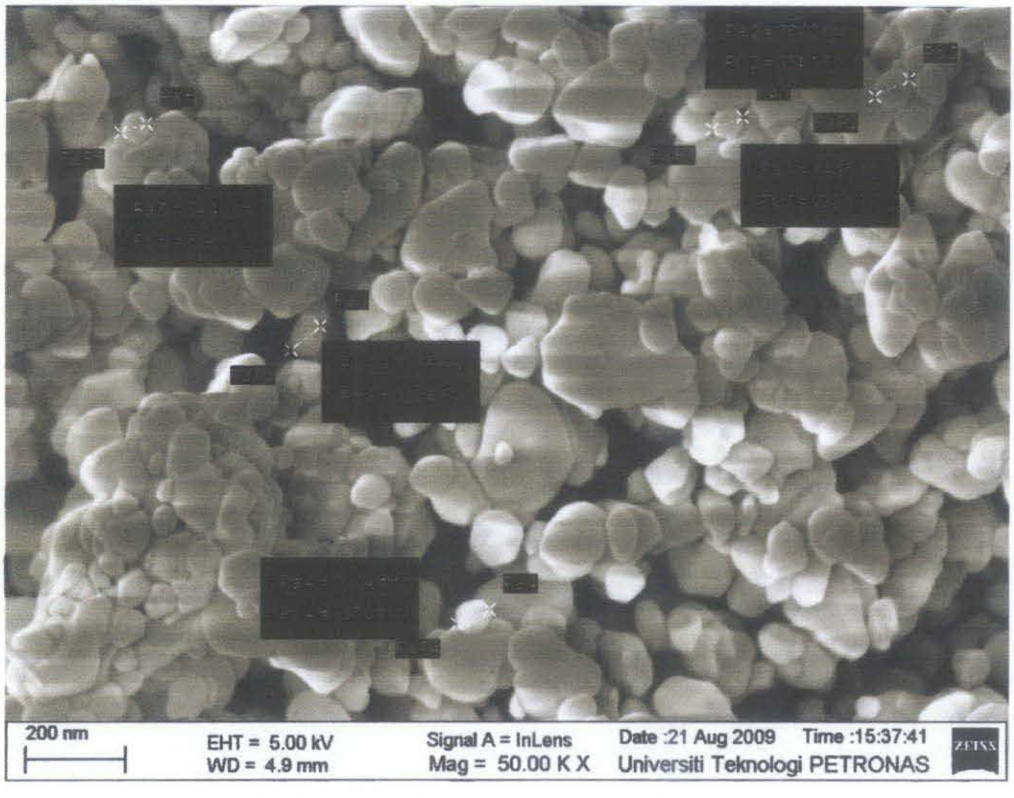


Figure 30: ZnO SG350 morphology with the dimension

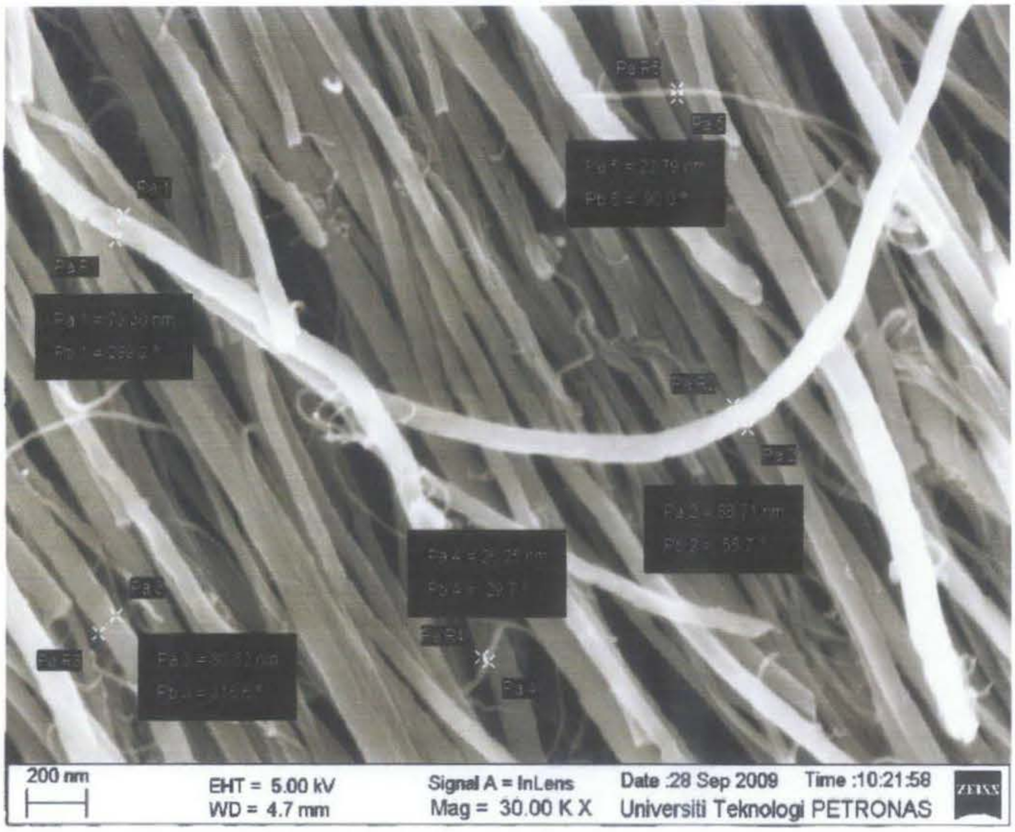


Figure 31: CNTs morphology with dimension

4.2.3 Energy Dispersive X-Ray (EDX) Results

This technique is used in conjunction with SEM and this technique used for the elemental analysis or chemical characterization of a sample. By using periodic table, a weight of zinc is 80.34% [20] and oxygen is 19.65% [21]. The atomic percentage for ZnO should be 50% of Zinc (Zn) and 50% of Oxide (O). For ZnO SC250, the weight percentage for Zn is 79.84% and for O is 20.16% while the atomic percentage is 49.22% and 50.78% respectively. For ZnO SC350, the weight percentage for Zn is 76.41% and for O is 23.59% while the atomic percentage is 49.21% and 50.78% respectively. For ZnO SG250, the weight percentage for Zn is 80.74% and for O is 19.26% while the atomic percentage is 50.64% and 49.36% respectively. For ZnO SG350, the weight percentage for Zn is 78.72% and for O is 21.28% while the atomic percentage is 47.51% and 52.49% respectively. All the samples results satisfy these theoretical values. Refer to Figure 32 for the diagram of molecule weight percentage and atomic percentage. Refer to Table 3 for the EDX results.

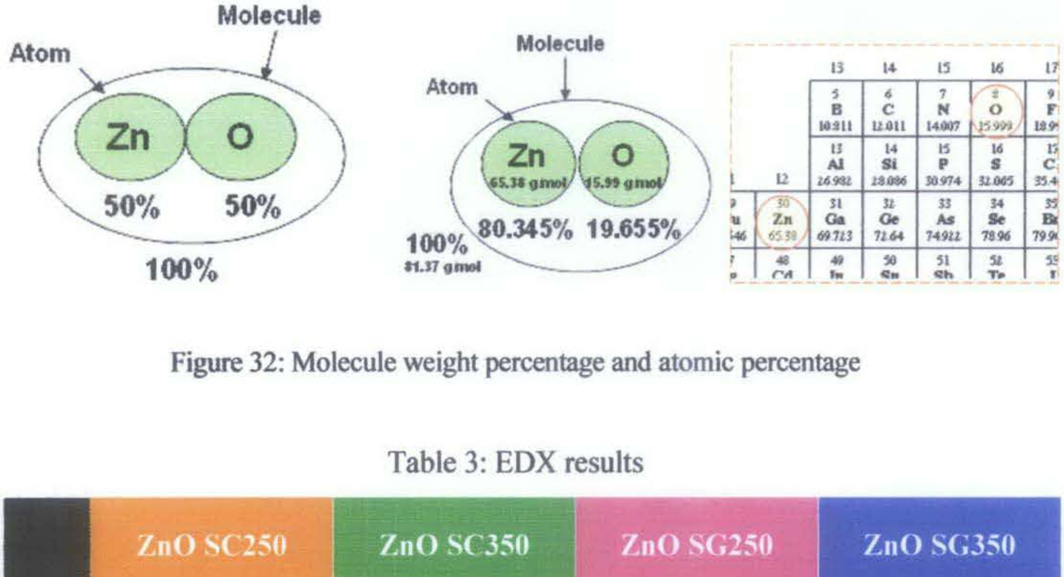


Figure 32: Molecule weight percentage and atomic percentage

Table 3: EDX results

	ZnO SC250		ZnO SC350		ZnO SG250		ZnO SG350	
	Weight %	Atomic	Weight %	Atomic	Weight %	Atomic	Weight %	Atomic
O	20.16	50.78	23.59	50.79	19.26	49.36	21.28	52.49
Zn	79.84	49.22	76.41	49.21	80.74	50.64	78.72	47.51

4.3 EM Detector Construction/Synthesis

4.3.1 Synthesis of detector

PVA composite was synthesized and used as a holder for the samples (ZnO SC250, ZnO SC350, ZnO SG250 and ZnO SG350) that have been synthesized earlier. This PVA composite filled with ZnO and CNTs is the prototype for EM detector. There are six type of detector was prepared. Refer to the Figure 32 for the apparatus used for the synthesis of the detectors. Refer Table 4 for the details of the detector synthesis.

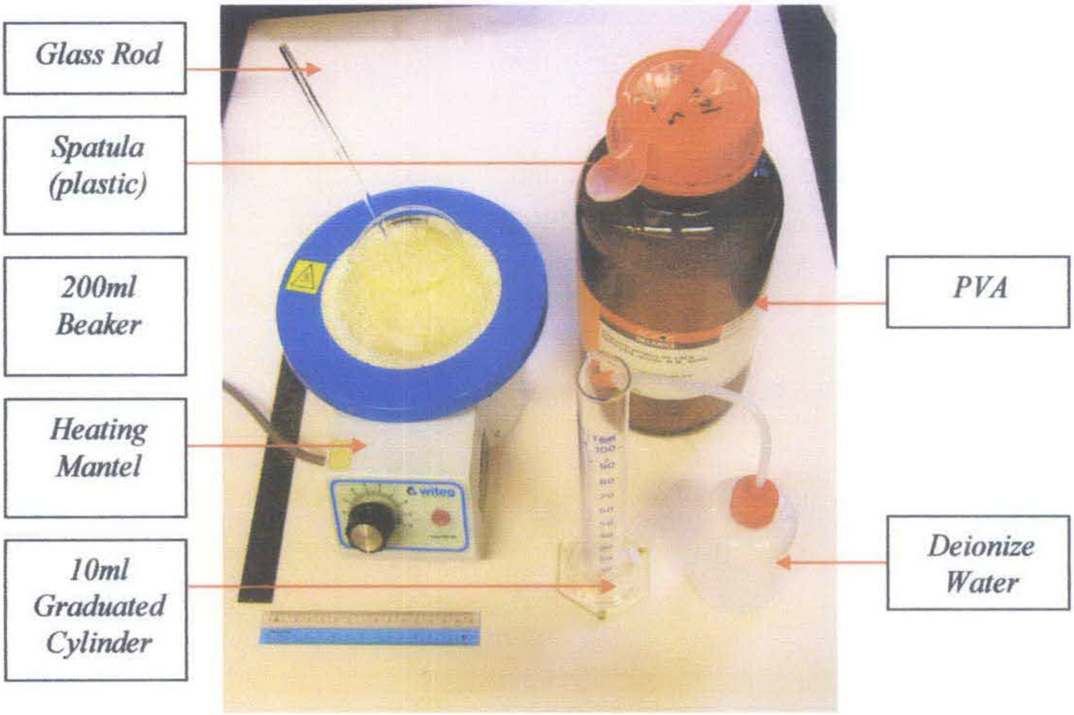


Figure 32: Apparatus needed to synthesis the PVA composite.

4.3.1.1 PVA Detector Without additive

With the ratio of 66mL deionize water to 3.6 gram lump of PVA, the amount was scaled down to 22.2mL of deionize water to 1.2 gram lump of PVA and mix it in a 250mL beaker. The mixture is going to dissolve by using the heating mantle at temperature around 60°C. After that, the dissolve mixture is shaped in a specified mould and dried out for 12 hours under the spotlight. Then, the PVA is shaped into the specified dimension, 60mm x 20mm square. The silver paste is used to connect the copper wire to the detector. Refer to Figure 33 for the preparation of PVA detector.

4.3.1.2 PVA detector + Zinc Oxide as additive

The different for this type of detector preparation from the previous detector is the specific amount of ZnO was mixed to the dissolve mixture of PVA with deionize water. The amount of ZnO is used from the self-combustion and sol-gel sample is at starting value, 0.1 mg of the sample was used as detector additive/filler. Refer to Figure 15 for the preparation of ZnO (from self-combustion and sol gel techniques) and ZnO. Refer Figure 33 for the preparation of detector. Refer Table 4 for the details of ZnO samples used.

4.3.1.3 PVA detector + Zinc Oxide and CNTs as additives

For this type of detector, the preparation quite similar with the PVA + Zinc Oxide detector but the CNTs sample needs to be prepared (crushing for 2 hours). The CNTs used is about 0.1mg and need to be crush together with the ZnO sample. 0.1mg of ZnOSC250 sample was decided to be added together with the CNTs as additive/filler to the PVA composite because the result from characterization of ZnOSC250 was most satisfied. Refer to Figure 33 for the preparation of ZnO-CNTs PVA composite detector. Refer to Table 4 for the details of ZnO and CNTs sample used.

