EFFECT OF DIFFERENT DOPING METHOD ON THE PERFORMANCE OF NIO AND NIO DOPED COPPER FOR HTL IN INVERTED PEROVSKITE SOLAR CELL

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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This report is submitted in partial fulfilment of the requirements for the degree of Bachelor of Computer Engineering with Honours

Faculty of Electronic and Computer Technology and Engineering
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PROJEK SARJANA MUDA II

Tajuk Projek : Effect of Different Doping Methods on the Performance of NiO and NiO doped Copper for HTL in Invented Perovskite Solar Cell.

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DEDICATION

I dedicate this thesis to my beloved parents, Mr Low Yam Lee, Mrs Ng Thai Pee, my supervisor, Dr. Zul Atfyi Fauzan bin Mohammed Napiah, and co-supervisor, Dr. Muhammad Idzdihar bin Idris, as well as all my friends and Mine lab’s colleagues for their support, encourage and cooperation. Thank you very much.

ABSTRACT

Perovskite solar cells (PSCs) show great potential as a replacement to traditional silicon-based solar cells due to their high productivity and low price. However, freshly inverted perovskite solar cells (IPSC) which is the inverted of PSC have gotten a lot of interest in recent years because of their consistent operational stability, reduced hysteresis, and low fabrication procedure. Among the materials used in p-type semiconductor, nickel oxide (NiO) is a material used in hole transport layer (HTL) due to its transparency and conductivity. But the problem is that while NiO contacts with perovskite it will cause low conductivity and unsatisfactory interface contact. So, the objective is to synthesis the NiO and doped NiO, and to characterize undoped and doped NiO as HTL in IPSC. As a result, the characteristics test for pure and doped NiO using ultraviolet-visible spectra (UV-Vis), scanning electron microscope (SEM), and X-ray Diffraction (XRD). From these results, it can be concluded that doped NiO is a potential material to replace pure NiO as HTL for IPSCs.

ABSTRAK

Sel suria perovskite (PSC) berpotensi besar sebagai pengganti kepada sel suria berasaskan silikon kerana produktivitinya yang tinggi dan harga yang rendah. Walau bagaimanapun, sel suria perovskite terbalik (IPSC) iaitu PSC yang terbalik di mana ia telah mendapat minat yang meluas sejak kebelakangan ini kerana kestabilan operasinya yang konsisten, histeresis yang berkurangan dan prosedur fabrikasi yang rendah. Antara bahan yang digunakan dalam semikonduktor jenis-p, nikel oksida (NiO) merupakan bahan yang digunakan dalam lapisan pengangkutan lubang (HTL) kerana ketelusan dan kekonduksian yang lebih baik. Tetapi masalahnya apabila NiO bersentuhan dengan perovskite ia akan menyebabkan kekonduksiannya menjadi rendah dan hubungan antara muka menjadi tidak memuaskan. Oleh itu, objektif projek ini ialah untuk sintesis NiO tulen dan NiO terdop, dan mencirikan NiO yang tidak didop dan yang didop sebagai HTL dalam IPSC. Hasilnya, ujian ciri untuk tulen dan NiO terdop menggunakan spektrum boleh dilihat - ultraungu (UV-Vis), mikroskop pengimbasan elektron (SEM),) dan Belauan sinar-X (XRD). Kesimpulannya, NiO terdop berpotensi untuk menggantikan NiO tulen sebagai HTL untuk IPSC.

ACKNOWLEDGEMENTS

Firstly, this bachelor’s degree project and the report was successfully completed with given timeline and gratitude to supervisor, Dr Zul Atfyi Fauzan bin Mohammed Napiah, and co-supervisor, Dr Muhammad Idzdihar bin Idris, for mentor and give advice during reviewing reports, presentation slides, and project progress. Besides that, students are pleased to seniors and friends who have given help when needed during this project. Students would like to share joy with parents for their motivation and cheer up, which on his suffer time during this project. Lastly, students thank towards the faculty for having a well-resourced laboratory to let students able to complete the project.

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LIST OF SYMBOLS AND ABBREVIATIONS

HTL	:	Hole transport layer
IPSC	:	Inverted Perovskite Solar Cell
FTO	:	Fluorine doped tin oxide
ETL	:	Electron transport layer
ITO	:	Indium Tin Oxide
KOH	:	Potassium hydroxide
NiO	:	Nickel Oxide
NaOH	:	Sodium hydroxide
SEM	:	Scanning electron microscope
UV-Vis	:	Ultraviolet–visible spectroscopy
XRD	:	X-ray powder diffraction
EDX		Energy-dispersive X-ray Spectroscopy
PCE	:	Power conversion efficiency

CHAPTER 1

INTRODUCTION

The project introduction is covered in this chapter. Furthermore, it emphasizes the issue of the current state of industry, which leads to the project's development. In addition, the research background, objective, problem statement and scope of work will all be covered in this chapter.

.

1.1 Background

Energy plays a pivotal role in our daily lives, and its demand continues to grow. Sustainable development is nearly impossible without reliable energy sources. Globally, electrical energy consumption stands at approximately 12-13 Terawatts (TW) [1]. Remarkably, our planet receives more solar energy in just one hour than the entire annual global solar energy usage, thanks to the sun’s constant radiance in the upper atmosphere. Solar energy offers a renewable, pollution-free, and sustainable solution. Solar cells, which convert sunlight into electricity, emit no hazardous gases contributing to global warming.

Photovoltaic solar energy, harnessed from sunlight, boasts several advantages over other energy sources. It is free, requires no fuel, and generates no waste or pollution [2]. Over the years, various photovoltaic technologies have emerged, including non-organic, hybrid, and organic solar cells.

Therefore, in this project focused on IPSC, which is under the third generation of solar cells. IPSC, part of the third generation of solar cells, utilize a perovskite-structured compound as the light-harvesting active layer. In IPSC, the HTL is deposited first, followed by the ETL. These cells hold promise due to their simple fabrication technique, PCE, and low hysteresis. The general structure of IPSCs includes the substrate, transparent electrode (ITO or FTO glass), HTL (collecting holes), perovskite layer (transporting charge), ETL (extracting electrons), and top electrode (metal back contact) [3]. Researchers explore both organic and inorganic HTLs, with nickel oxide (NiO) emerging as a stable and low cost.

In summary, IPSCs represent a clean and efficient path toward harnessing solar energy, and ongoing research aims to optimize their performance and stability.

Moreover, the research findings could potentially lead to the development of more efficient and durable solar cells. This would be a significant contribution to the field of renewable energy, particularly in the context of increasing global energy demand and the urgent need for sustainable and environmentally friendly solutions.

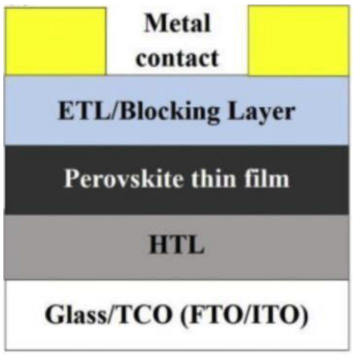


Figure 1.1: General structure of IPSC [3].

1.2 Problem Statement

In recent years, Inverted Perovskite Solar Cells (IPSC) have been quite common and developed rapidly due to their cheap cost fabrication, good stability and high-power conversion efficiency (PCE), with the certified record exceeding 22% as per NREL. Despite these, they still have challenges remain, such as durability of HTL material are not stable that can cause fast degradation over time. So, the selection of material are crucial factors that directly influence the device's performance. Nickel Oxide (NiO) has emerged as a promising HTL material, which is the most widely used as HTL to provide intrinsically higher stability and exhibits lower cost compared to organic polymer-material. Besides that, a variety of improvements have been explored, for example surface passivation, interface engineering, doping, and cross-linking of the perovskite film can enhance IPSC's PCE [4].

However, to improve further, the doping strategies are still the best introduce to optimize its characteristics and boost overall performance. This leads to the which doped NiO with what materials could be a better solution and what efforts able to improve quality of deposited for NiO and doped NiO. There must be a reasonable concern on which choice of materials, their doping level, and their effects on the performance and stability of IPSC. So, this study can address the problem or issue that might face during progression and to overcoming the challenges faced by IPSC.

In conclusion, this research aims to investigate different doping materials, such as gold [5], silver [6], and etc on their impacts on NiO when used as the HTL in IPSC. At the same time, the goal is to draw conclusions on the performance and efficiency of different dopants as HTL in IPSC and would pave the way for contributing to the progress of sustainable and renewable green energy technology.

1.3 Objective

The project aiming to synthesize Nickel Oxide (NiO) and Copper-doped NiO as a Hole Transport Layer (HTL) using the sol-gel spin coating and electrodeposition method. Following the synthesis, the pure NiO and Copper-doped NiO are characterized using Scanning Electron Microscopy (SEM) and Ultraviolet-Visible (UV-Vis) spectroscopy. SEM images reveal the formation of NiO nanoparticles of spherical forms. At the same time, UV-Vis spectroscopy confirms the absorption of films and their character. So, the objective is summarized as below:

i. To synthesis the NiO and NiO doped copper as HTL in IPSC using sol gel spin coating and electrodeposition method.
ii. To characterize using SEM and UV-Vis for pure NiO and NiO doped copper as HTL in IPSC.

1.4 Scope of Project

This section discusses the project application for both undoped and doped NiO as HTL in IPSC, and their characteristic or properties. Based on the current study and research NiO dope with zinc oxide, silver and copper are very common. First, the project has to synthesis using different method of different type doping on NiO and doped NiO. This synthesis process for pure NiO and doped NiO can be use different method, among the common methods such as sol-gel spin coating [7], combustion [8], electrodeposition due to the low cost and easy to fabricate. So, in the project sol gel spin coating and electrodeposition are used to synthesize and fabricate the NiO and doped NiO. Finally, the analysis and characteristics test for pure NiO and doped NiO as HTL in IPSC is make. For the test, normally the characteristic devices will test using UV Visible spectrophotometer (UV-Vis) and Scanning Electron Microscope (SEM). SEM images reveal the formation of NiO nanoparticles of spherical forms. While UV-Vis spectroscopy confirms the absorption of films and their character.

1.5 Environment and sustainability

Due to abundant raw materials and low processing costs, with a quick energy payback and low overall CO_2 emissions, this project is environmentally friendly and sustainable. Unlike typical fossil fuel technologies, solar cells do not actively emit any emissions when converting solar energy to electrical energy. As a non-polluting source of energy, perovskite solar cells are good for the environment. Thus, this project fulfills the Sustainable Development Goal 7, Clean Energy & which aims to access affordable, reliable, and sustainable energy.

Figure 1.2: Sustainable Development Goal 7, Clean Energy

1.6 Thesis outline

There is a total of five chapters in this report. First, Chapter 1 obtain the fundamental knowledge about the project introduction with several point such as background, problem statement, objectives, and scope of project.

Chapter 2 presents an encompassing review of the literature on the latest materials used in the current pure NiO and doped NiO. The methodologies for synthesizing thin films in this project also include. It contains an analysis of the materials, methods, and techniques employed by prior researchers.

Chapter 3 outlines the methodology employed for the project’s procedure. First, the description of the flow on the full preparation and how to synthesis for both pure NiO and doped NiO. Subsequently, it discusses the deposition method of NiO and doped NiO in the thin film using the sol gel spin coating technique and deposit using electrodeposition method.

Chapter 4 delves into result and analysis the sample will be employing a variety of characterization test such as X-Ray Diffractometer (XDR), UV-Visible, Scanning Electron Microscope (SEM), and Energy-dispersive X-ray Spectroscopy (EDX), and etc.

Last but not least, Chapter 5 presents an all-around conclusion. Furthermore, suggestion and possible potential directions for future research also get discuss, and setting the step for continued progress and discovery in this domain might continue.

CHAPTER 2

BACKGROUND STUDY

In this chapter, an introduction on the investigation and examination of the fundamental for the study. As a result, methods and equipment utilized by other researchers, literature review, books scholarly articles, etc. determined the methodologies, strengths, and limitations of this project to improve the solar cell's efficiency. Moreover, it is based on a past project, previous relevant studies are studied and discussed to gain additional knowledge for this study to be practical.

2.1 Introduction

Photovoltaic cells also commonly called as solar cells. It is a promising solution for renewable green energy source that can converts sunlight directly into electricity. In recently years, the evolution of solar cell technology has rapidly led to the creation of several generations of solar cells.

The initial or first generation of solar cells was based on crystalline silicon, which have a good efficient, but it was costly [9]. The second generation brought in a thin-film technologies like cadmium telluride and copper indium gallium selenide, which were less expensive but and have a bit less efficient compared to first generation [10]. The third generation have a perovskite solar cell (PSC) which are these use of the perovskite-structured compound as the active layer for light harvesting and can providing benefits such as high efficiency and low-cost production at the same time [11].

Newly in third generation has introduce the invented perovskite solar cells. IPSC involve an innovative design and aimed for having a better efficiency and good longevity in the solar cell application. One of the main concerns was the hole transport layer (HTL). The correct material used as HTL can enhance solar cell efficiency and important in IPSCs. In current research and study believes that certain materials, such as nickel oxide (NiO), are compatible with HTL. Despite NiO's already have an impressive power conversion efficiency (PCE), researchers are exploring ways to further improve it by introduced doped NiO idea.

So, to have a higher stability, efficiency, and durability in IPSC, NiO is being doped with other materials. Some of the research projects involves doping NiO with copper [12], [13], [14] shown to NiO doped copper improve the power conversion efficiency (PCE) compared to undoped NiO as HTL in IPSC. Additionally, many research and study have also been performed on different doping materials.

2.2 Solar Cell

Solar cells, also called photovoltaic cells, it specifically designed to convert sunlight into electrical energy directly. This process, also known as the photovoltaic effect, photovoltaic effect triggers an electric current when some fixed materials are exposed to sunlight [15]. By using semiconducting materials like silicon, solar cells can absorb photons from sunlight, which subsequently energize electrons and start the electricity flow [15]. So, solar cells have special ability to turn renewable energy from sunlight makes solar cells as important application to produce a clean and green power.

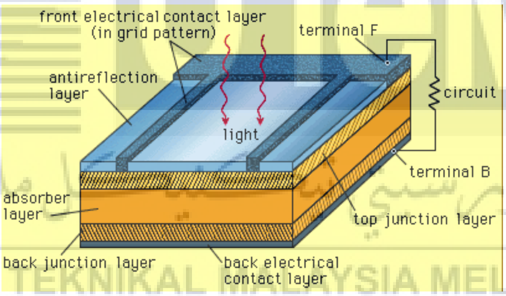


Figure 2.1: Solar cell diagram

2.3 Perovskite Solar Cell

A Perovskite Solar Cell (PSC) comes with multiple layers, but normally only consists of five important layers, each contributing to the conversion of sunlight into electrical energy. These five important layers encompass a transparent conductive electrode layer, a metal top contact layer, a perovskite layer, and electron transport layer (ETL) and hole transport layer (HTL).

2.4 Inverted Perovskite Solar Cell

As the current technology of silicon-based solar cells approaches its limit, the inverted perovskite solar cell materials are emerged as a promising alternative for the absorber layer in solar cells[16]. In IPSC the layer is reversed compared to the normal perovskite solar cell. The study shown that the PCE of inverted perovskite solar cells (IPSC) has seen a significant increase in recent years, it rise from 3.8% with the first prototype to an impressive 26% [17]. This efficiency is remarkable when compared to other thin-film photovoltaics material.

2.5 NiO as HTL materials

Hole transport layers play an important role in the performance characteristics of n-i-p (PSC) and p-i-n (IPSC) perovskite solar cells [18]. Additionally, stability and PCE are heavily dependent on the transporting layers used in the IPSC structure. Meanwhile, NiO is one of the most suitable HTL material in PSC and IPSC, because NiO has a ability to efficiently collect and transport positively charges holes from the perovskite layer to the electrode [19]. Many studies have shown that doping metal elements into NiO can significantly increase electrical conductivity. According to [20], Park mentioned that NiO can be doped with a variety of elements such as Cu, Li, Mg, Cs, and Co to enhance conductivity and PSC efficiency by lowering interfacial resistance at the HTL/perovskite interface. In conclusion, using NiO as HTL has a very good in its desirable properties, including high transparency, great electrical conductivity (movement in electrode), and match with perovskite layer.

2.6 NiO deposition method

Deposition method for NiO and doped NiO as a Hole Transport Layer (HTL) in IPSC can be performed through with two common methods, chemical and physical vapor deposition. For chemical deposition it typically comes with the direct synthesis of nanoparticles. One of the direct synthesis of nanoparticles are sol gel process, it was a particularly effective method to synthesis NiO as it enables for creation of smaller size particles with a larger surface area. Not only there have alternative method for chemical deposition for NiO as HTL material but also it also currently being utilized by research from [21]. Yang mentioned that the research had used a homemade CVD system with a vertical tube furnace and rotary vacuum pump to synthesis NiO and the nanoparticles have less than 20mn diameter which are the nice idea to synthesis NiO. On the other hand, physical vapor deposition normally utility traditional physical techniques to deposit the NiO as HTL layer. Most of the physical method offers more precise control, accuracy, and uniformity in the deposition than chemical vapours method because physical deposition is more environmentally friendly process compared to chemical deposition that involves mixing many sources material more than one precursor. However, the choice between using PVD or CVD are depending on the research requirements. In conclusion, both chemical and physical deposition methods are effective for depositing NiO and doped NiO, but the depends always on what is most reasonable or suitable for your research or experiment application. Table 2.1 below shown comparison of different method used.

Table 2.1: Comparison of deposition methods

No	Type	Technique	Description	Ref
1	Physical	Sputtering	A target material is bombarded with ions in a vacuum, causing atoms to be ejected and deposited onto a substrate.	[22]
2	Physical	Evaporation	The material is heated in a vacuum, and the vaporized atoms condense on the substrate.	[23]
3	Physical	Pulsed Laser Deposition (PLD)	PLD involves using a laser to ablate material from a target, forming a plume of material that deposits onto the substrate.	[24]
4	Physical	Electron Beam Evaporation	A high-energy electron beam is directed at a target material in a vacuum, causing the material to evaporate and condense on a substrate.	[25]
5	Chemical	Sol gel	The transformation of a solution (sol) into a gel, followed by thermal treatment to obtain the desired material.	[26]
6	Chemical	Combustion	The ignition of a mixture of reactants, leading to a self-sustaining combustion wave that propagates through the powder bed, resulting in the formation of desired products.	[27]
7	Chemical	Spary-pyrolysis	The formation of thin films by spraying a precursor solution onto a heated substrate.	[28]
8	Chemical	Atomic Layer Deposition (ALD)	A cyclic process where gaseous precursors are introduced sequentially, allowing the deposition of one atomic layer at a time.	[29]

2.7 NiO doped different materials.

Although Nickel Oxide (NiO) has been proving it had effectively material as a HTL in IPSC, but its stability and durability may not be as great as that of doped NiO. This section highlights of some high-quality dopants into NiO that can enhances its effectiveness, thereby increase the durability and stability of IPSC also. When some suitable dopants material doped with NiO, they blend well and can go further enhancing the properties of it. According to [20], Park mentioned that NiO doped with Cu, Li, Mg, Cs, and Co able to enhance conductivity efficiency.

In the paper Cu-doped NiO films also show high optical transparency in the visible region, making them suitable as window layers in solar cells. the conductivity and carrier charge of it can be highly improved the crystallinity of NiO films is enhanced through copper doping, leading to larger crystallite sizes. The depositions surface of NiO doped copper thin films glass are characterized by their smoothness, absence of holes and cracks, and display a uniform density, which is beneficial for optoelectronic applications. The energy band gap values reduce with an increase in copper concentration, indicating that Cu:NiO is suitable for optoelectronic applications. The electrical properties of devices using Cu:NiO show improved charge mobility and electrical conductivity with copper doping[13], [14]. Additionally, the surface resistance of NiO film decreases significantly after Cu doping. These advantages suggest that copper is an excellent dopant for NiO, potentially paving the way for its usage in optoelectronic applications and enhancing the performance of IPSC.

Table 2.2: Comparison of different doping materials

No.	Dopant	Description	PCE (%)	Ref
1.	Molybdenum oxide doped NiO	A simple spin coating process was used to create various mol of Ni-doped MoO3 thin film.	4.17	[30]
2.	Copper doped NiO	Spin coating was used to make copper-doped nickel oxide thin films spin at 2500 to 3000 rpm for 45 seconds and repeated 20 times.	-	[31]
3.	Fe2O-NiO doped carbon	Metal oxide nanoparticles (Fe2O3 and NiO) doped carbon electrode can enhance hole transfer in OHTM-free PSCs.	10.76	[32]
4.	Zinc doped NiO	The substrate spin 3000 rpm, 30 sec using spin coating method and repeated five times	-	[33]
5.	Pulsed Laser Deposited Cu-Doped NiO	Prepared using pulsed laser deposition but very expensive method.	20.41	[24]
6.	Lithium and Silver Co-Doped Nickel Oxide	A double doped material been used Li and Ag. The doping process is very complex and expensive.	19.24	[34]
7.	Silver-Doped NiO	Ag doped NiO using solution process method and the device use ITO/Ag:NiOx/CH3NH3PbI3/PCBM/BCP/Ag	17.3	[35]
8.	Cesium Doped NiO	Cs doped NiO prepared by solution process and the sample are smooth and transparent.	19.35	[36]

9.	Lanthanum doped NiO	A very rare material with complex measurement were needed (photoluminescence and density of states (DOS)) analysis.	19.4	[37]
10.	NiO doped nitrogen	Prepare using a facile method and the bandgap energy drop from -5.32 eV - -5.43 eV.	17.02	[38]

2.8 Summary

In this chapter 2, the conclusion is Perovskite Solar Cells (PSC) and Inverted Perovskite Solar Cells (IPSC) are a one of the great promising application solutions to have a more efficient and low-cost renewable green energy source in the future. Nickel Oxide (NiO) is widely used as material due to its transparency, electrical conductivity, and compatibility. While NiO can serves as a good HTL material, but recently research shown that its durability can be enhanced with doping. Currently many research and investigations have identified copper as one of the promising dopants for NiO due to it can increase the conductivity and durability of NiO. Therefore, copper can be considered a promising dopant material to achieve high efficiency and extended durability of IPSC.