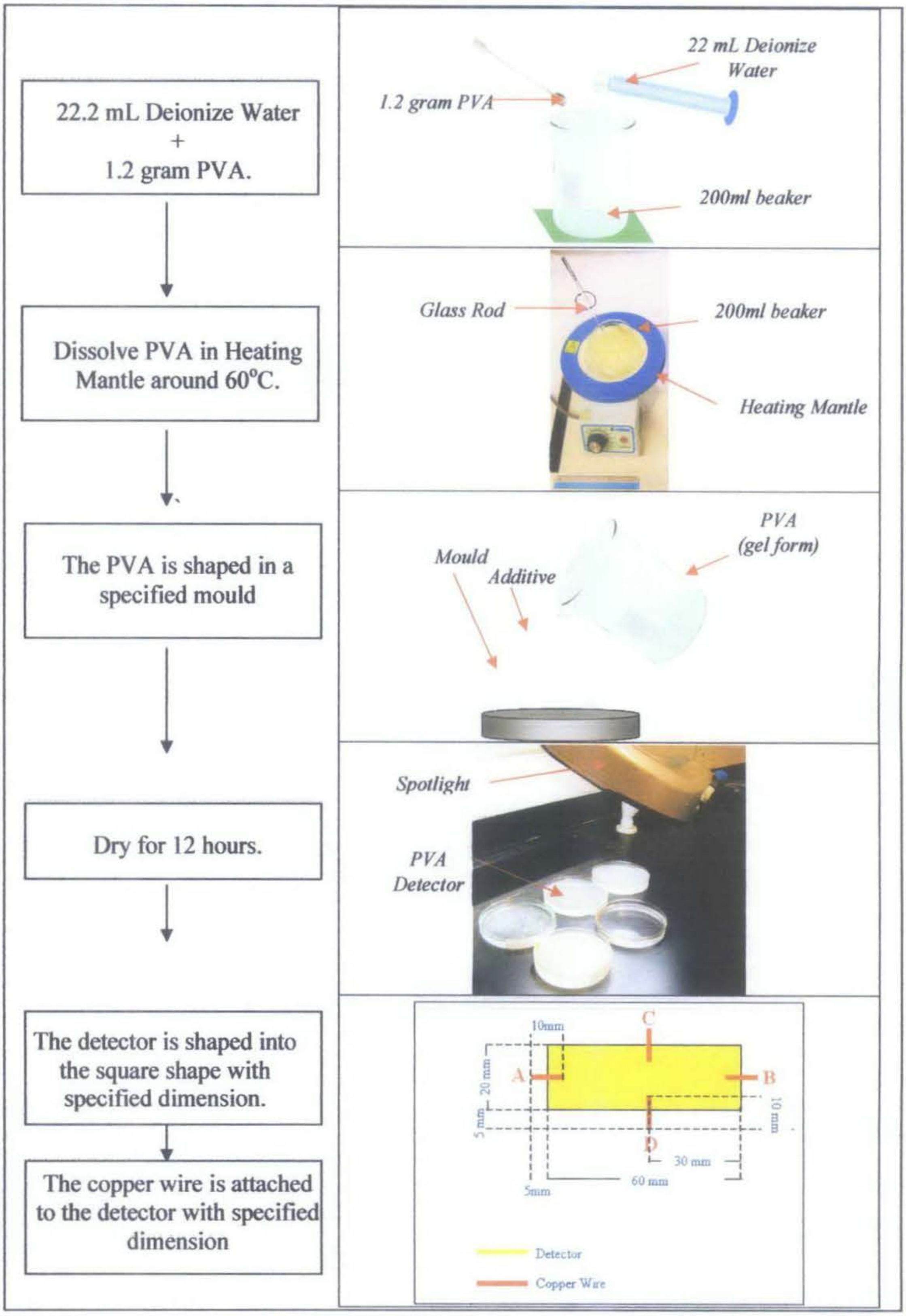


Figure 33: PVA composite filled additive synthesis flow

Table 4: Details of EM detector synthesis

No.	Detector	Preparation specification			Remark
		PVA (mg)	Deionize Water (ml)	Additive	
1	PVA	1.2 mg	22 ml	-	PVA detector
2	PVA composite filled ZnOSC250	1.2mg	22 ml	ZnOSC250 (0.1mg)	PVA + ZnO detector
3	PVA composite filled ZnOSC350	1.2 mg	22 ml	ZnOSC350 (0.1mg)	
4	PVA composite filled ZnOSG250	1.2 mg	22 ml	ZnOSG250 (0.1mg)	
5	PVA composite filled ZnOSG350	1.2 mg	22 ml	ZnOSG350 (0.1mg)	
6	PVA composite filled ZnOSC250 +CNTs	1.2 mg	22 ml	ZnOSC250 (0.1mg) + CNTs (0.1mg)	PVA + ZnO + CNTs Detector

4.4 Experiment 1

The experiment was done to detect the EM wave. Three detectors was used, the detector without additive (PVA composite only) and another two detector with additive (PVA + ZnOSC250 and PVA + ZnOSG250).

Configuration 1: Point A to B

Table 5: EM Detection Results for configuration 1 detector

	PVA		PVA + ZnO(SC)		PVA + ZnO(SG)	
TX (MHz)	PK-PK (mV)	FREQUENCY (kHz)	PK-PK (mV)	FREQUENCY (kHz)	PK-PK (mV)	FREQUENCY (kHz)
10	576	51.98	624	48.36	632	52.85
20	584	51.12	576	50.45	640	51.9
30	592	50.2	592	50.6	616	50.18
40	576	51.71	616	50.92	664	49.21
50	600	51.41	632	51.23	712	49.02
60	568	50	624	51.87	648	48.92
70	616	50.18	584	52.41	672	49.67
80	600	51.33	736	51.55	672	49.5

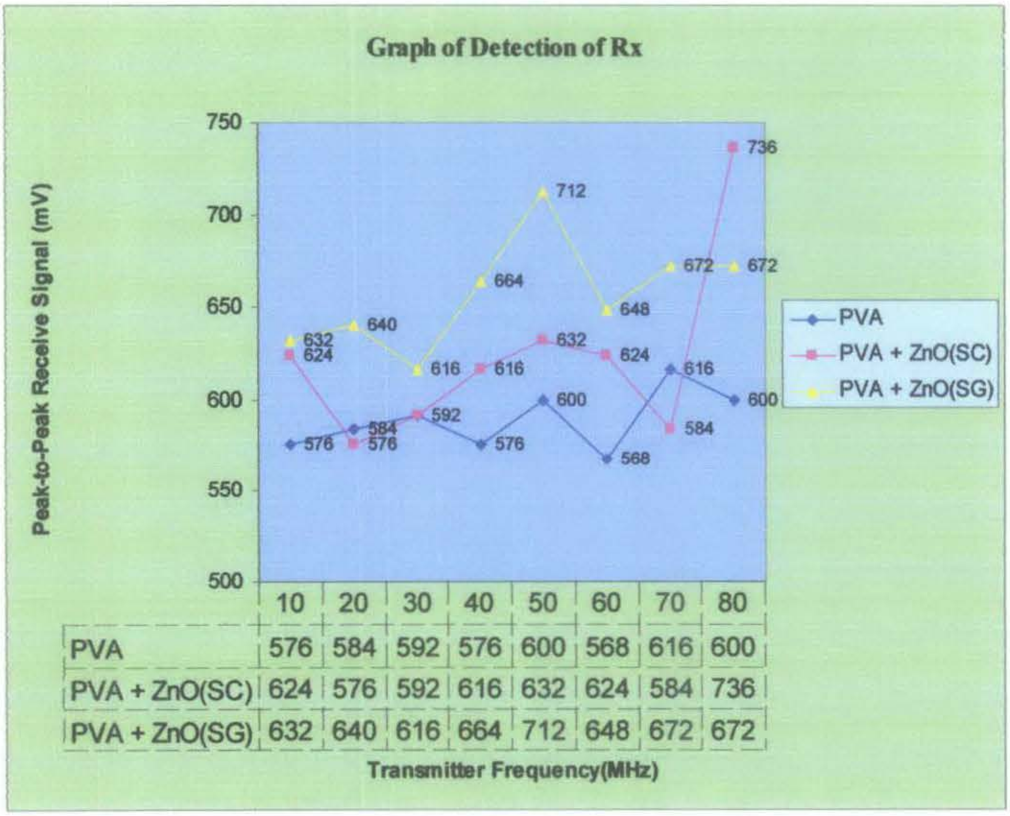


Figure 34: Detection result for configuration 1 detector

From this result, the effect of existence of ZnO in the PVA detector can detect better signal of EM waves generate from the transmitter. Based on 50MHz, detector contained ZnOSG250 sample was found that give the better EM detection. The percentage of increment was calculated (based on 50 MHz) as shown below, with the PVA detector is to be a set point.

For PVA + ZnOSC250

% Increment = [(632-600) / 600] x 100%

= *5.33%*

For PVA + ZnOSG250

% Increment = [(712-600) / 600] x 100%

= *18.67%*

The calculation show that the PVA composite filled with ZnOSC250 detector increase the EM detection performance about 5.33% while for PVA filled with ZnOSG250 give 18.67% enhancement. The ZnOSG250 have a better performance on EM detection.

Configuration 2: Point C to D

Table 6: EM Detection Results for configuration 2 detector

	PVA		PVA + ZnO(SC)		PVA + ZnO(SG)	
TX (MHz)	PK-PK (mV)	FREQUENCY (kHz)	PK-PK (mV)	FREQUENCY (kHz)	PK-PK (mV)	FREQUENCY (kHz)
10	536	51.41	600	50.51	624	48.5
20	536	51.23	624	50.16	608	50
30	584	53.76	640	50.99	608	50.45
40	584	50.81	664	51.49	696	49.13
50	520	49.6	648	50.2	648	50.16
60	528	49.12	632	51.44	616	50.35
70	544	51.76	672	49.7	680	50.25
80	552	50.61	696	48.92	632	49.31

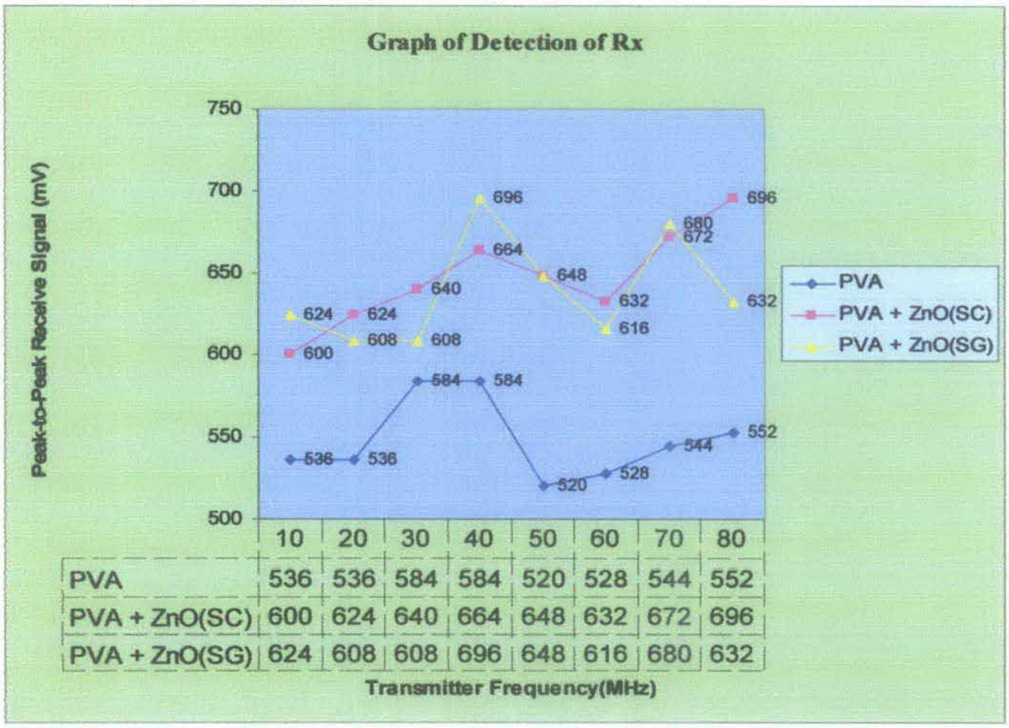


Figure 35: Detection result for configuration 2 detector

From this result, the effect of existence of ZnO in the PVA detector can detect better signal of EM waves generate from the transmitter. Based on 50MHz, the detector contained ZnOSG250 sample was found that give the better EM detection. The percentage of increment was calculated (based on 40 MHz) as shown below, with the PVA detector is to be a set point.

For PVA + ZnOSC250

% Increment = [(664-584) / 584] x 100%

= *13.70%*

For PVA + ZnOSG250

% Increment = [(696-584) / 584] x 100%

= *19.17%*

The calculation show that the PVA composite filled with ZnOSC250 detector increase the EM detection performance about 13.70% while for PVA filled with ZnOSG250 give 19.17% enhancement. The ZnOSG250 have a better performance on EM detection.

4.4.1 Analysis of results

From the graph (Figure 34 and Figure 35), PVA composite with the ZnO as additive to the composite detector increase the EM detection. For configuration 1, the percentage increment is about 5.33% for ZnOSG250 as additive while18.67% for ZnO SC250 as additive. For the configuration 2, the percentage increment is about 13.70% for ZnOSG250 as additive and 19.17% for ZnOSG250 as additive. The samples ZnOSG250 was used as additive to the PVA composite for both configurations give the better performance to the EM detection. Changing in configuration will affect the EM detection performance which shows the configuration 2 detectors give the better results.