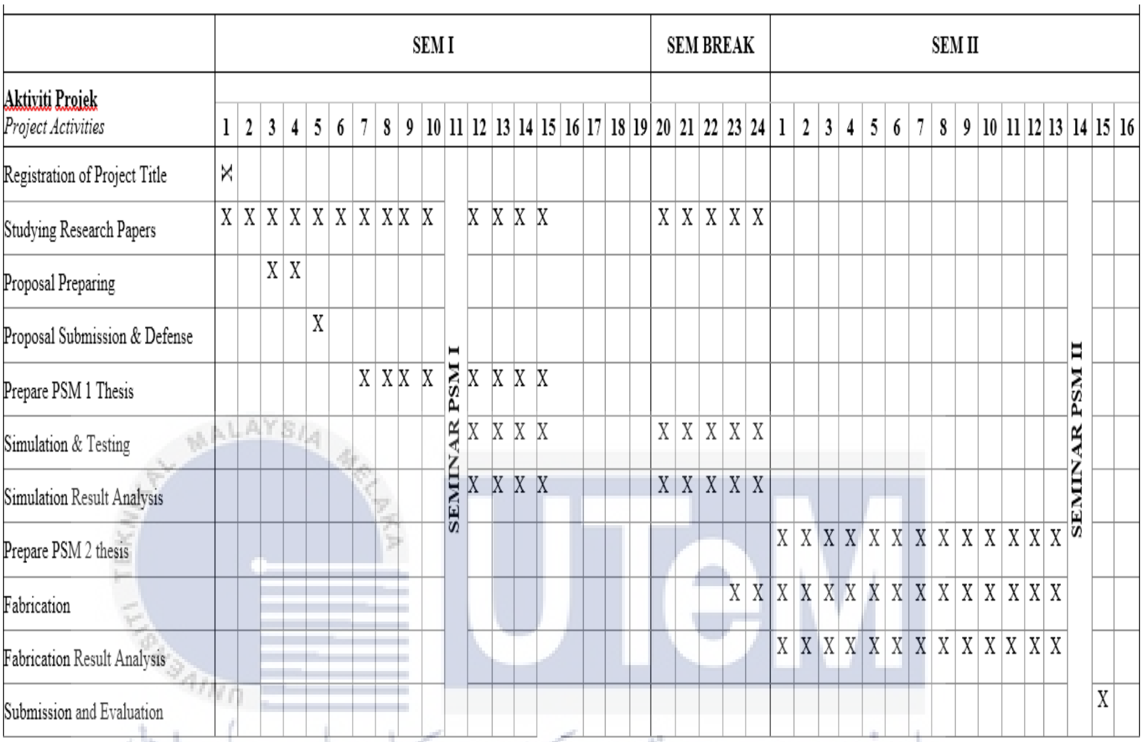
CHAPTER 3

METHODOLOGY

Methodology is closely linked to the rules and use of flow charts to show the journey of a project. The preparation of the NiO and copper doped NiO nanoparticles and also the deposition of thin film using sol gel method and electrodeposition method was discussed in this chapter. All of the implementation steps, as well as the flow chart and Gantt chart, will be thoroughly detailed in this chapter.

3.1 Gantt Chart

The Gantt chart indicates when an event is expected to occur in the project by presenting an overview of the sequence in which the activites must be completed. Figure 3.1 shows the Gantt chart of the project.



	SEM I																			SEM BREAK					SEM II															
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Registration of Project Title	X																																							
Studying Research Papers	X	X	X	X	X	X	X	X	X	X	**SEMINAR PSM I**	X	X	X	X					X	X	X	X	X														**SEMINAR PSM II**		
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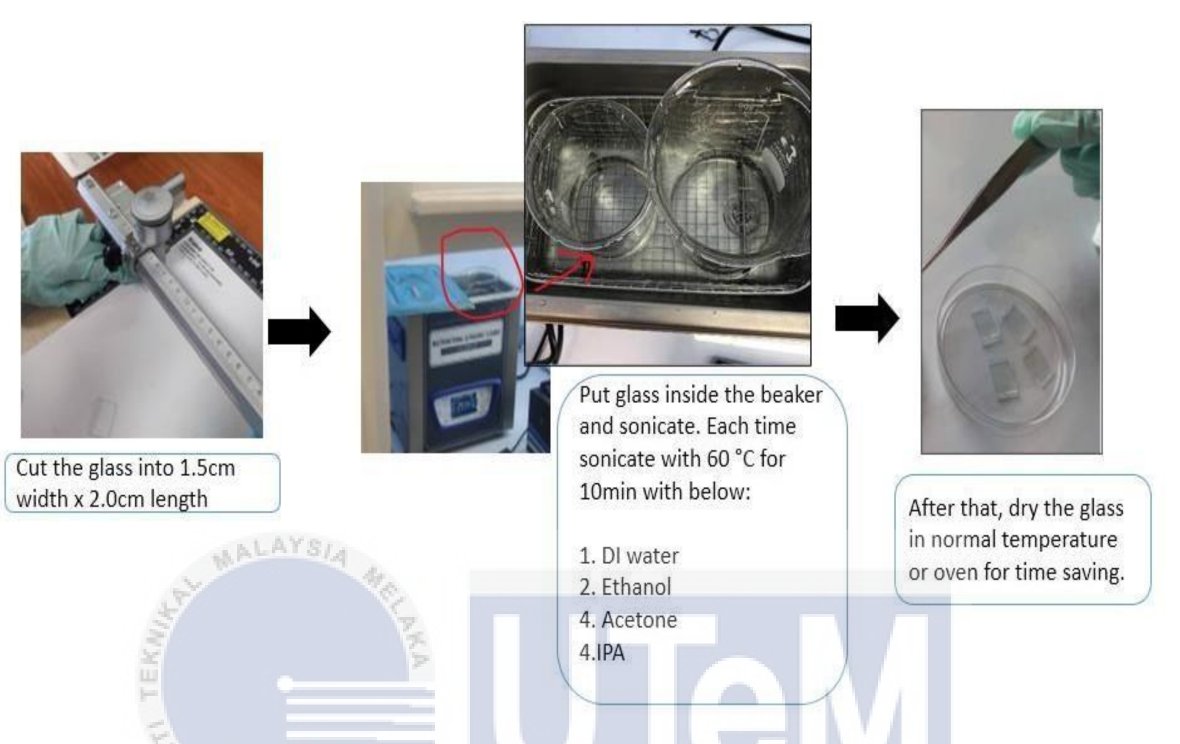
Submission and Evaluation																																							X	

Figure 3.1: Gantt chart

3.2 Glass Preparation

To begin, use a glass cutting tool to cut the glass into 2cm × 1.5cm glass (W x H). Wash the glasses with soap. Repeat this step until the glasses are washed 5 times or sonicate with DI water for 10 min with 60 °C. Then glasses are then fully immersed in ethanol. Put the beaker in Ultrasonic cleaner for 10 minutes a. After 10 minutes, drain the leftover ethanol and fill the beaker with IPA. The glasses should then be sonicated for 10 minutes before being rinsed with distilled water. Remove the glasses from the beaker and place them on the petri dish. For drying the glasses, place them over the dry oven for 10 minutes. Figure 3.2 shows the method to get clean glass.



Cut the glass into 1.5cm width x 2.0cm length

Put glass inside the beaker and sonicate. Each time sonicate with 60 °C for 10min with below:

1. DI water
2. Ethanol
4. Acetone
4.IPA

After that, dry the glass in normal temperature or oven for time saving.

Figure 3.2: Glass preparation

3.3 Synthesis using Sol Gel with Spin Coating Method

The sol-gel method is a chemical technique that produces solid materials, particularly metal oxides, from small molecules. The other technique also used spin coating. In this work, creating a thin film of NiO uses the sol gel with spin coating method. It is common practice to obtain uniform nanometers to a few micrometers of thin films using centrifugal gravity and solvent evaporation of solvent coated thin films. Sol gel spin coating is a low-cost and easy method to prepare on thin film. The result is a relatively uniform thin film of a specific thickness.

The process starts with turning the solution into “sol”, a colloidal solution that normally is dissolved in water or alcohol. Next this sol will transform into a gel form that consists of both a liquid and a solid. Then, the solvent in the gel is removed through a drying process like thermal treatment or firing process. Finally, the sol-gel is done, and this method can be used in the deposit NiO and Cu-doped NiO on thin firm glass. In Figure 3.3 shows how sol gel procedure prepared and in Figure 3.4 the spin shows a step that involves deposition, spinning, and evaporation/drying.

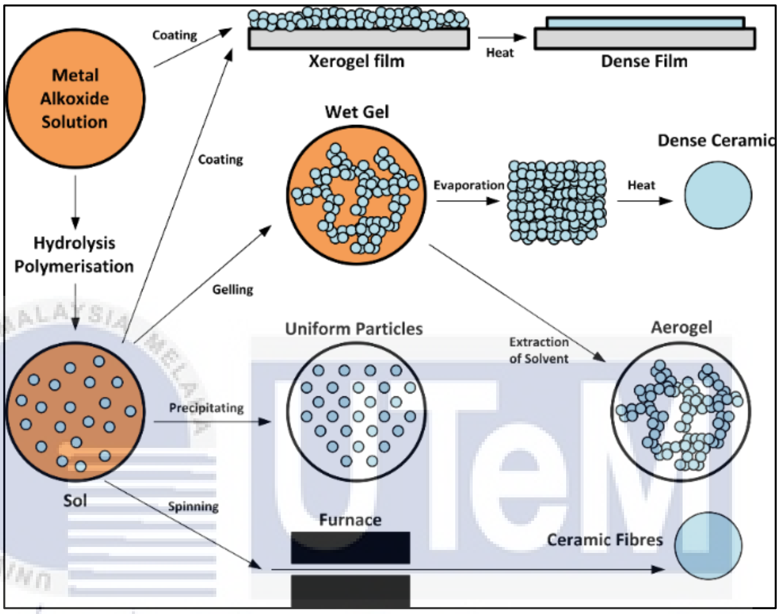


Figure 3.3: How to form a sol gel

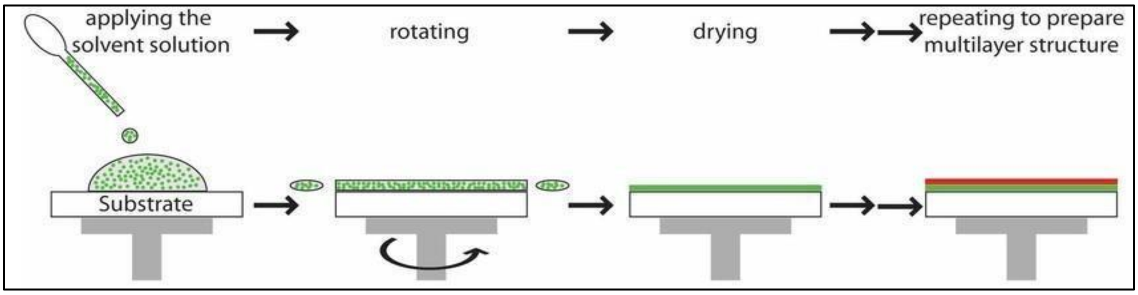


Figure 3.4: The procedure of the spin coating

3.3.1 Pure NiO preparation using spin coating sol gel. [39]

The pH meter must be calibrated before preparing the nickel oxide precursor solution. Put the pH electrode in buffer solutions of pH7, then follow by pH4 and pH10. Check that the slope efficiency is more than 92%. To make the nickel oxide precursor solution, weigh the nickel (II) acetate ($C_4H_6NiO_4$) and KOH in 0.62g and 0.56g respectively. Then prepare two solutions, which are KOH solution and $C_4H_6NiO_4$ solution. For KOH solution, add 0.56g KOH to 100ml distilled water. Stir the mixture at 220rpm in 30°C by using hotplate stirrer until KOH is dissolved. For $C_4H_6NiO_4$ solution, add 15ml of ethanol and 10ml of IPA with 0.62g of nickel acetate. Stir the solution until dissolved at 210rpm in 25-26°C by using hotplate stirrer.

The initial pH for this solution is pH 6. Slowly add the KOH solution by using pipette to increase the pH value to pH11. Then, raise the temperature of the hotplate stirrer to 60°C. Check the pH again after reaching 60°C. If the pH lowers, add the KOH solution again and wait 10 minutes before testing the pH. Stir the NiOx solution for 2 hours if the pH is consistently at pH11. Rest the solution for 1 day after 2 hours.

After 1 day rest, the solution will separate into two densities. Then, wash with 10ml ethanol 5 times. Each time the washing process needs to wait until there is a layer separation. Then use syringe to take out the top layer and transfer the top layer to new beaker. Refrigerate the solutions after wrapping them with parafilm. Store the gel in a tube also for better spacing.

To begin the NiO deposition process, sonicate the NiO solutions for 15 minutes. Then, drop the solution onto the glass and spin coat for 30 seconds at 3000rpm. Then, dry oven the glass for 5minutes at 150°C before repeating the dropping and drying process 5 times more. Then to anneal the NiO film, set the temperature at 500°C in

30min. Summary of the process is shown in Figure 3.5 and for detailed procedure for each part is in Figure 3.6, 3.7, 3.8.

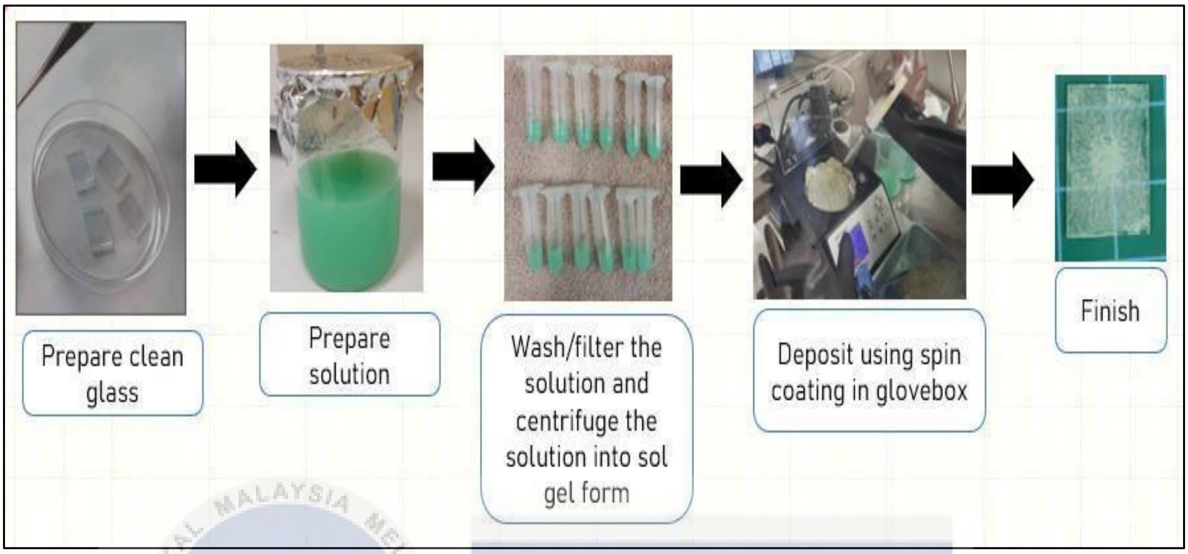
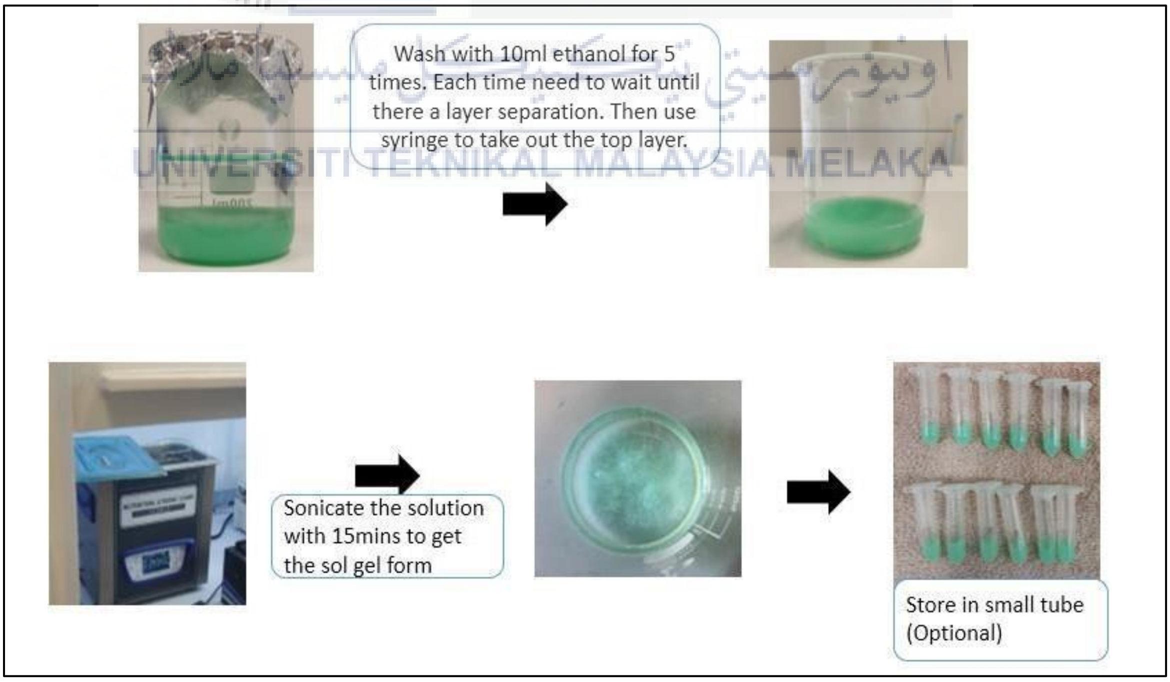


Figure 3.5: Summary NiO using sol gel spin coating method.



Wash with 10ml ethanol for 5 times. Each time need to wait until there a layer separation. Then use syringe to take out the top layer.

Sonicate the solution with 15mins to get the sol gel form

Store in small tube (Optional)

Figure 3.6: Prepare the NiO solution.

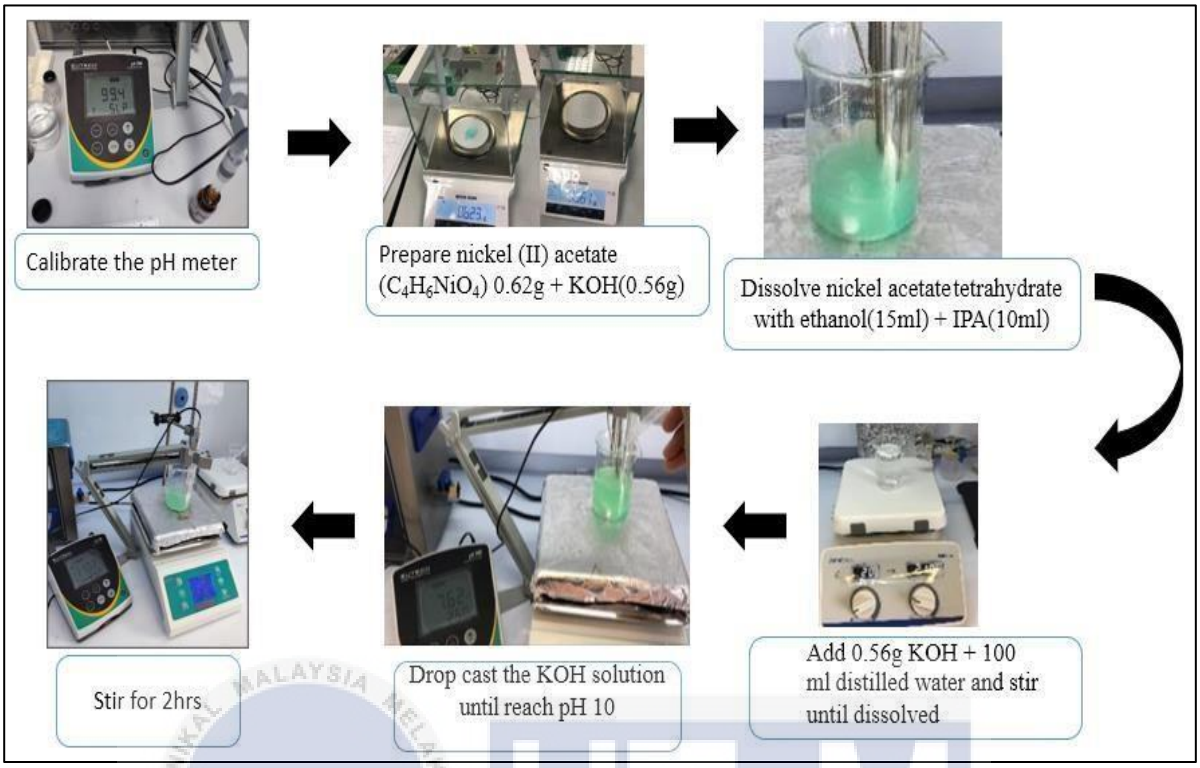


Figure 3.7: Process of preparing the NiO solution.