4.5 Finalizing the EM detector prototype

The detector holder was designed to hold the detector to make sure the experimental work done to the detector is going to be smooth and easy to handle the detector without spoil the detector and to display the detector in a better ways. The dimension of the holder is shown in the Figure 36

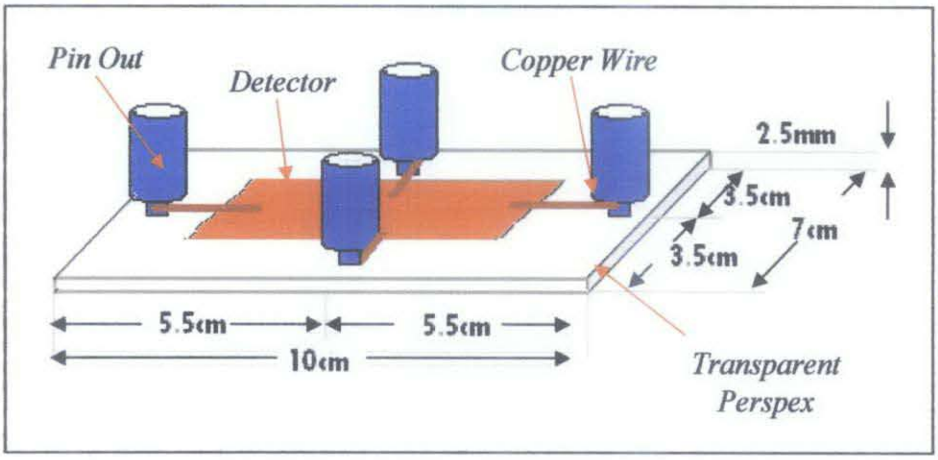
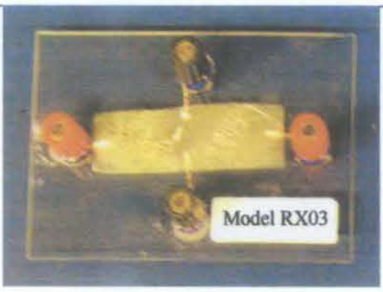
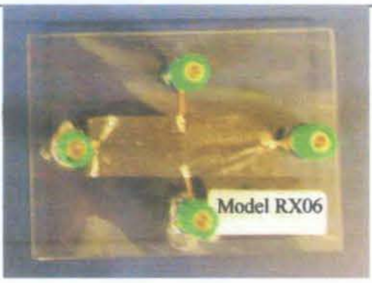


Figure 36: Detector holder dimension

Table 7: EM detector prepared

No.	Detector	Type	Figures
1	PVA	Model RX01	 Figure 37: Model RX01 EM detector

No	Detector	Type	Figures
2	ZnOSC250 + PVA	Model RX02	 Figure 38: Model RX02 EM detector
3	ZnOSC250 + PVA	Model RX03	 Figure 39: Model RX03 EM detector
4	ZnOSG250 + PVA	Model RX04	 Figure 40Model RX04 EM detector
5	ZnOSG350 + PVA	Model RX05	 Figure 41: Model RX05 EM detector
6	ZnO-CNT + PVA	Model RX06	 Figure 42: Model RX06 EM detector

4.6 Experiment 2

The experiment was started on the detector for the three types of detector, which is to Model RX01, Model RX02, and Model RX06 detector. The setup for the experiment is shown in Figure 43.

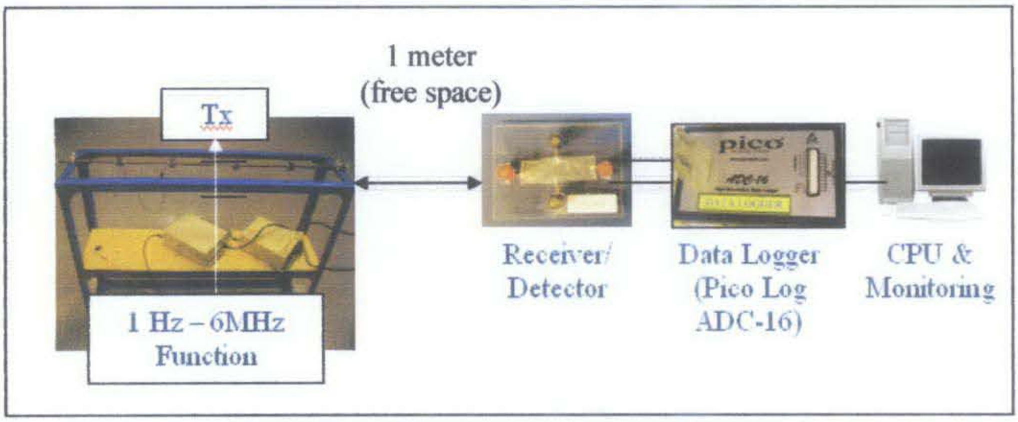


Figure 43: Experiment on EM detecting set up

The EM signal is generated by the function generator, Goodwill INSTEK GFG-8250A (1Hz to 6MHz) and being transmitted by the transmitter. This EM wave will be captured by the detector and produce induced current. This induced current will flowing through the data logger and the CPU will act as oscilloscope in a better way and display the results in induced voltage (Emf) reading by using the Pico-Log-Recorder software. The data logger (Pico Log ADC-16) was used to record the voltage induced by the detector instead of using the common oscilloscope. The Pico Log ADC-16 data logger offers high resolution (16 bits sign) and is capable of detecting signal changes as small as 40μV. It provides a PC with 8 highly accurate input channels. Pairs of channels can be used differentially to reject noise. Refer Figure 44 for the Pico Log ADC-16.

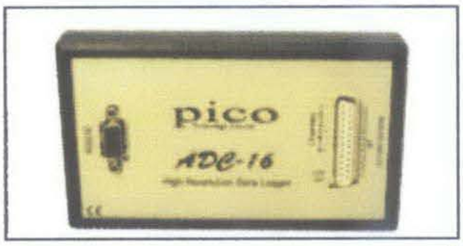


Figure 44: Data Logger (Pico Log ADC-16) [22]

Figure 45 shows the dimension of the detector (PVA composite). The dimension of the detector is being standardized to 20mm X 60mm. The copper wire is used as a contact to the detectors. The silver paste was used to contact the copper wire to this thin film detector. Point A and B were paired and same goes to the point C and D.

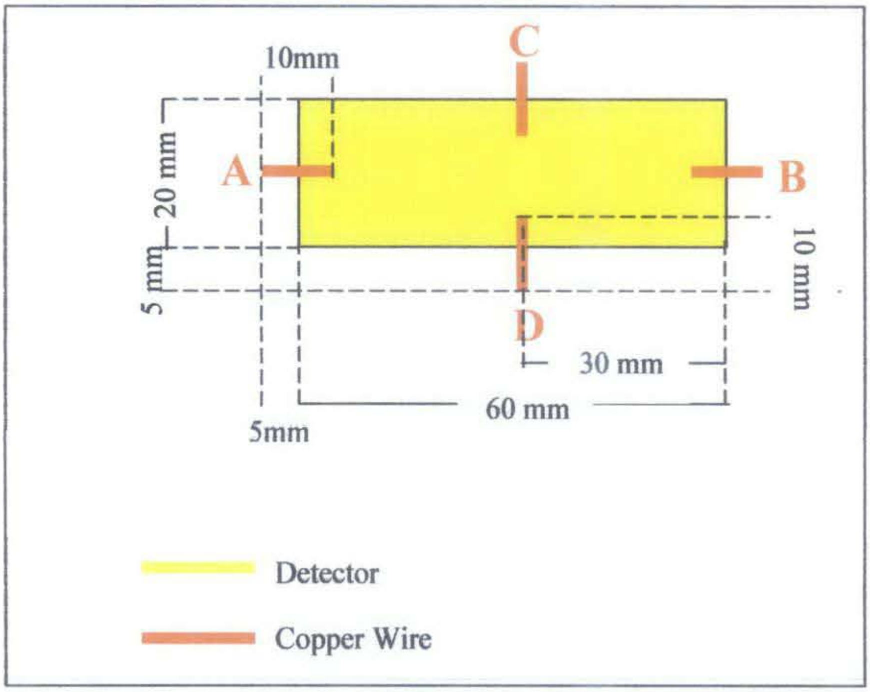


Figure 45: Detector dimension

For each detector, the reading of detection is first taken from the point A to point B (Configuration 1) and after that, from point C to point D (Configuration 2). See Figure 46 and Figure 47 for the diagram of the detector configuration for this experiment. For the Configuration 1, the distance between the copper wires is 50mm while for the Configuration 2 is 10mm which mean the distance between copper wires for the Configuration 1 is shorter than Configuration 2 .

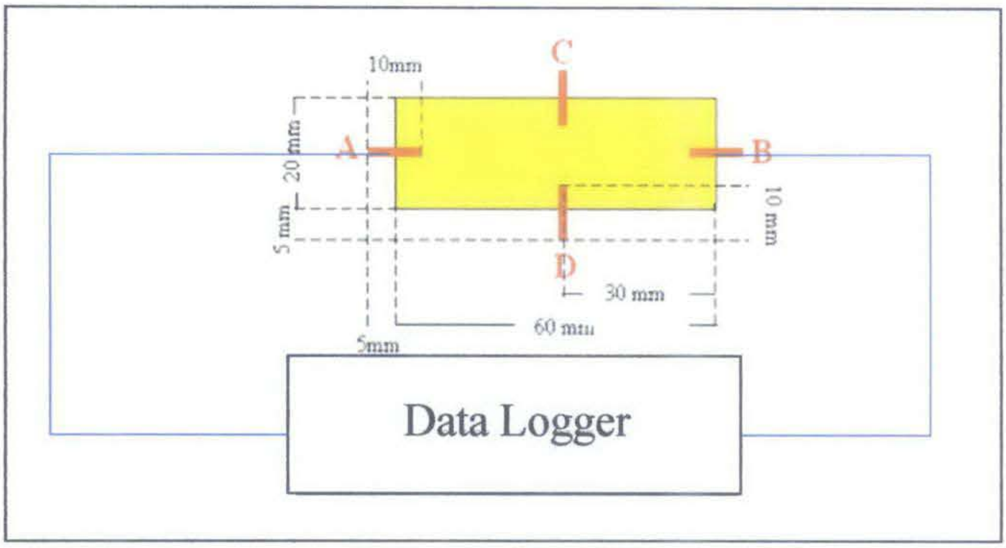


Figure 46: Detector Configuration 1

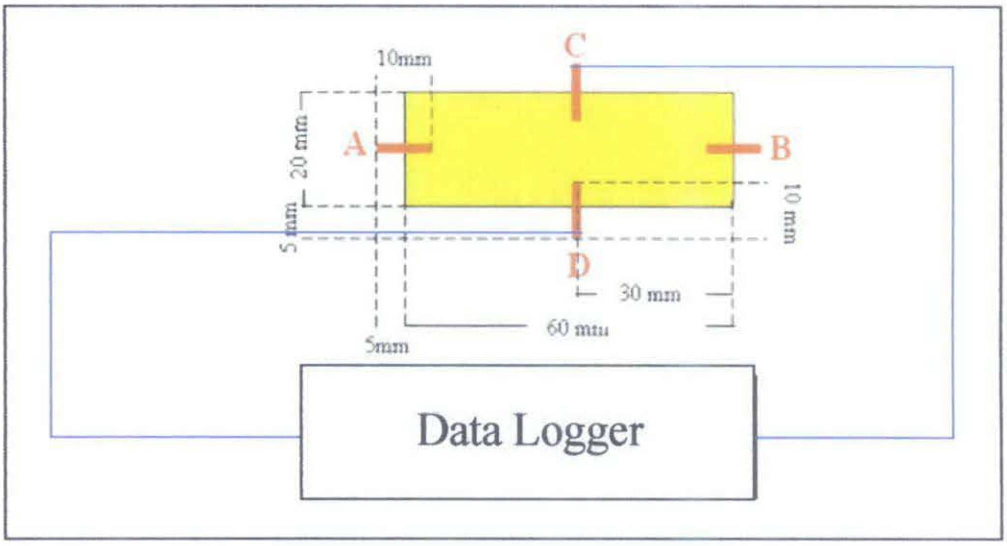


Figure 47: Detector Configuration 2.

Three detectors were decided to test, which is Model RX01, Model RX02 and Model RX06 because of the time constrain. Model RX01 is the detector without the additive which means it just an empty detector. Model RX02 is representative of detector with the ZnO as an additive. Model RX06 is the detector filled with the both ZnO and CNTs. The ZnO and CNTs as additive were used to make the better enhancement in EM detection and for initial hypothesis; CNTs can be a booster for the detector to enhance EM detection. Refer to Table 4 for the detail specification of the detector.

4.6.1 Results

Configuration 1: Point A to B

The graph was generated and shown in Figure 48, and the graph is zoomed at the where the difference of the detection of EM signal can be analyze.