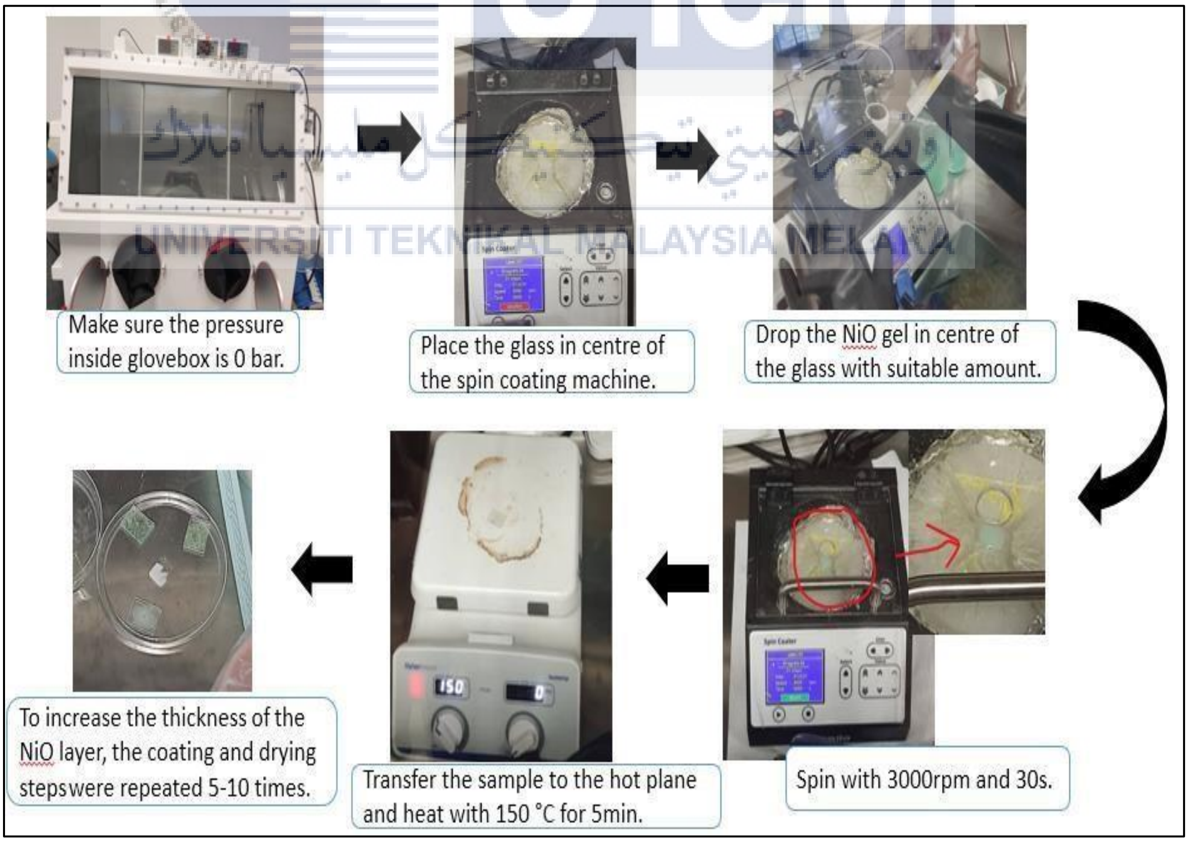


Figure 3.8: NiO deposition with spin coating

3.3.2 Cu doped NiO preparation using sol gel method. [40]

The pH meter must be calibrated before preparing the nickel oxide doped copper precursor solution. Put the pH electrode in buffer solutions of pH7, then follow by pH4 and pH10. Check that the slope efficiency is more than 92%. To make the NiO doped copper precursor solution, weigh the $NiCl_2 \cdot 6H_2O$ and $CuSO_4 \cdot 5H_2O$ with 2.37g and 0.071g respectively. For NaOH solution, normally it depends on how many moles been used. Example 0.1M is used, the molar mass of NaOH is 40g/mol, so add the 0.4g NaOH with 100ml distilled water to get 0.1M. Stir the mixture at 220rpm in 30°C by using hotplate stirrer until NaOH is dissolved.

Th initial pH for this solution is around pH 6. Slowly add the NaOH solution by using pipette to increase the pH value to pH10. Then, raise the temperature of the hotplate stirrer to 60°C. Check the pH again after reaching 60°C. If the pH lowers, add the NaOH solution again and wait 10 minutes before testing the pH. Stir the NiO-Cu solution for 2 hours if the pH is consistently at pH10. Rest the solution 1 day before used.

After 1 day rest, the solution will separate into two densities. Wash with 10ml DI water at least 5 times to get clear NiO-Cu base. Each time the washing process needs to wait until there is a layer separation. Then use syringe to take out the top layer and transfer the top layer to new beaker. Refrigerate the solutions after wrapping them with parafilm. To begin the NiO-Cu deposition process, sonicate the NiO-Cu solutions for 15 minutes. Then, drop the solution onto the glass and spin coat for 30 seconds at 3000rpm. Then, dry oven the glass for 5 minutes at 150°C and repeat the dropping and drying process 5 to 10 times to get suitable thickness. Summary of the process is shown in Figure 3.9 and for detailed procedure for each part is in Figure 3.10, 3.11, 3.12.

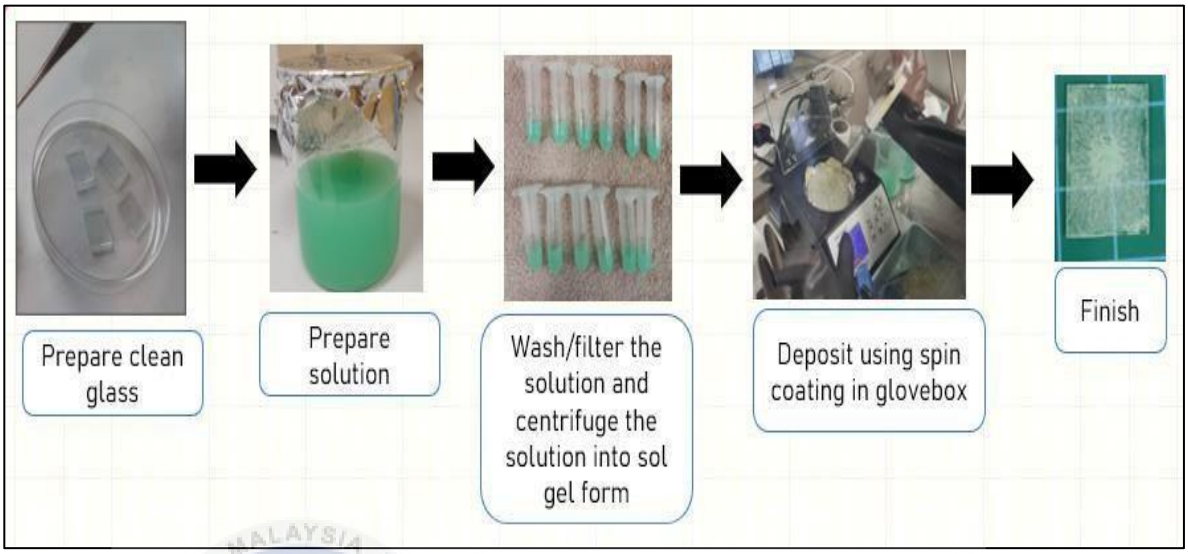


Figure 3.9:Summary NiO:Cu using spin coating method

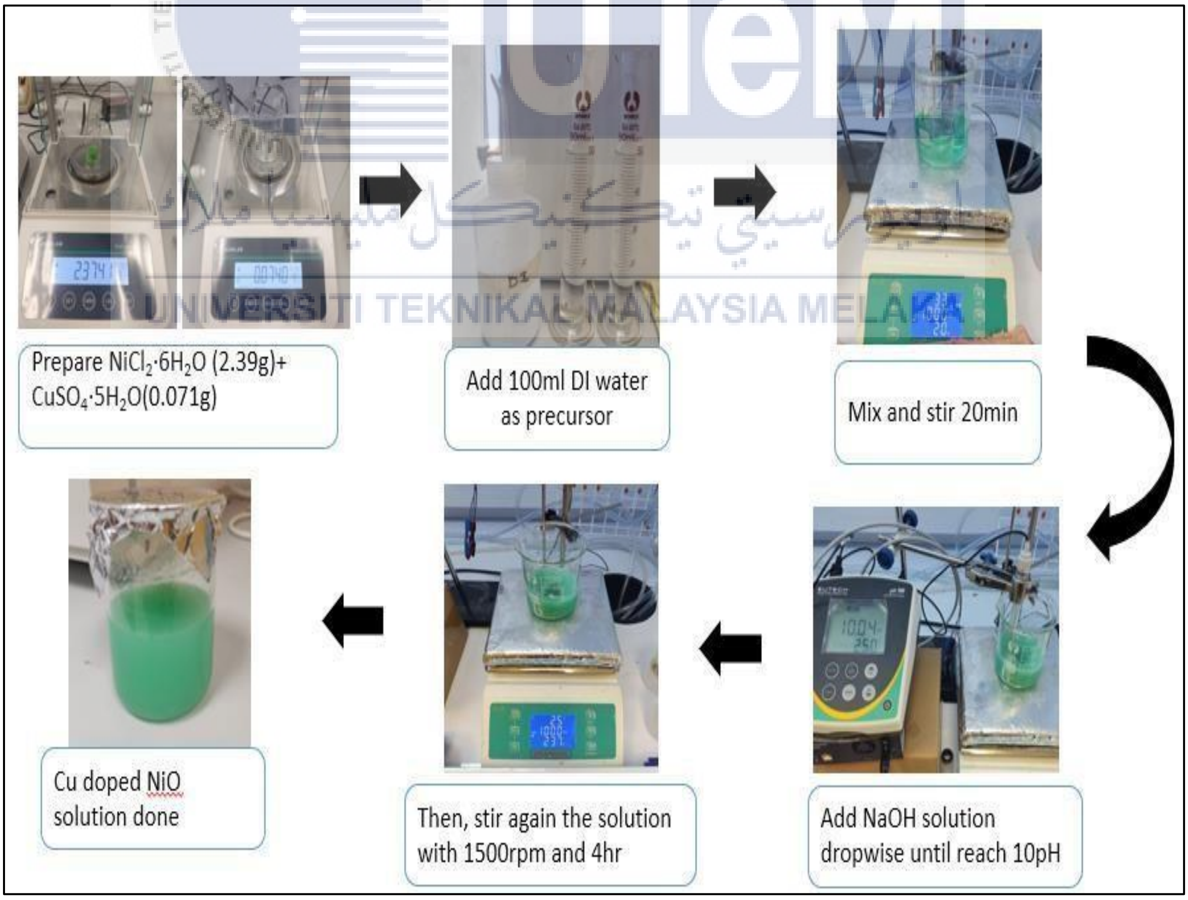
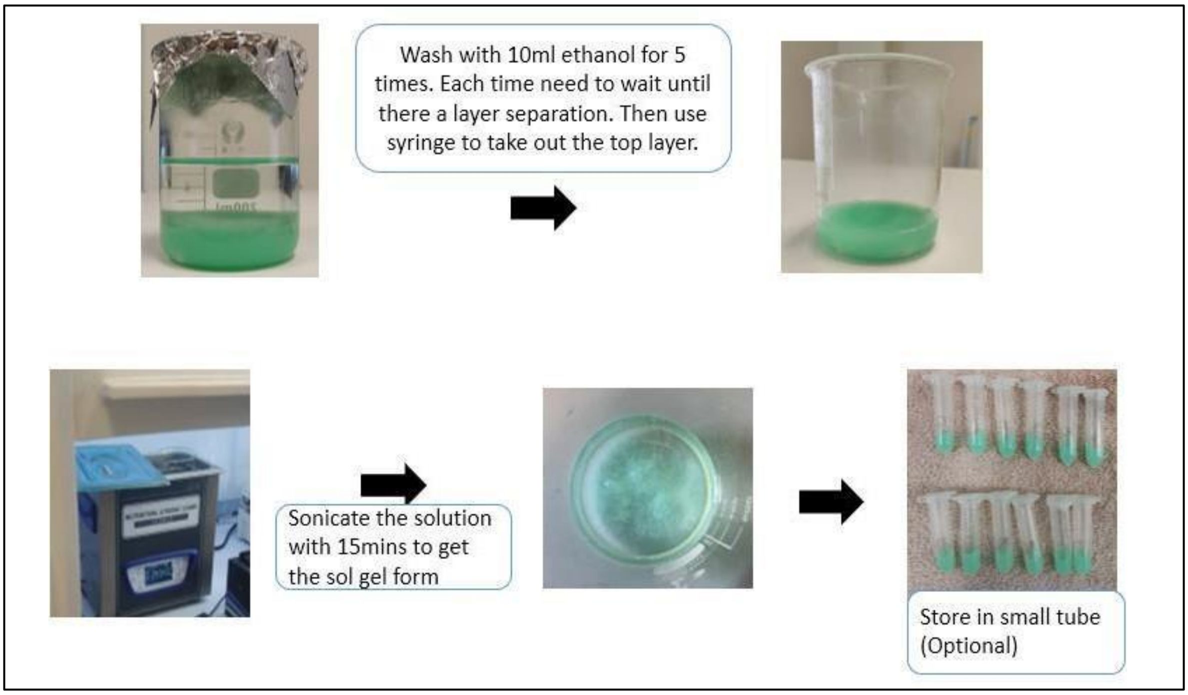


Figure 3.10: Prepare Cu dope NiO solution.