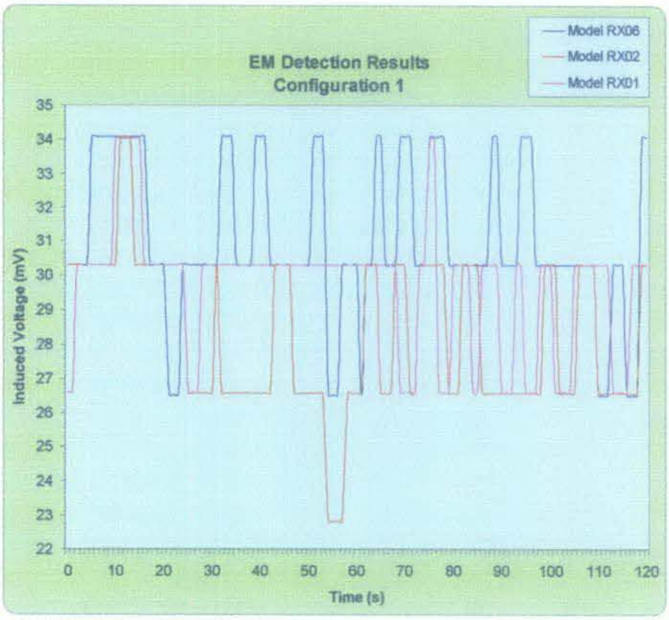
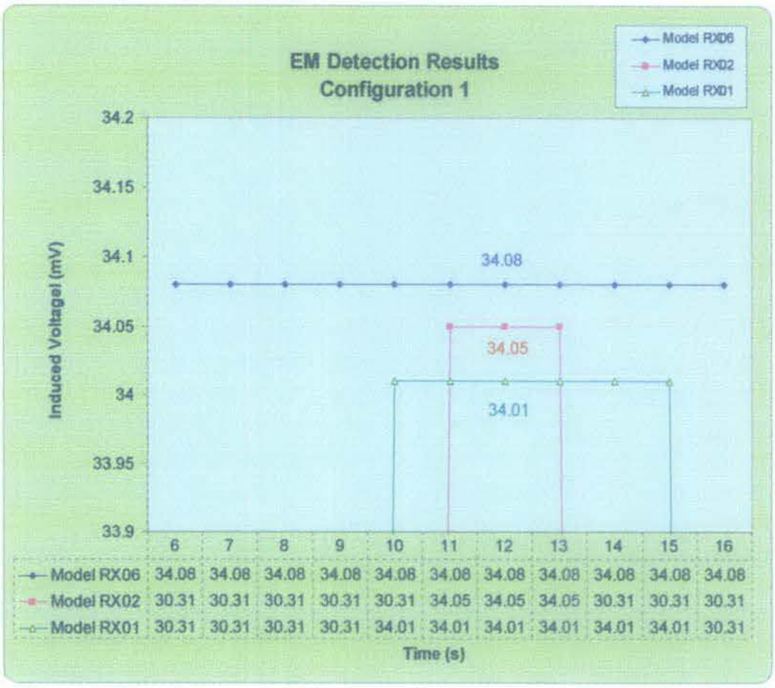


Figure 48: EM detection result (Configuration 1) graph.



	6	7	8	9	10	11	12	13	14	15	16
Model RX06	34.08	34.08	34.08	34.08	34.08	34.08	34.08	34.08	34.08	34.08	34.08
Model RX02	30.31	30.31	30.31	30.31	30.31	34.05	34.05	34.05	30.31	30.31	30.31
Model RX01	30.31	30.31	30.31	30.31	34.01	34.01	34.01	34.01	34.01	34.01	30.31

Figure 49: Zooming of EM detection result (Configuration 1) graph

Based on the graph generated and zoomed between 6th sample per second and 16th sample per second, the highest detection is result on Model RX06. Model RX02 is second higher while Model RX01 is the lowest. The calculations have been done at 12th sample per second to calculate the percentage of increment on detection. Model RX01 detection is set to be set point for the calculation.

<u>Model RX02 increment percentage</u>

(34.05 – 34.01) / 34.01 x 100% = 0.12 %

The increment percentage of detection for Model RX02 is 0.12% from Model RX01. This result is based on the detector construction ratio shown below; which is 0.1 g of ZnO, 0.1 g of CNTs and 1.2 g of PVA.

[PVA : CNTs : ZnO]
[12 : 0 : 1]

<u>Model RX06 increment percentage</u>

(34.08-34.01)/34.01 x 100% = 0.21 %

The increment percentage of detection for Model RX06 is about 2 times more than Model RX02. This result is based on the detector construction ratio shown below; which is 0.1 g of ZnO, 0.1 g of CNTs and 1.2 g of PVA.

[PVA : CNTs : ZnO]
[12 : 1 : 1]

The increment percentage of EM detection for Model RX06 is about 2 times more than Model RX02. The performance of the detection can be improved by changing the ratio of additive used such as the ratio shown below;

[PVA : CNTs : ZnO]
[12 : 2 : 2], [12 : 3 : 3], [12 : 3 : 3], …

The result for the changing in ratio for additive used is discussed in the linear extrapolation graph. Refer Figure 52 and Figure 53 for the extrapolation results.

Configuration 2: Point C to D

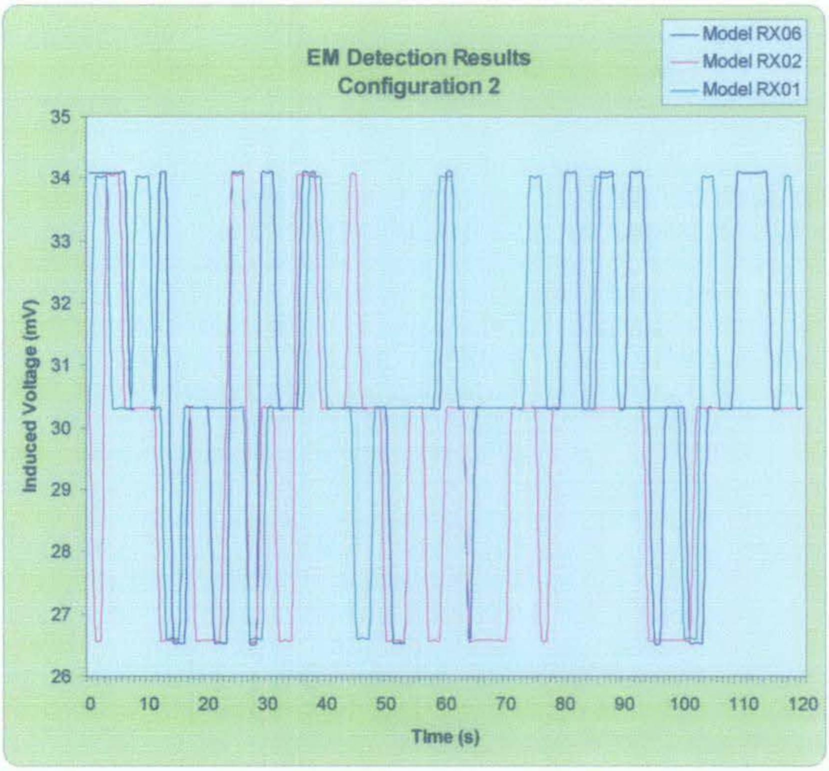
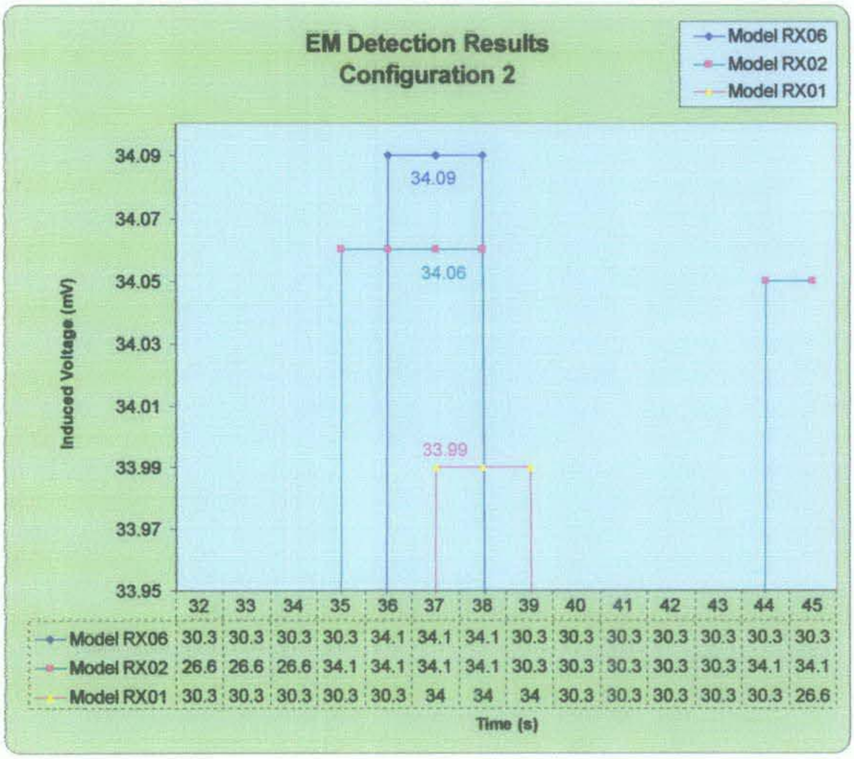


Figure 50: EM detection result (Configuration 2) graph



	32	33	34	35	36	37	38	39	40	41	42	43	44	45
Model RX06	30.3	30.3	30.3	30.3	34.1	34.1	34.1	30.3	30.3	30.3	30.3	30.3	30.3	30.3
Model RX02	26.6	26.6	26.6	34.1	34.1	34.1	34.1	30.3	30.3	30.3	30.3	30.3	34.1	34.1
Model RX01	30.3	30.3	30.3	30.3	30.3	34	34	34	30.3	30.3	30.3	30.3	30.3	26.6

Figure 51: Zooming of EM detection result (Configuration 2) graph

Based on the graph generated and zoomed between 32nd sample per second and 45th sample per second, the highest detection is result on Model RX06. Model RX02 is second higher while Model RX01 is the lowest. The calculation was done at the 37th sample per second to calculate the percentage of increment on detection. Model RX01 detection is set to be set point for the calculation.

<u>Model RX02 increment percentage</u>

(34.06 – 33.99) / 33.99 x 100% = 0.21 %

The increment percentage of detection for Model RX02 is 0.21% from Model RX01. This result is based on the detector construction ratio shown below; which is 0.1 g of ZnO, 0.1 g of CNTs and 1.2 g of PVA.

[PVA : CNTs : ZnO]
[12 : 0 : 1]

<u>Model RX06 increment percentage</u>

(34.09-33.99) / 33.99 x 100% = 0.29 %

The increment percentage of detection for Model RX06 is about 0.29%. This result is based on the detector construction ratio shown below; which is 0.1 g of ZnO, 0.1 g of CNTs and 1.2 g of PVA.

[PVA : CNTs : ZnO]
[12 : 1 : 1]

The performance of the detection can be improved by changing the ratio of additive used such as the ratio shown below;

[PVA : CNTs : ZnO]
[12 : 2 : 2], [12 : 3 : 3], [12 : 3 : 3], ...

The result for the changing in ratio for additive used is discussed in the linear extrapolation graph.

4.6.2 Analysis of the results

From the result, changing in Configuration 1 to Configuration 2 will effect the EM signal detection. For configuration 1 detector, the existence of ZnO nanoparticles in the PVA can enhances the detection by 0.12% while the existence of the CNTs will boost up the detection to the 0.21% and can be increase up to 70% by changing the ratio of additive used. For configuration 2 detector, the existence of ZnO nanoparticles in the PVA can enhances the EM detection by 0.21% while the existence of CNTs as booster will boost up the detection to the 0.29%. The result from configuration 2 is better than configuration 1 because for the configuration 2, the way that electron move from point C to D is shorter than configuration 1 (point A to B). By doing linear extrapolation to the configuration 2 results, EM detection can be increase up to 70 %. Linear extrapolation for the result shows that the increment of additive amount to the detector will increase the EM detection up to 70%. Refer Figure 52 and Figure 53 for the extrapolation results.

Based on the graph in Figure 53 generated by Figure 52, by adding the amount of the ZnO and CNTs as additive to the detector, the percent increment will be higher until some relevant point. The detection can reach up to 70% increment when the amount of ZnO and CNTs is increase to 0.5mg. The existence of the ZnO and CNT as additive to the EM detector can enhance the EM detection of the EM detector up to 70% increment.