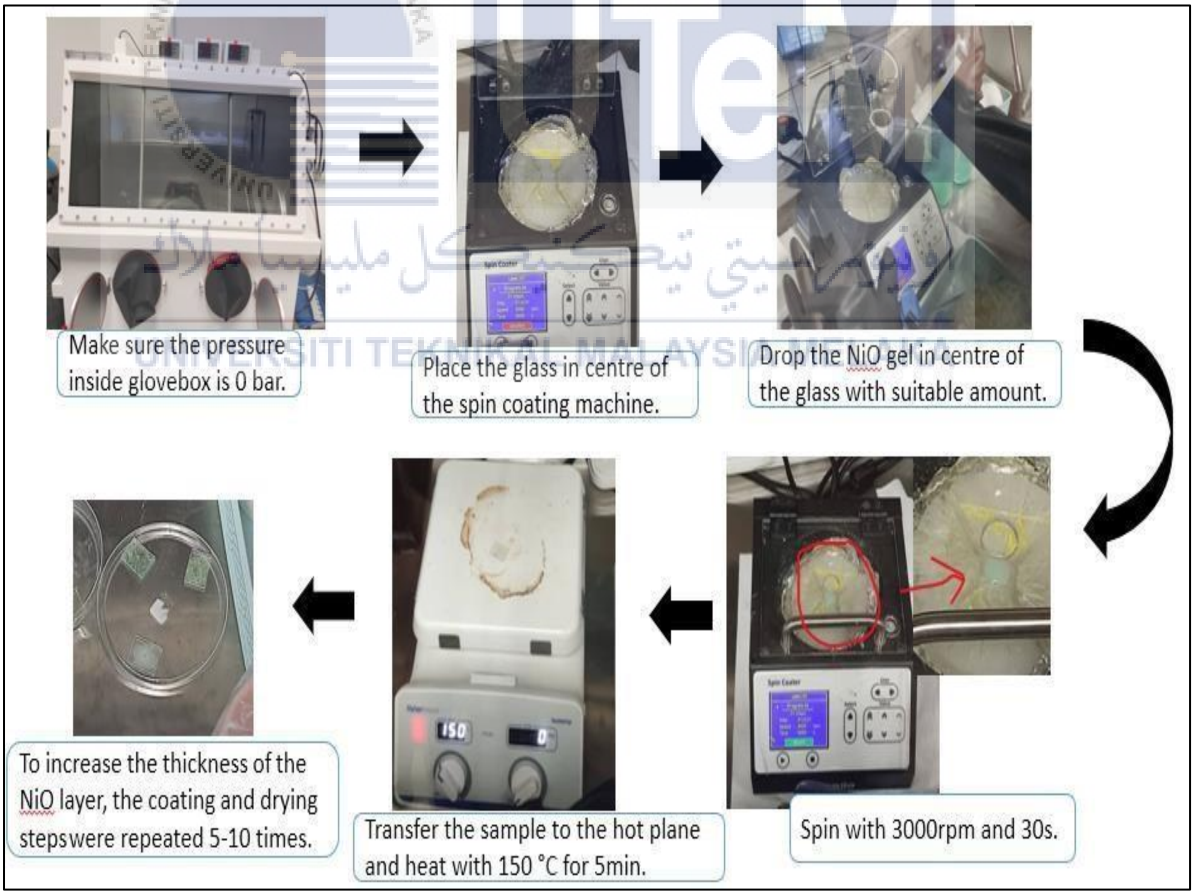


Wash with 10ml ethanol for 5 times. Each time need to wait until there a layer separation. Then use syringe to take out the top layer.

Sonicate the solution with 15mins to get the sol gel form

Store in small tube (Optional)

Figure 3.11: Process of preparing the NiO solution



Make sure the pressure inside glovebox is 0 bar.

Place the glass in centre of the spin coating machine.

Drop the NiO gel in centre of the glass with suitable amount.

Spin with 3000rpm and 30s.

Transfer the sample to the hot plane and heat with 150 °C for 5min.

To increase the thickness of the NiO layer, the coating and drying stepswere repeated 5-10 times.

Figure 3.12: NiO:Cu deposition with spin coating

3.4 Synthesis using Electrodeposition.

Electrodeposition is a technique utilized in the field of analytical chemistry for the analysis of metal ions. The standard procedure for electrodeposition is normally contains of use of an electrode, which commonly are platinum. However, used of platinum as electrode are too expensive, and it is very rare and precious metals, so in this experiment the platinum is change graphite because graphite is very good alternative material for platinum due to economically.

When using electrodeposition method to deposit the setup for the process is quite complex compared to other method because it requires a carefully controlled setup, including an electrolyte solution, power supply, and specific conditions such as temperature and pH. Although there are some challenges when preparing but the deposition rates are very high as they are controlled by the applied current density, which also allows to produce thicker films. However, due to sensitive temperature, during the deposition the deposition rate can affect on morphology, and properties of the deposited material. On the other hand, the electrodeposition results will having a more uniform film surface.

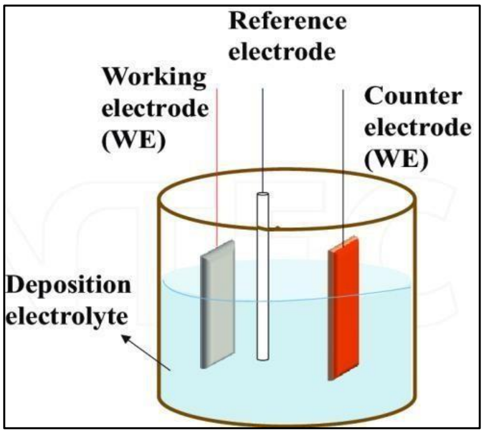


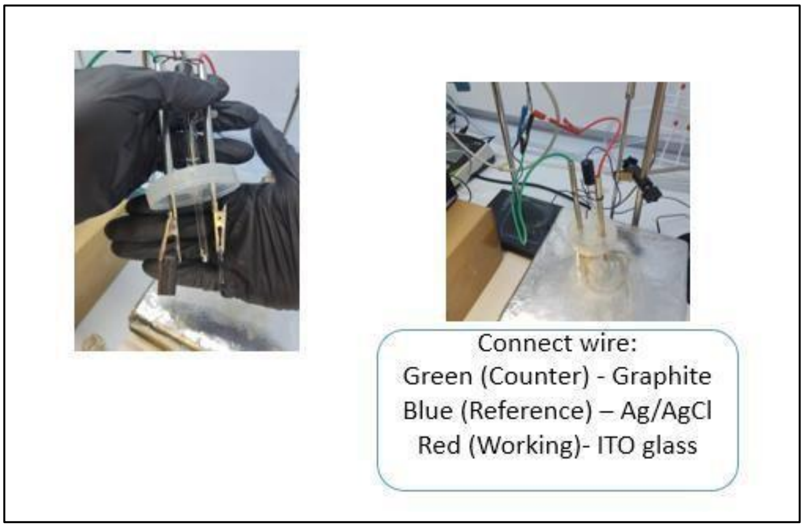
Figure 3.13: Electrodeposition process

3.4.1 Pure NiO preparation using Electrodeposition [41].

The pH meter must be calibrated before preparing the NiO precursor solution. Put the pH electrode in buffer solutions of pH 7, then follow by pH4 and pH10. Check that the slope efficiency is more than 92%. To make the NiO precursor solution, weigh the $NiCl_2\cdot 6H_2O$ and KCI in 3.24g and 0.37g respectively. Then, prepare KOH solutions. For KOH solution, normally it depends on how many moles been used. Example 0.1M is used, the molar mass of KOH is 56.1g/mol, so add the 0.561g KOH with 100ml distilled water to get 0.1M.

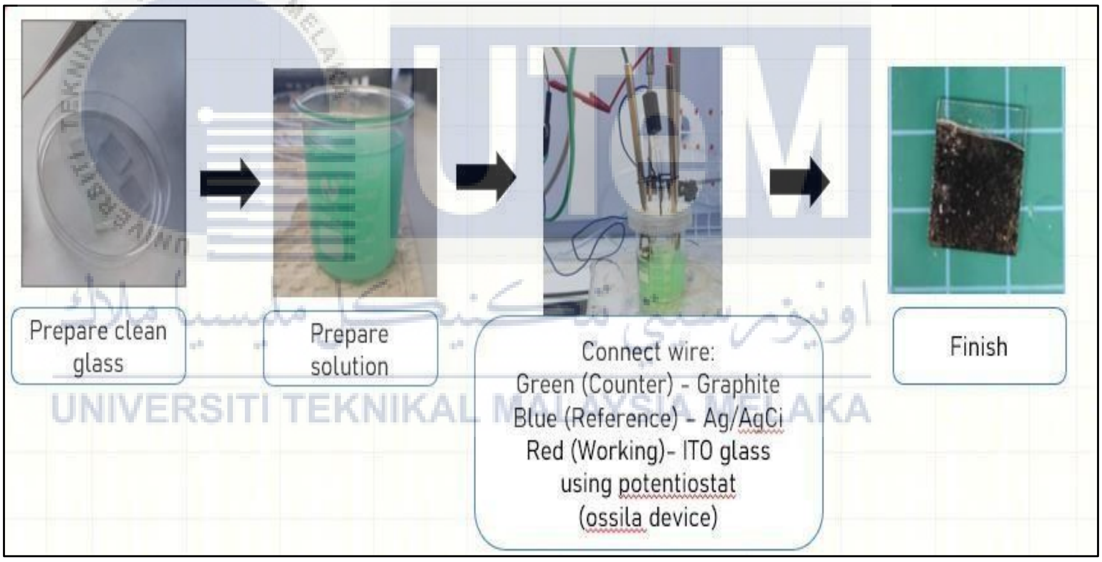
Next, stir the mixture at 220rpm in 30°C by using hotplate stirrer until KOH is dissolved. Then, stir the $NiCl_2\cdot 6H_2O$ and KC solution until dissolved at 210rpm in 25-26°C by using hotplate stirrer. The initial pH for this solution is pH6. Slowly add the KOH solution by using pipette to increase the pH value to pH8. Then, raise the temperature of the hotplate stirrer to 60°C. Check the pH again after reaching 60°C. If the pH lowers, add the KOH solution again and wait 10 minutes before testing the pH. Stir the solution for 2 hours if the pH is consistently at pH8. Rest the solution for 1 day before use.