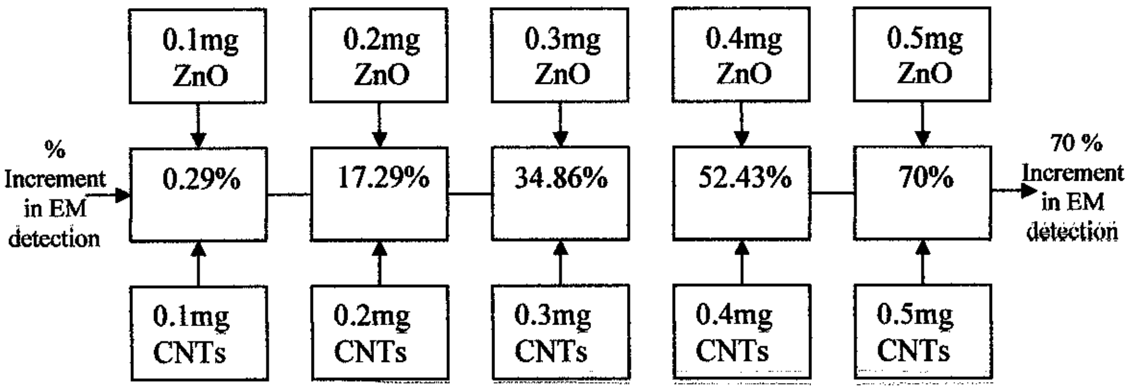


Figure 52: Extrapolation Block Diagram

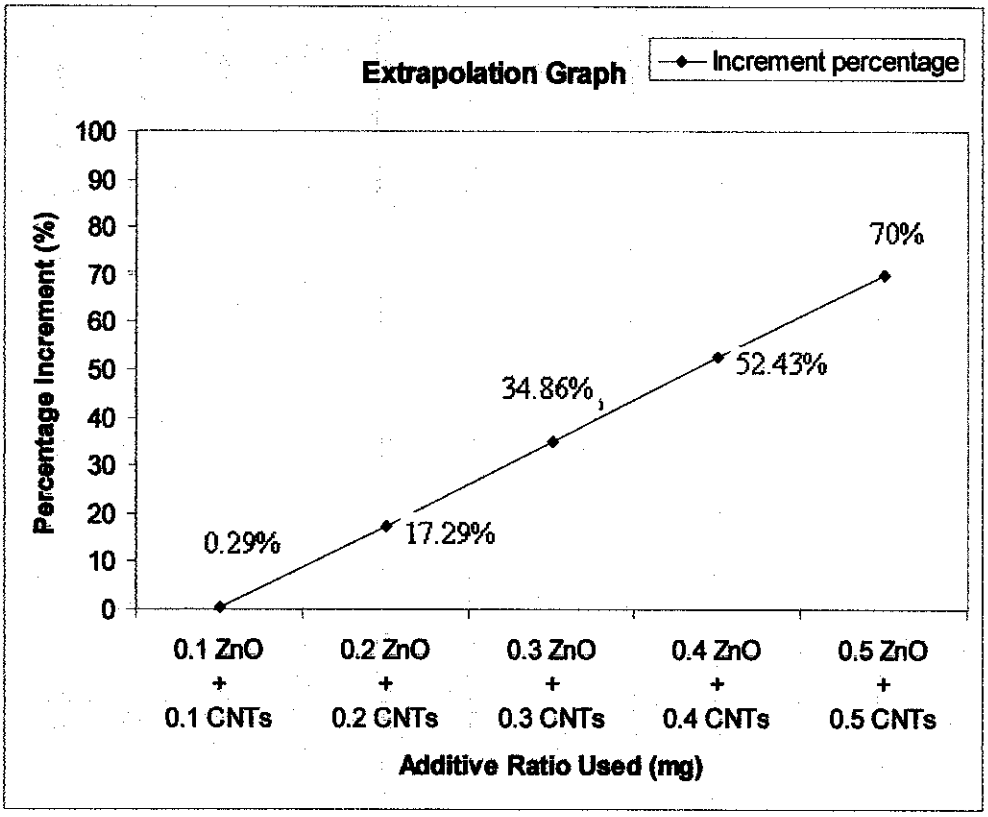


Figure 53: Linear Extrapolation Graph

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In this project, ZnO nanoparticles were successfully synthesized by sol-gel and self-combustion techniques. Based on the characterization result, it is shown that with increasing of an annealing temperature, the crystallite size will increase too. From the FESEM, the structure and the dimension of the ZnO can be determined. The sample ZnO SG 350 shows the rounded structures and the dimension of all the samples are in the range of nano meter sized. The XRD result shows that the plane [100], [002], [101], [102], [110], [103], [112], and [200] for the sample is matched with the standard card of zinc oxide. The Scherer Formula is used to determine the average size of zinc oxide. The elemental analysis of all synthesized samples is highly satisfied provide by the EDX results. The detector is successfully constructed by using PVA composite and the ZnO and CNTs exist as additive for EM signal detection. The testing and validation result really meet the research expectation which the detection enhancement is about 70% by using developed prototype. The ZnO-CNTs based EM detector was successfully developed.

5.2 Recommendation

ZnO-CNTs filled PVA composite detector test can be done in the water or salt water. The effect of EM waves to the layers can be studied.

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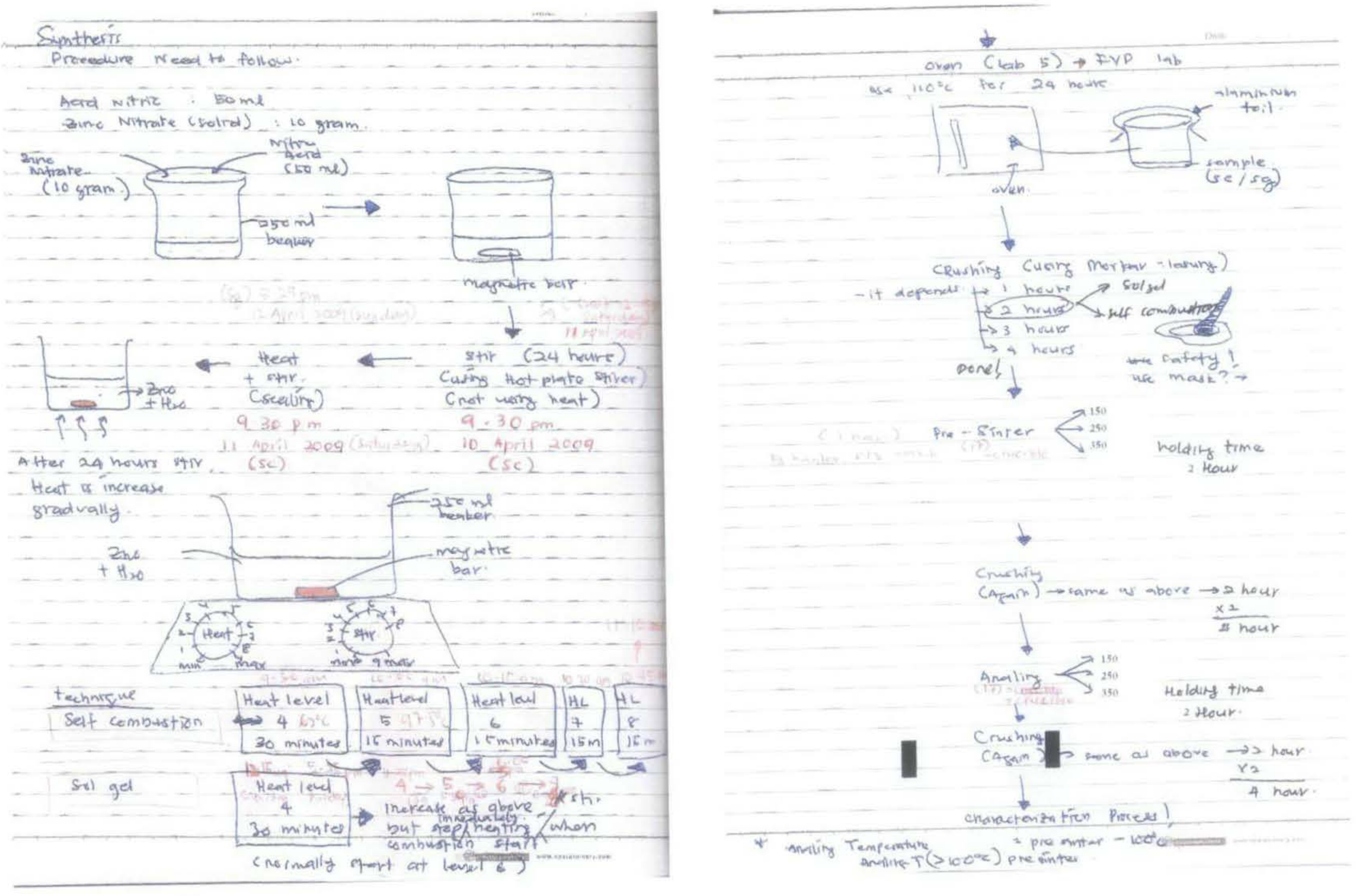
APPENDICES

Synthesis of ZnO Nanoparticles (Scanned from jotter book)



Synthesis

Procedure Need to follow.

Acid nitric : 50 ml

Zinc Nitrate (solid) : 10 gram.

stir (24 hours)
(using Hot plate Stirer)
(not using heat)
9.30 pm
10 April 2009
(SC)

Heat + stir. (Scaling)
9 30 pm
11 April 2009 (Saturday)
(SC)

After 24 hours stir, Heat is increase gradually.

technique

Self combustion	Heat level 4 65°C 30 minutes	Heat level 5 97.5°C 15 minutes	Heat level 6 15 minutes	HL 7 15m	HL 8 15m
Sol gel	Heat level 4 30 minutes	4 → 5 → 6 → 7			

Increase as above but stop heating immediately when combustion start

(normally start at level 6)

oven (lab 5) → FYP lab

use 110°C for 24 hours

Crushing (using Mortar - lesung)

- it depends → 1 hours
→ 2 hours → Sol gel
→ self combustion
→ 3 hours
→ 4 hours

safety! use mask? →

Done!

Pre - Sinter → 150, 250, 350

holding time 2 Hour

Crushing (Again) → same as above → 2 hour
x 2
4 hour

Annealing → 150, 250, 350

Holding time 2 Hour.

[redacted] Crushing (Again) [redacted] → same as above → 2 hour
x 2
4 hour.

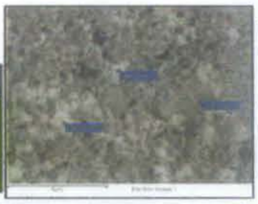
characterization Process!

* Annealing Temperature = pre sinter - 100°C
Annealing T (> 100°C) pre sinter

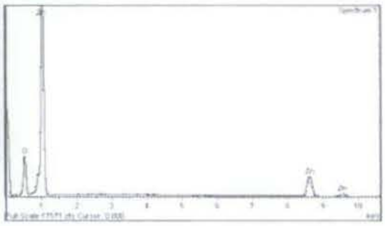
EDX Results for ZnO (Self combustion technique)

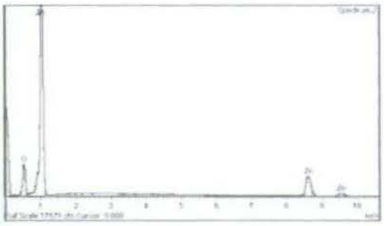
EDX ZnO SC 250

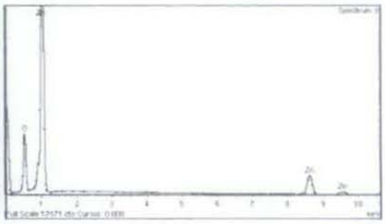
Expected Result		Weight %	Atomic %
Spectrum 1	O	50.00	50.00
	Zn	50.00	50.00
	Total	100	100



Actual Results		Weight %	Atomic %	Peak Possible Omitted
Spectrum 1	O	20.16	50.78	0.252 keV
	Zn	79.84	49.22	
	Total	100	100	
Spectrum 2	O	16.97	45.50	No peaks omitted
	Zn	83.03	54.50	
	Total	100	100	
Spectrum 3	O	22.07	53.65	0.252 keV
	Zn	77.93	49.35	
	Total	100	100	



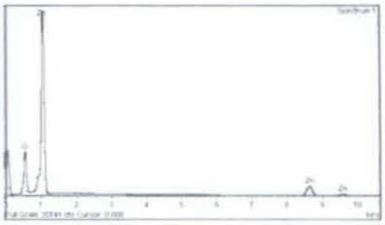


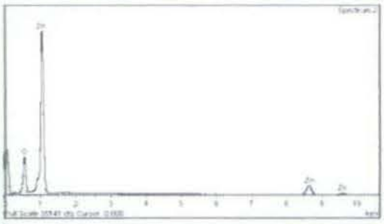


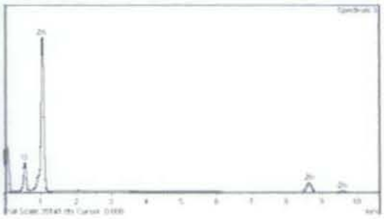
EDX ZnO SC 350

Expected Result		Weight %	Atomic %
Spectrum 1	O	50.00	50.00
	Zn	50.00	50.00
	Tota	100	100

Actual Results		Weight %	Atomic %	Peak Possible Omitted
Spectrum 1	O	23.59	50.79	0.250 keV
	Zn	76.41	44.21	
	Total	100	100	
Spectrum 2	O	23.82	56.09	0.255, 5.450 keV
	Zn	76.18	43.91	
	Total	100	100	
Spectrum 3	O	20.92	51.95	0.251 keV
	Zn	79.08	48.05	
	Total	100	100	



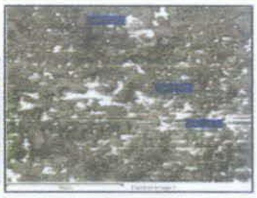




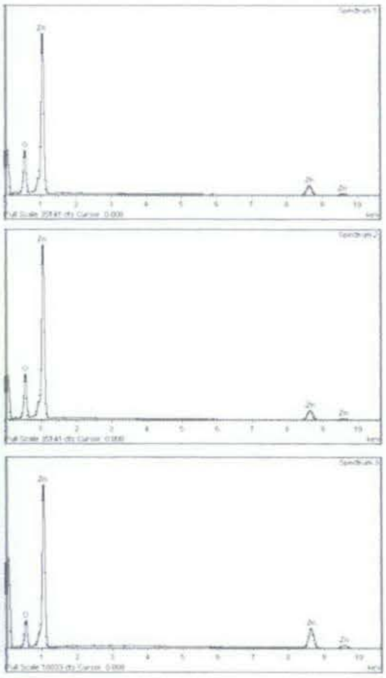
EDX Results for ZnO (Sol-gel technique)

EDX ZnO SG 250

Expected Result		Weight %	Atomic %
Spectrum 1	O	50.00	50.00
	Zn	50.00	50.00
	Tota	100	100

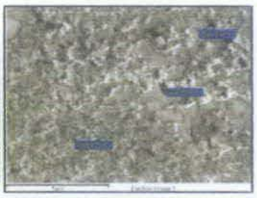


Actual Results		Weight %	Atomic %	Peak Possible Omitted
Spectrum 1	O	26.10	59.07	0.256 keV
	Zn	73.90	40.93	
	Total	100	100	
Spectrum 2	O	25.47	58.27	0.251 keV
	Zn	74.53	41.73	
	Total	100	100	
Spectrum 3	O	19.26	49.36	No peaks omitted
	Zn	80.74	50.64	
	Total	100	100	

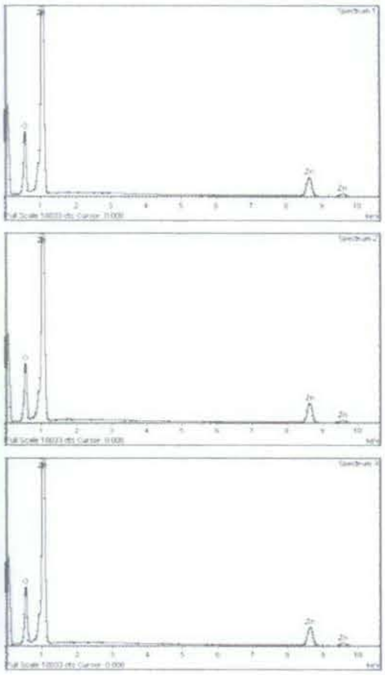


EDX ZnO SG 350

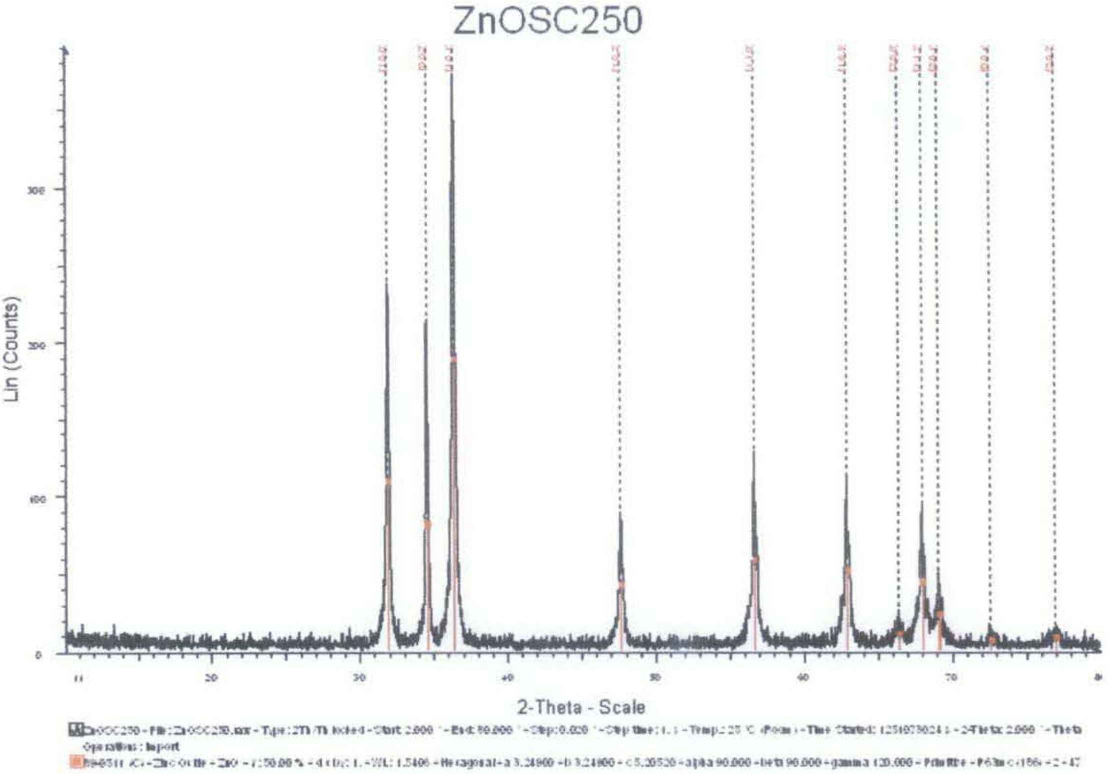
Expected Result		Weight %	Atomic %
Spectrum 1	O	50.00	50.00
	Zn	50.00	50.00
	Tota	100	100

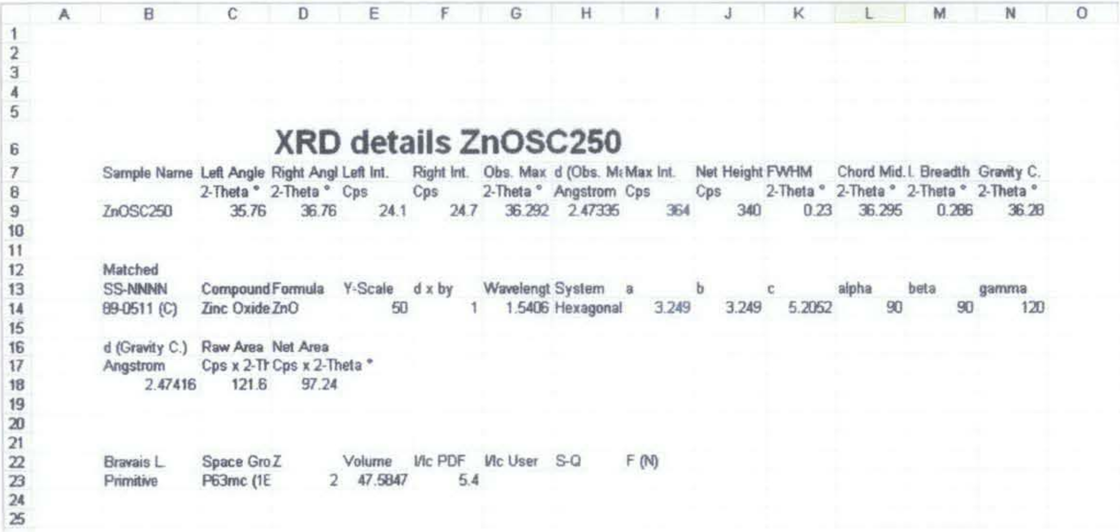


Actual Results		Weight %	Atomic %	Peak Possible Omitted
Spectrum 1	O	21.74	53.16	0.250 keV
	Zn	78.26	46.84	
	Total	100	100	
Spectrum 2	O	21.28	52.49	0.251 keV
	Zn	78.72	47.51	
	Total	100	100	
Spectrum 3	O	21.38	52.63	0.252 keV
	Zn	78.62	47.37	
	Total	100	100	



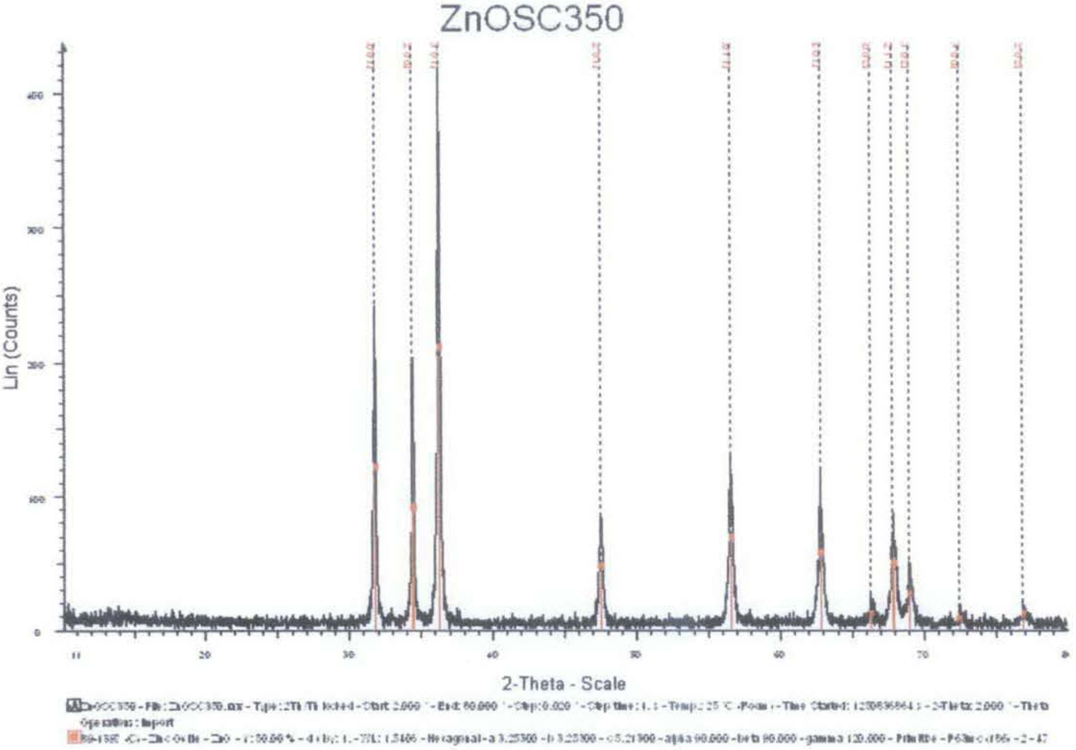
XRD Results for ZnOSC250

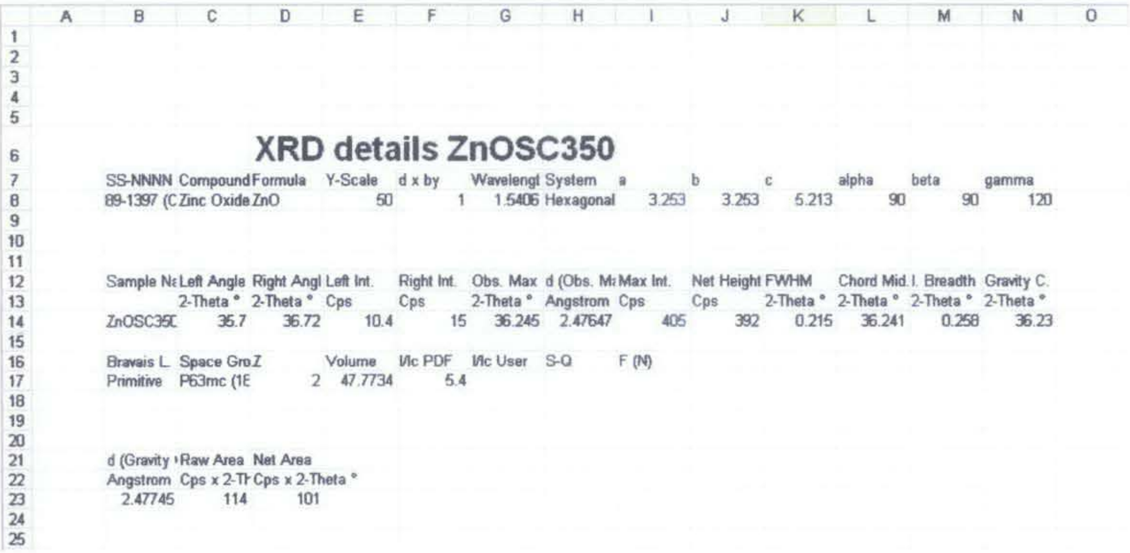




	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1															
2															
3															
4															
5															
6				**XRD details ZnOSC250**											
7		Sample Name	Left Angle	Right Angl	Left Int.	Right Int.	Obs. Max	d (Obs. M	Max Int.	Net Height	FWHM	Chord Mid.	I. Breadth	Gravity C.	
8			2-Theta °	2-Theta °	Cps	Cps	2-Theta °	Angstrom	Cps	Cps	2-Theta °	2-Theta °	2-Theta °	2-Theta °	
9		ZnOSC250	35.76	36.76	24.1	24.7	36.292	2.47335	364	340	0.23	36.295	0.286	36.28	
10															
11															
12		Matched													
13		SS-NNNN	Compound	Formula	Y-Scale	d x by	Wavelengt	System	a	b	c	alpha	beta	gamma	
14		89-0511 (C)	Zinc Oxide	ZnO	50	1	1.5406	Hexagonal	3.249	3.249	5.2052	90	90	120	
15															
16		d (Gravity C.)	Raw Area	Net Area											
17		Angstrom	Cps x 2-Th	Cps x 2-Theta °											
18		2.47416	121.6	97.24											
19															
20															
21															
22		Bravais L.	Space Gro	Z	Volume	I/c PDF	I/c User	S-Q	F (N)						
23		Primitive	P63mc (1E	2	47.5847	5.4									
24															
25															