To begin the NiO electrodeposition process, prepare graphite, ITO glass and Ag/AgCl. Double check the ITO glass with multimeter to make sure the conductive face inside. Then start connecting all wires as show in Figure 3.14. In this electrodeposition, NiO films were applied to substrates made of ITO-coated glass. A graphite rod and Ag/AgCl were utilized as the counter electrode and reference electrode, respectively. The NiO thin films were deposited at room temperature for 30 minutes under a potentiostatic condition. The summary procedure is in Figure 3.15 and for the entire procedure is illustrated in Figure 3.16, 3.17.



Connect wire:
Green (Counter) - Graphite
Blue (Reference) – Ag/AgCl
Red (Working)- ITO glass

Figure 3.14: Wire Connection for electrodeposition



Prepare clean glass

Prepare solution

Connect wire:
Green (Counter) - Graphite
Blue (Reference) - Ag/AgCi
Red (Working)- ITO glass
using potentiostat
(ossila device)

Finish

Figure 3.15: Summary NiO using electrodeposition.

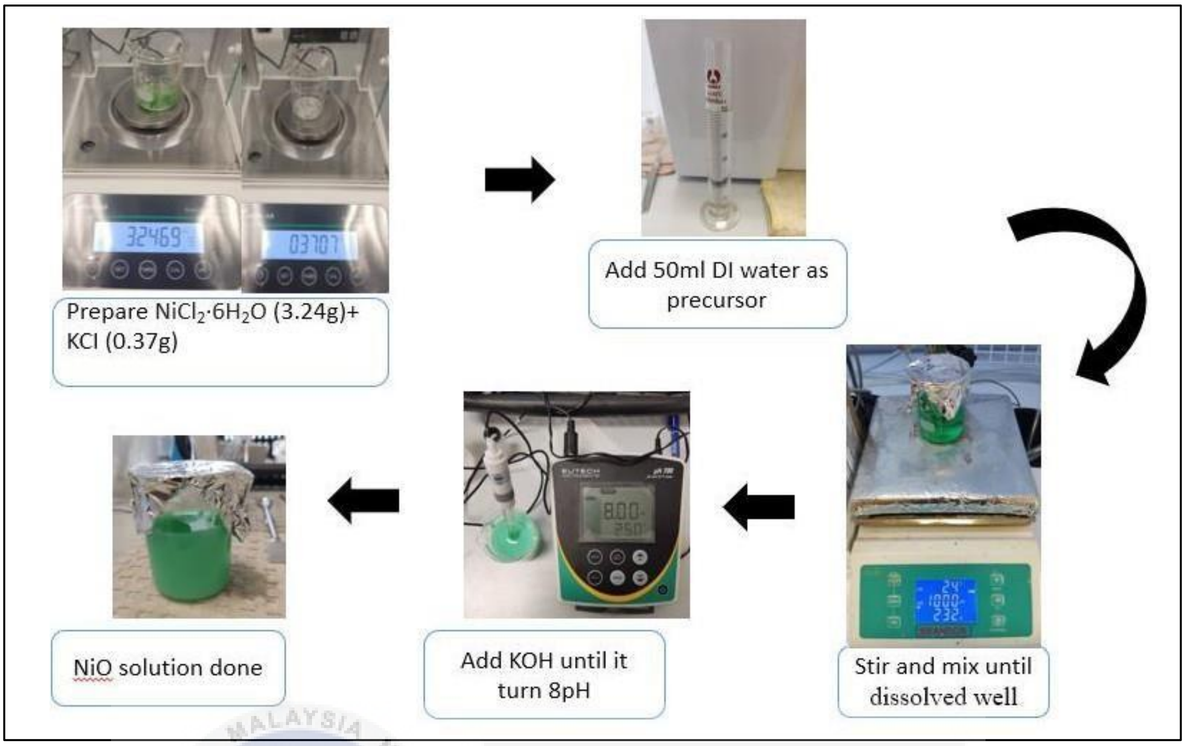


Figure 3.16: Pure NiO solution process

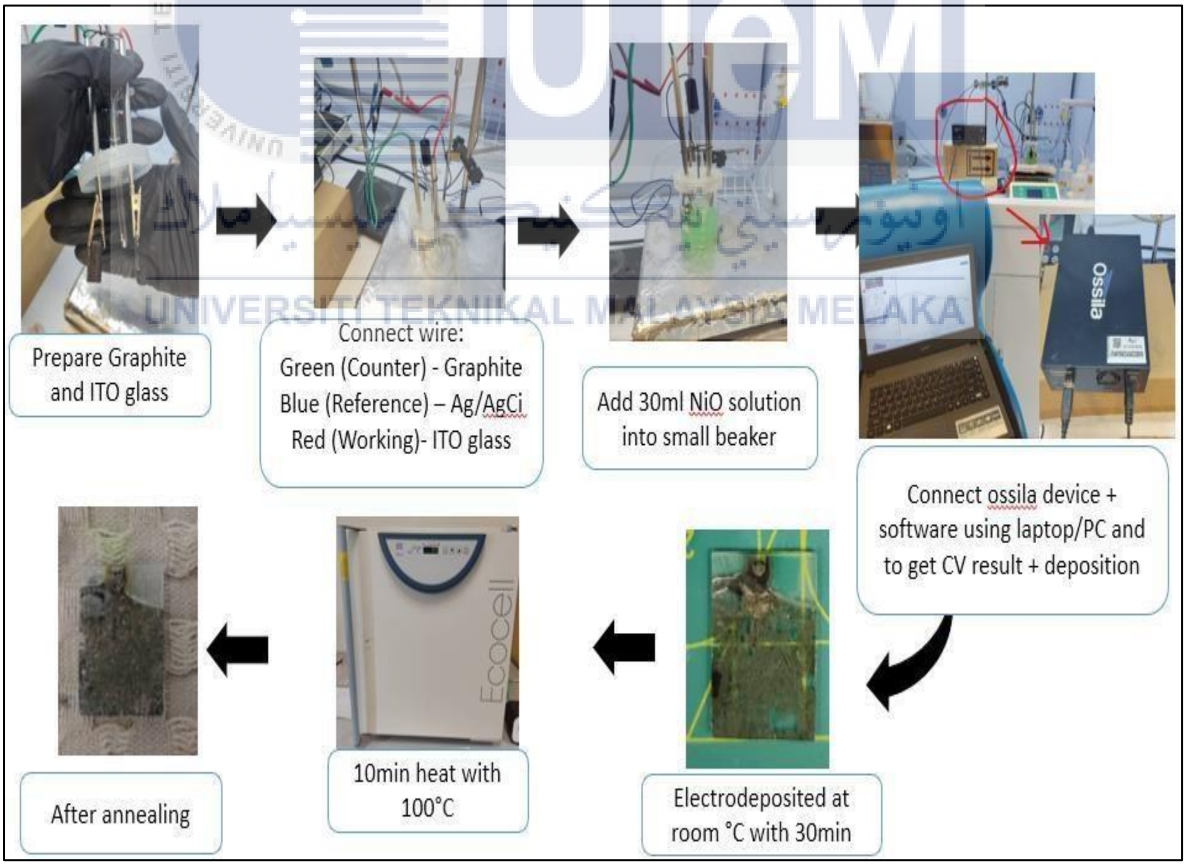


Figure 3.17: NiO deposition with electrodeposition

3.4.2 Cu doped NiO preparation using electrodeposition. [42]

The pH meter must be calibrated before preparing the NiO precursor solution. Put the pH electrode in buffer solutions of pH7, then follow by pH4 and pH10. Check that the slope efficiency is more than 92%. To make the NiO precursor solution, weigh the $NiCl_2 \cdot 6H_2O$ and KCI in 3.24g and 0.37g respectively. Then, prepare KOH solutions. For KOH solution, normally it depends on how many moles been used. Example 0.1M is used, the molar mass of KOH is 56.1g/mol, so add the 0.561g KOH with 100ml distilled water to get 0.1M. Next, stir the mixture at 220rpm in 30°C by using hotplate stirrer until KOH is dissolved. Then, stir the $NiCl_2 \cdot 6H_2O$ and KC solution until dissolved at 210rpm in 25-26°C by using hotplate stirrer.

The initial pH for this solution is pH6. Slowly add the KOH solution by using pipette to increase the pH value to pH 8. Then, raise the temperature of the hotplate stirrer to 60°C. Check the pH again after reaching 60°C. If the pH lowers, add the KOH solution again and wait 10 minutes before testing the pH. Stir the solution for 2 hours if the pH is consistently at pH8. Rest the solution for 1 day before use. To begin the NiO electrodeposition process, prepare graphite, ITO glass and Ag/AgCl. Double check the ITO glass with multimeter to make sure the conductive face inside.

In this electrodeposition, NiO films were applied to substrates made of ITO-coated glass. Then start connecting all wires as shown in Figure 3.14. A graphite rod and Ag/AgCl were utilized as the counter electrode and reference electrode, respectively. The NiO thin films were deposited at room temperature for 30 minutes under a potentiostatic condition. The summary procedure is in Figure 3.15 and for the entire procedure is illustrated in Figure 3.19 and 3.20.