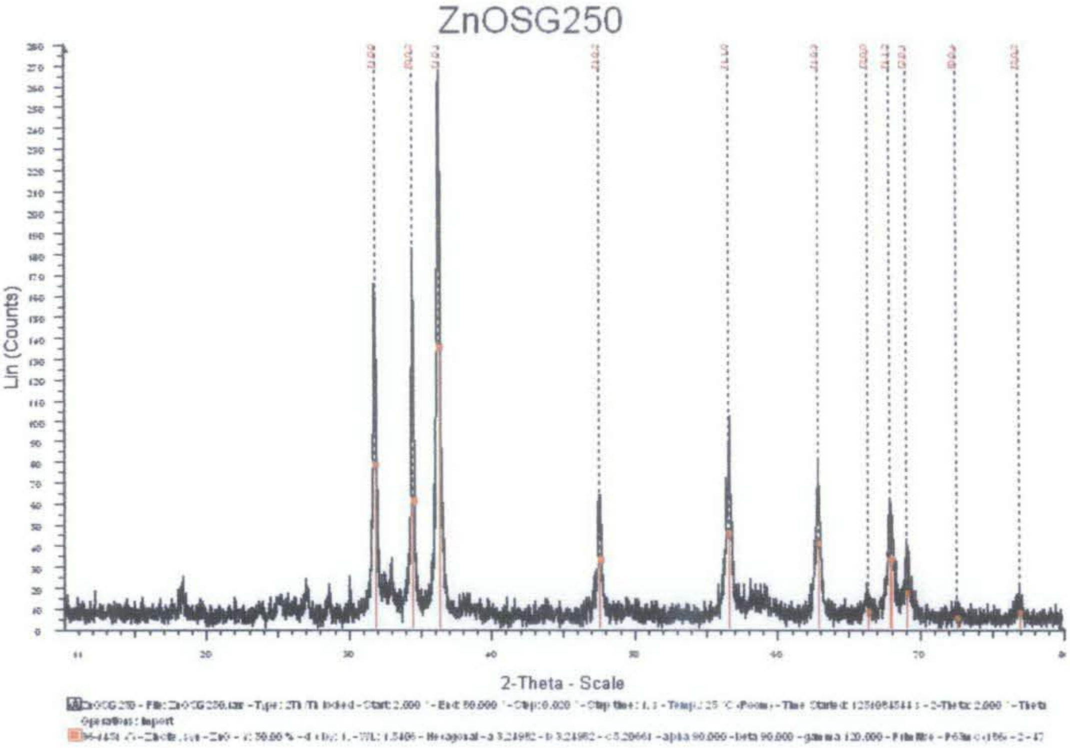
XRD Results for ZnOSC350

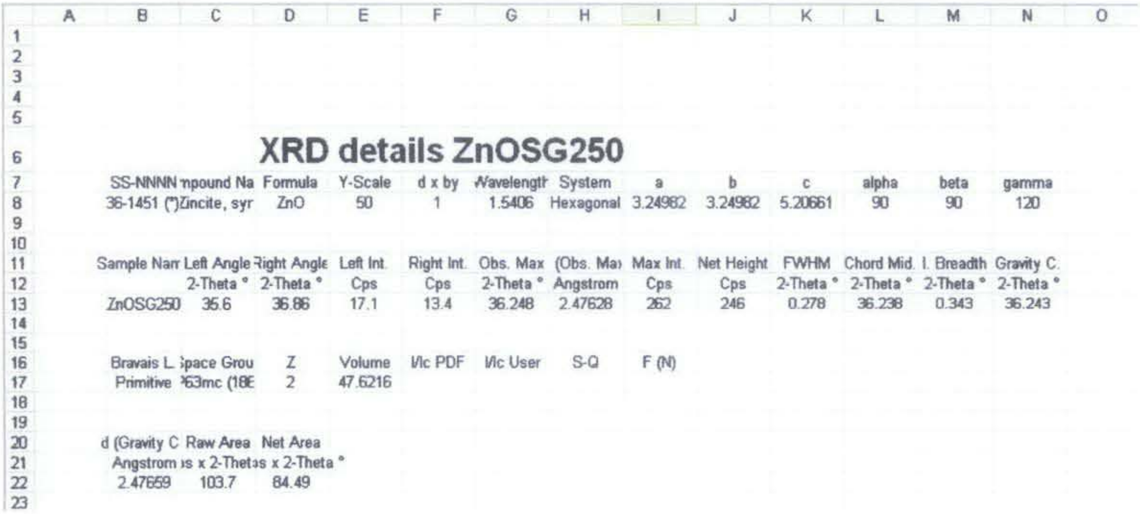




	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1															
2															
3															
4															
5															
6				XRD details ZnOSC350											
7		SS-NNNN	Compound	Formula	Y-Scale	d x by	Wavelengt	System	a	b	c	alpha	beta	gamma	
8		89-1397 (C	Zinc Oxide	ZnO	50	1	1.5406	Hexagonal	3.253	3.253	5.213	90	90	120	
9															
10															
11															
12		Sample N	Left Angle	Right Angl	Left Int.	Right Int.	Obs. Max	d (Obs. M	Max Int.	Net Height	FWHM	Chord Mid.	I. Breadth	Gravity C.	
13			2-Theta °	2-Theta °	Cps	Cps	2-Theta °	Angstrom	Cps	Cps	2-Theta °	2-Theta °	2-Theta °	2-Theta °	
14		ZnOSC35	35.7	36.72	10.4	15	36.245	2.47647	405	392	0.215	36.241	0.258	36.23	
15															
16		Bravais L	Space Gro	Z	Volume	I/Ic PDF	I/Ic User	S-Q	F (N)						
17		Primitive	P63mc (1	2	47.7734	5.4									
18															
19															
20															
21		d (Gravity	Raw Area	Net Area											
22		Angstrom	Cps x 2-Th	Cps x 2-Theta °											
23		2.47745	114	101											
24															
25															

XRD Results for ZnOSG250





XRD details ZnOSG250

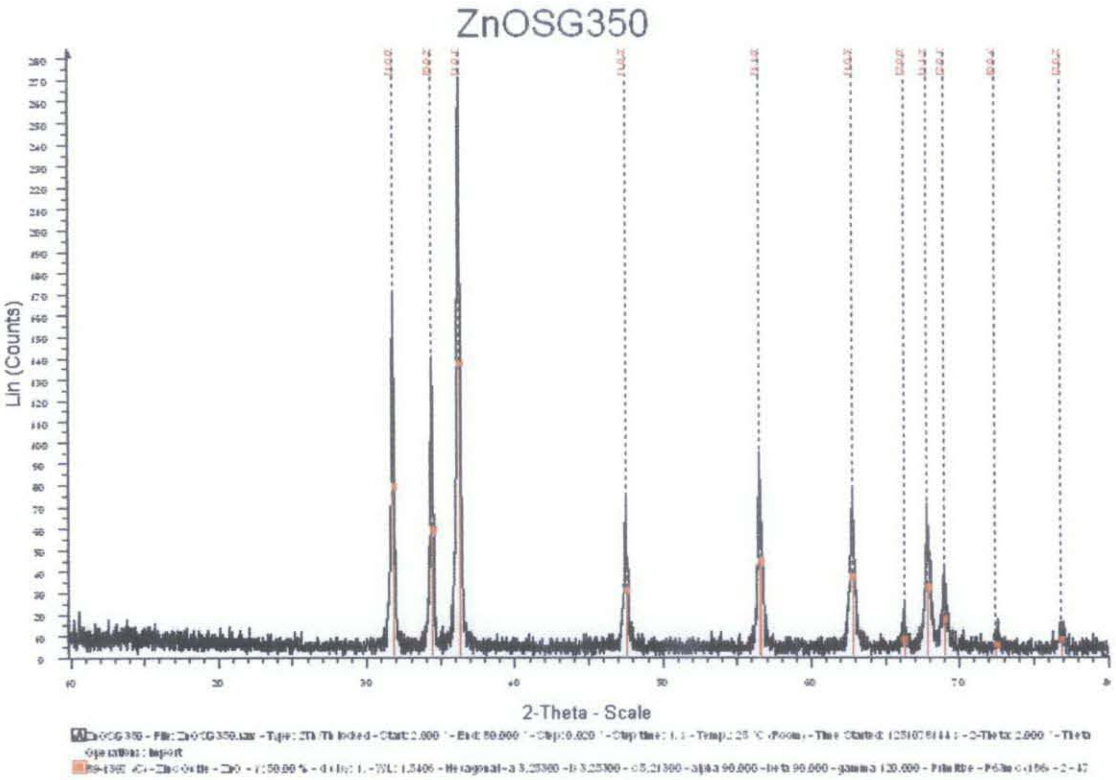
SS-NNNN	npound Na	Formula	Y-Scale	d x by	Wavelengtl	System	a	b	c	alpha	beta	gamma
36-1451 (*)	Zincite, syr	ZnO	50	1	1.5406	Hexagonal	3.24982	3.24982	5.20661	90	90	120

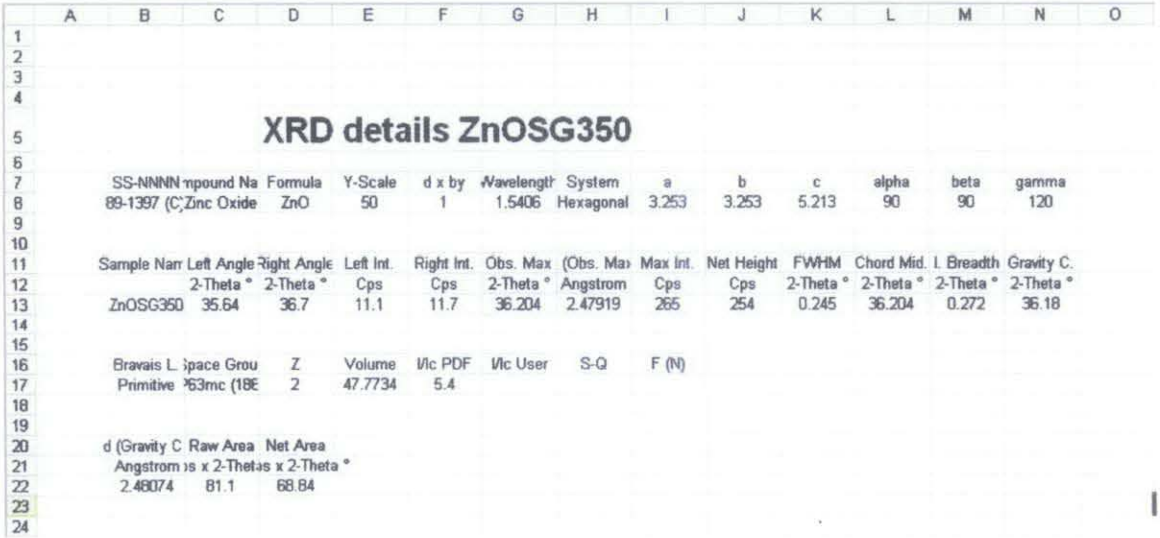
Sample Nam	Left Angle 2-Theta °	Right Angle 2-Theta °	Left Int. Cps	Right Int. Cps	Obs. Max 2-Theta °	(Obs. Ma Angstrom	Max Int. Cps	Net Height Cps	FWHM 2-Theta °	Chord Mid. 2-Theta °	I. Breadth 2-Theta °	Gravity C. 2-Theta °
ZnOSG250	35.6	36.86	17.1	13.4	36.248	2.47628	262	246	0.278	36.238	0.343	36.243

Bravais L.	Space Grou	Z	Volume	I/Ic PDF	I/Ic User	S-Q	F (N)
Primitive	P63mc (18E	2	47.6216				

d (Gravity C Angstrom	Raw Area ps x 2-Theta	Net Area ps x 2-Theta °
2.47659	103.7	84.49

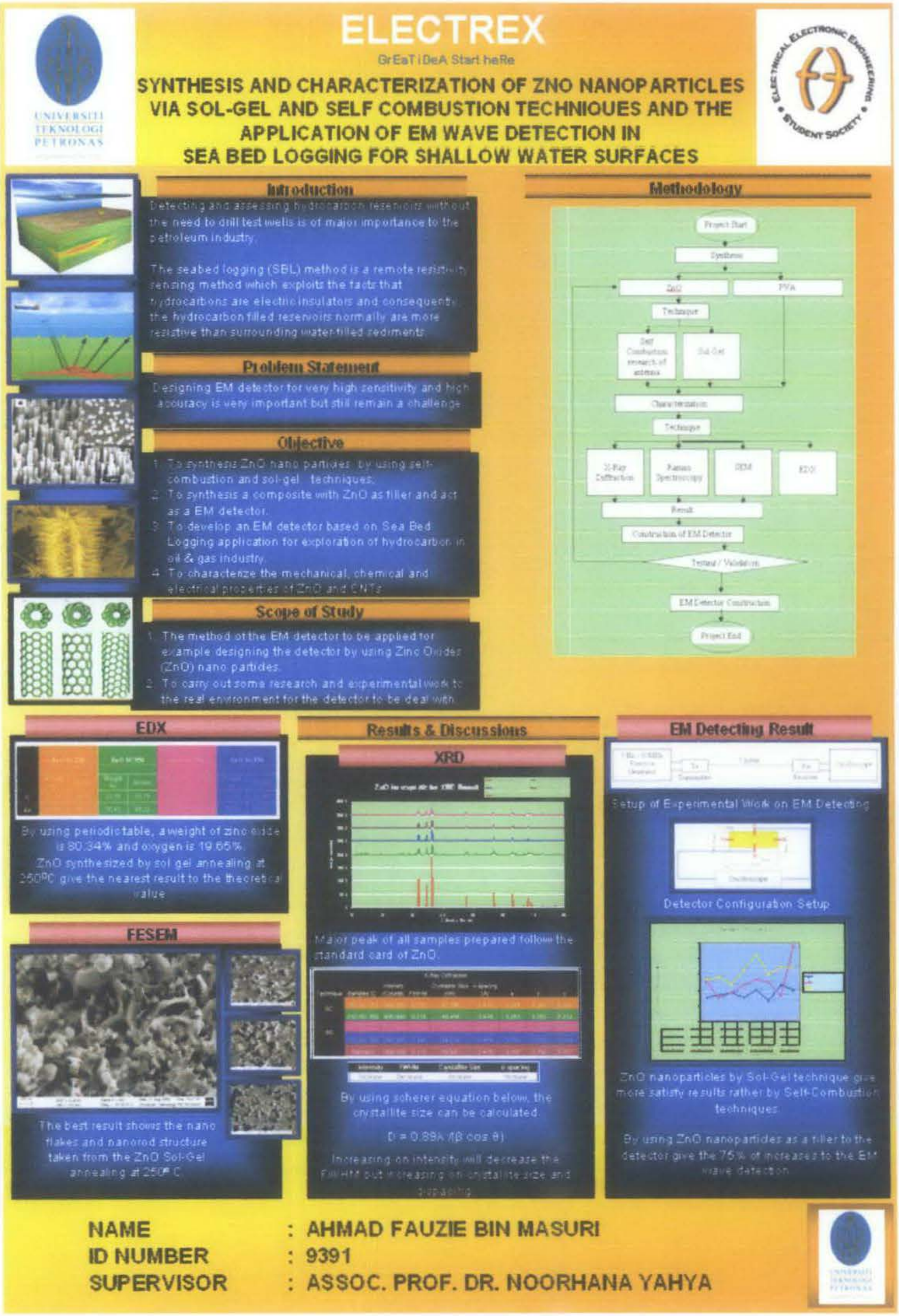
XRD Results for ZnOSG350





	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1															
2															
3															
4															
5				**XRD details ZnOSG350**											
6															
7		SS-NNNN	mpound Na	Formula	Y-Scale	d x by	Wavelength	System	a	b	c	alpha	beta	gamma	
8		89-1397 (C	Zinc Oxide	ZnO	50	1	1.5406	Hexagonal	3.253	3.253	5.213	90	90	120	
9															
10															
11		Sample Nam	Left Angle	Right Angle	Left Int.	Right Int.	Obs. Max	(Obs. Max	Max Int.	Net Height	FWHM	Chord Mid.	I. Breadth	Gravity C.	
12			2-Theta °	2-Theta °	Cps	Cps	2-Theta °	Angstrom	Cps	Cps	2-Theta °	2-Theta °	2-Theta °	2-Theta °	
13		ZnOSG350	35.64	36.7	11.1	11.7	36.204	2.47919	265	254	0.245	36.204	0.272	36.18	
14															
15															
16		Bravais L	Space Grou	Z	Volume	I/Ic PDF	I/Ic User	S-Q	F (N)						
17		Primitive	P63mc (18E	2	47.7734	5.4									
18															
19															
20		d (Gravity C	Raw Area	Net Area											
21		Angstrom	s x 2-Theta	s x 2-Theta °											
22		2.48074	81.1	68.84											
23															
24															

Electrex Poster Exhibition



ELECTREX

GrEaT iDeA Start heRe

SYNTHESIS AND CHARACTERIZATION OF ZNO NANOPARTICLES VIA SOL-GEL AND SELF COMBUSTION TECHNIOUES AND THE APPLICATION OF EM WAVE DETECTION IN SEA BED LOGGING FOR SHALLOW WATER SURFACES

Introduction

Detecting and assessing hydrocarbon reservoirs without the need to drill test wells is of major importance to the petroleum industry.

The seabed logging (SBL) method is a remote resistivity sensing method which exploits the facts that hydrocarbons are electric insulators and consequently the hydrocarbon filled reservoirs normally are more resistive than surrounding water-filled sediments.

Problem Statement

Designing EM detector for very high sensitivity and high accuracy is very important but still remain a challenge.

Objective

1. To synthesis ZnO nano particles by using self-combustion and sol-gel techniques.
2. To synthesis a composite with ZnO as filler and act as a EM detector.
3. To develop an EM detector based on Sea Bed Logging application for exploration of hydrocarbon in oil & gas industry.
4. To characterize the mechanical, chemical and electrical properties of ZnO and CNTs.

Scope of Study

1. The method of the EM detector to be applied for example designing the detector by using Zinc Oxides (ZnO) nano particles.
2. To carry out some research and experimental work to the real environment for the detector to be deal with.

Methodology

EDX

By using periodic table, a weight of zinc oxide is 80.34% and oxygen is 19.66%.
ZnO synthesized by sol gel annealing at 250ºC give the nearest result to the theoretical value

FESEM

The best result shows the nano flakes and nanorod structure taken from the ZnO Sol-Gel annealing at 250º C.

Results & Discussions

XRD

Major peak of all samples prepared follow the standard card of ZnO.

By using scherer equation below, the crystallite size can be calculated.

$D = 0.89\lambda / (\beta \cos \theta)$

Increasing on intensity will decrease the FWHM but increasing on crystallite size and d-spacing

EM Detecting Result

Setup of Experimental Work on EM Detecting

Detector Configuration Setup

ZnO nanoparticles by Sol-Gel technique give more satisfy results rather by Self-Combustion techniques.

By using ZnO nanoparticles as a filler to the detector give the 75% of increases to the EM wave detection.

NAME : **AHMAD FAUZIE BIN MASURI**
ID NUMBER : **9391**
SUPERVISOR : **ASSOC. PROF. DR. NOORHANA YAHYA**

UNIVERSITI TEKNOLOGI PETRONAS

ZnO – CNTs Based EM SENSORS

ENGINEERING DESIGN EXHIBITION 24

Introduction

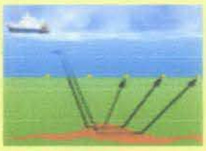
Detecting and assessing hydrocarbon reservoirs without the need to drill test wells is of major importance to the petroleum industry. The seabed logging (SBL) method is a remote resistivity sensing method which exploits the facts that hydrocarbons are electric insulators and consequently, the hydrocarbon filled reservoirs normally are more resistive than surrounding water-filled sediments.

Problem Statement

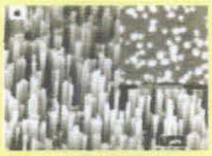
Designing EM detector for very high sensitivity and high accuracy is very important but still remain a challenge.

Objective

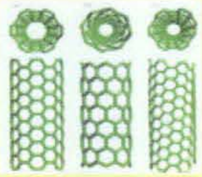
1. To synthesis ZnO nano particles by using self-combustion and sol-gel techniques;
2. To synthesis and characterize ZnO-CNTs filled PVA-composite as EM detector.
3. To develop EM detector prototype potentially used for Sea Bed Logging.
4. To test the detection of electromagnetic waves using a data logger (PicoLog ADC-16)



Sea Bed Logging

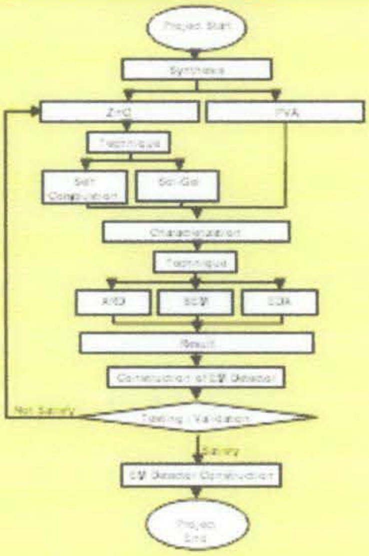


Nano Rod Structure



Carbon Nanotubes Structure

Methodology



Project Start

Synthesis

ZnO

PVA

Technique

Self Combustion

Sol-Gel

Characterization

Technique

XRD

SEM

EDX

Result

Construction of EM Detector

Testing / Validation

Not Satisfy

Satisfy

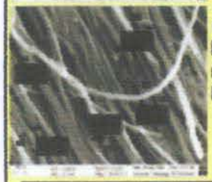
EM Detector Optimization

Project End

Results & Discussions

FESEM

ZnO Nanoparticles Morphology (used for EM sensor Model RX02)

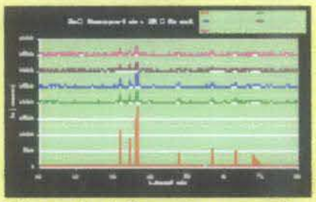


Carbon Nanotubes Morphology (used for EM sensor Model RX06)

EDX

By using periodic table, a weight of zinc oxide is 80.34% and oxygen is 19.65%. ZnO synthesized has follow the standard percentage.

XRD



Major peak of all samples prepared follow the standard card of ZnO.

By using scherer equation below, the crystallite size can be calculated.

$D = 0.89\lambda / (\beta \cos \theta)$

Increasing on intensity will decrease the FWHM but increasing on crystallite size and d-spacing.

TESTING & VALIDATION

Experimental Work Setup

Detector Configuration 1

Detector Configuration 2

MAJOR FINDING !

IT WAS FOUND THAT THE NEW ZnO-CNTs based EM SENSOR IS ABLE TO ENHANCE THE DETECTION BY 75%

ADVANTAGE OF OUR EM SENSORS

1. HIGH SENSITIVITY
2. CHEAP & EASY TO MAKE
3. LIGHT & FLEXIBLE
4. ENVIRONMENTAL FRIENDLY
5. EXTENDED TO EXTREMELY LARGE SURFACE AREA

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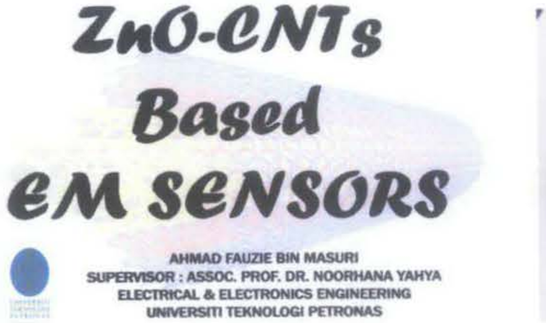
Achievements and Acknowledgements



ZnO-CNTs
Based
EM SENSORS

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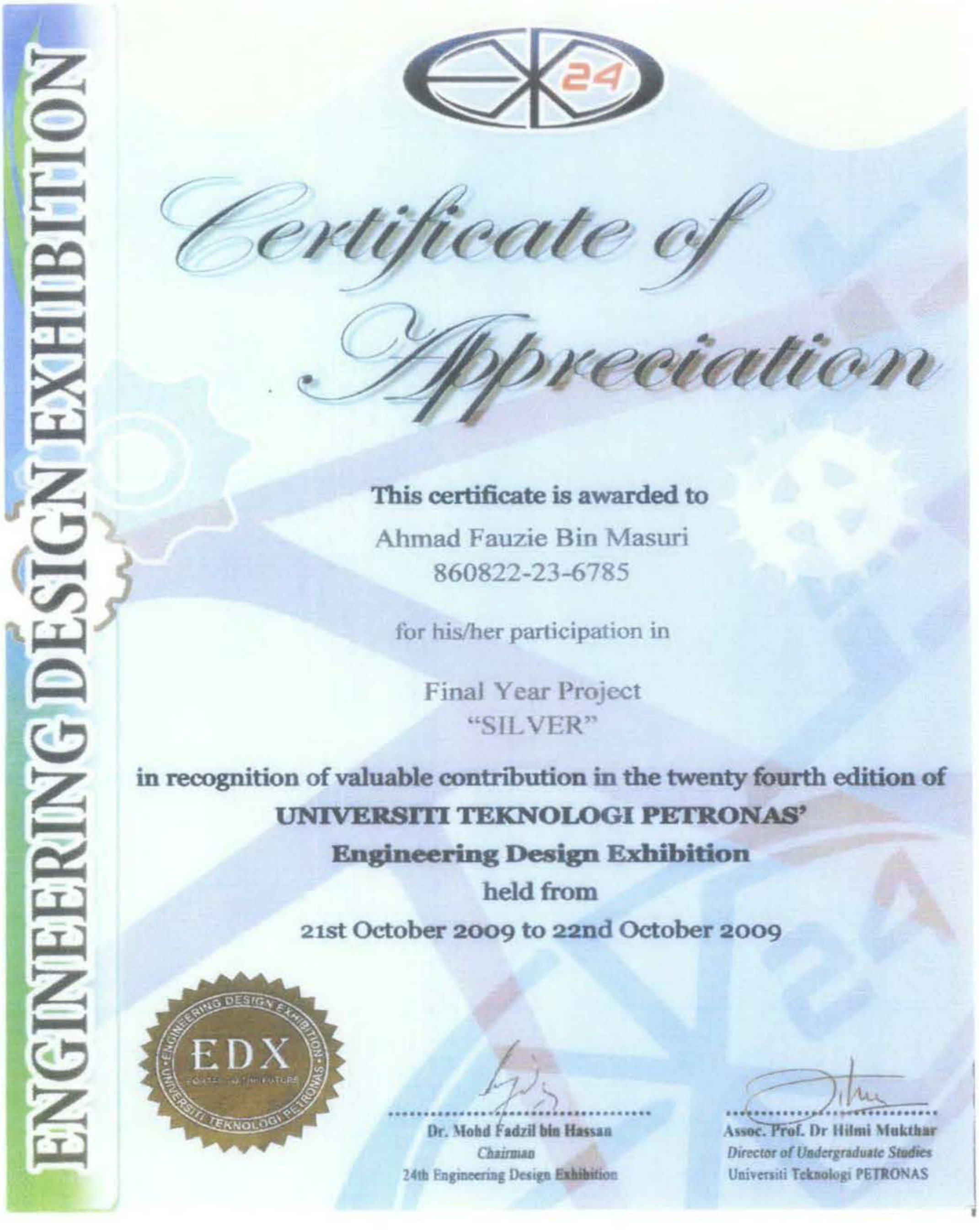

ENGINEERING DESIGN EXHIBITION

Certificate of Appreciation

This certificate is awarded to

Ahmad Fauzie Bin Masuri
860822-23-6785

for his/her participation in

Final Year Project
"SILVER"

in recognition of valuable contribution in the twenty fourth edition of
UNIVERSITI TEKNOLOGI PETRONAS'
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Dr. Mohd Fadzil bin Hassan
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