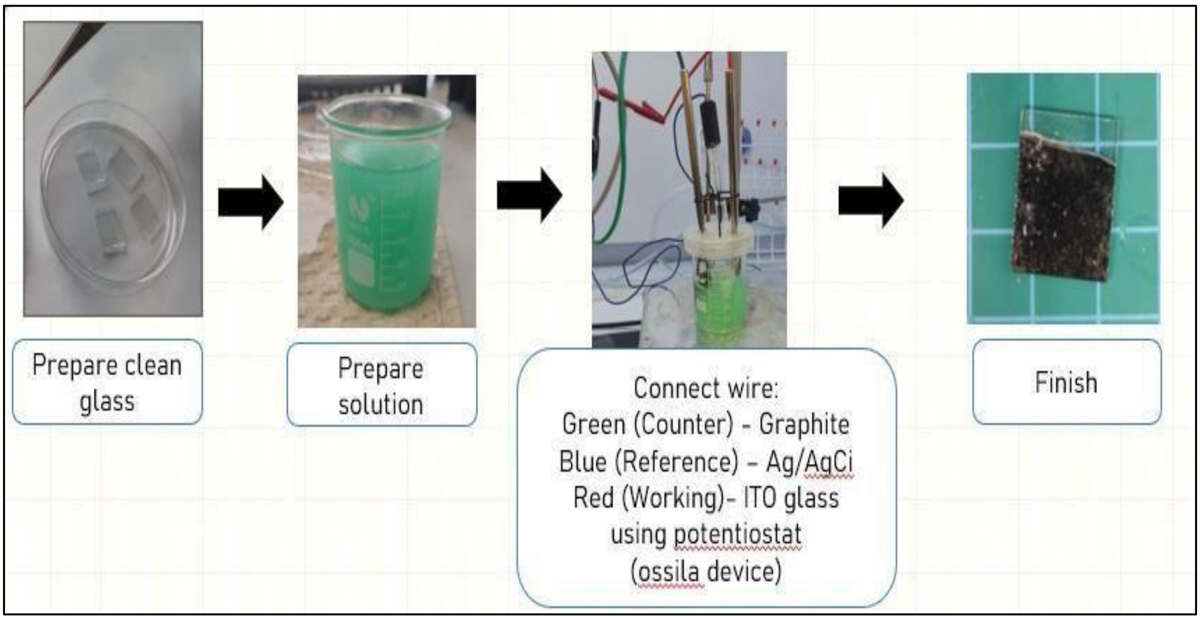


Figure 3.18: Summary of NiO:Cu using electrodeposition

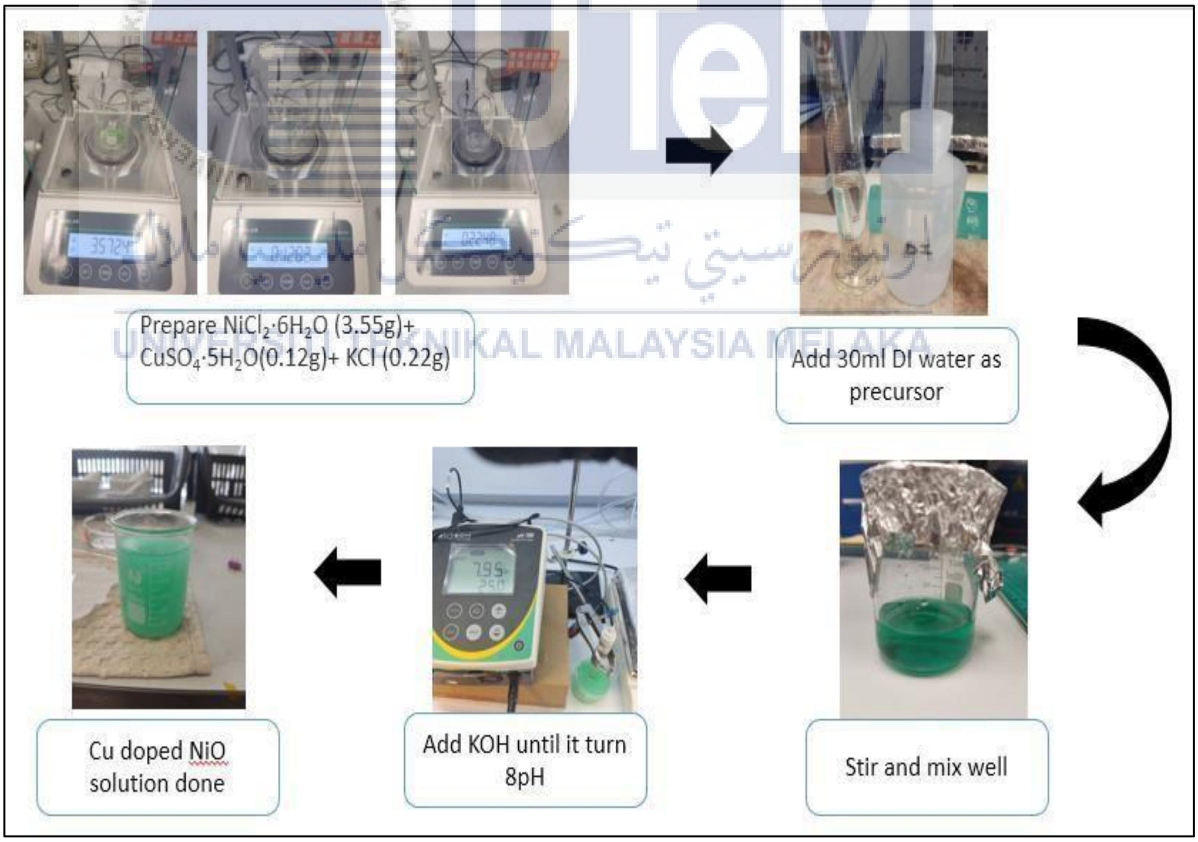


Figure 3.19: NiO:Cu solution process

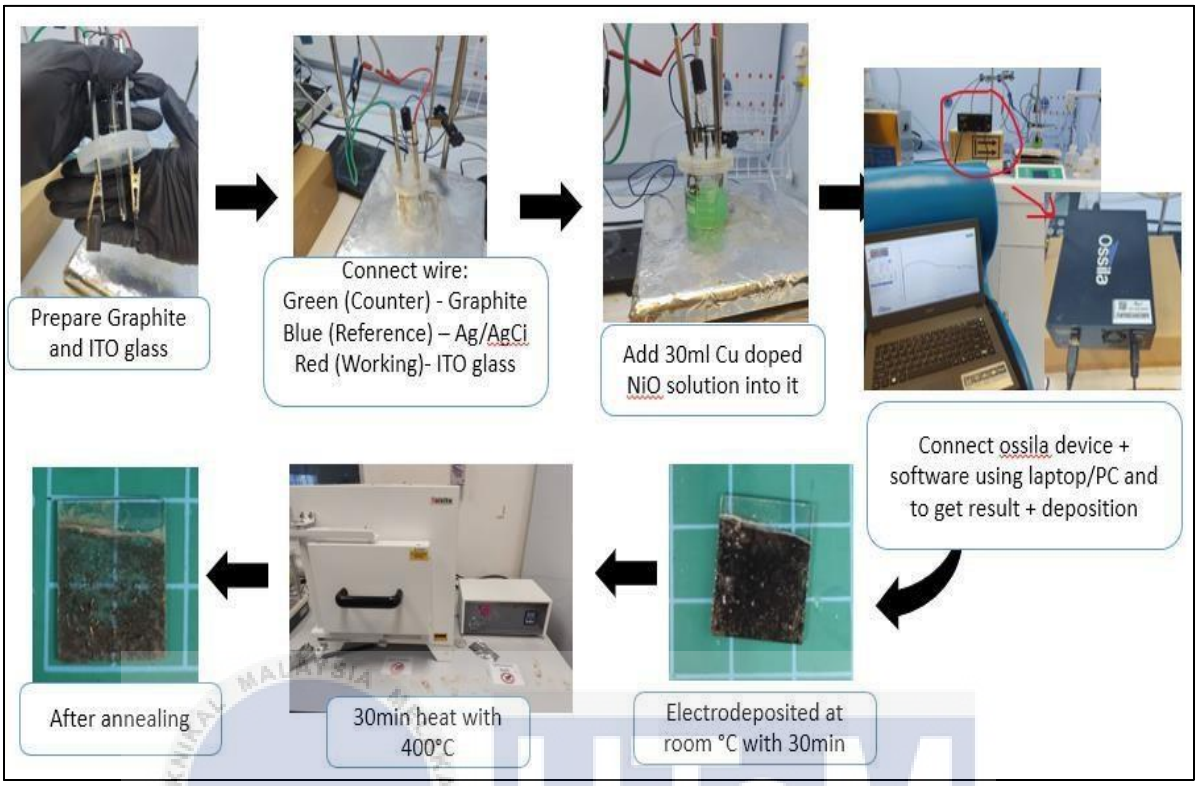


Figure 3.20: NiO:Cu deposition with electrodeposition

3.5 Summary

In the summary of the experiment for the sol gel spin coating and electrodeposition method are being studied and involved for depositing materials. First, sol gel spin coating is normally making a sol from solution and deposit on a glass and spin with suitable rpm speed on it to create a thin film. This method is simple but has a lower deposition rate and it produces very thin films when spinning at high rpm, it is also not sensitive to temperature during sol gel form or spinning. However, the surface may not be non-uniform film because over rpm speed spin or the quantity of sol gel are not enough during drop on glass. On the other hand, electrodeposition is an electrochemical process that is deposited onto a conductive substrate through an electric field. However, electrodeposition method may very complex to setup if without proper equipment and it is sensitive to temperature, sometime when start deposition the solution needs a certain temperature to deposit the layer but the deposition rates great and will have very uniform firm surface and can produce a very thick layer. The choice between these two methods depends on the specific requirements of the task at hand. Table 3.1 shows the comparison between sol gel spin coating vs electrodeposition method.

Table 3.1: Sol gel spin coating vs electrodeposition method

Method	**Sol-Gel Spin Coating**	**Electrodeposition**
Process	A sol is deposited on a firm, followed by spin to create film.	Material deposited via electric field
Setup	Simple	Complex
Deposition Rates	Lower	Higher
Film Thickness	Very thin	Thick
Temperature	Not sensitive	Sensitive
Film surface	Not uniform	Uniform

CHAPTER 4

RESULTS AND DISCUSSION

In this chapter, results and discussion have been discusses on how to synthesis, deposition, and fabrication result for pure NiO and NiO doped copper as hole transport layer in inverted perovskite solar cell. The discussion continues with the characterization of the thin film NiO and Cu-NiO on a glass substrate using UV-Vis, SEM Cyclic voltammetry (CV).

4.1 Deposit Sample

In this chapter a few samples have been deposited on normal thin firm glass and ITO/FTO. For sol gel spin coating method mostly are fabricated on normal glass only. However, for electrodeposition method it must fabricate on ITO/FTO glass because only ITO/FTO glass is able to bear on acidic environment. Next, both samples are classified into non-anneal and anneal status and is shown in Figure 4.1.2. Finally, there are a few failed samples also shown Figure 4.1.3.

There are seven example samples that been deposited and shown in Table 4.1 below. By using sol gel spin coating method, which is No 1, No 2, No 3, No 4, and No 5, No 6 are using electrodeposition method. From the observation by using the sol gel spin coating method deposit NiO are normally light green and slightly transparent meanwhile NiO:Cu has a light blue mix with NiO because the copper material been used are copper sulfate, copper sulfate are blue solid material. For electrodeposition method, since it is applied with current the sample will burn in the solution during deposition so the colour normally turn to dark colour. So, in No 5, pure NiO become drak green and in No 6, NiO dope copper become dark brown.

At the same time, the non-anneal and annealed sample are also shown in Figure 4.2. Finally, there are few examples failed sample be in view in Table 4.3.

Table 4.1: Example sample deposited.

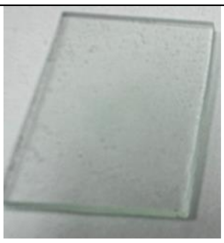
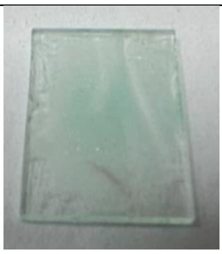
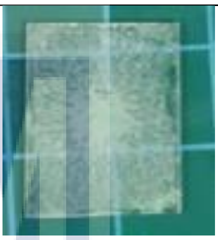
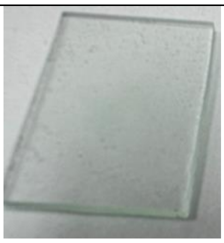
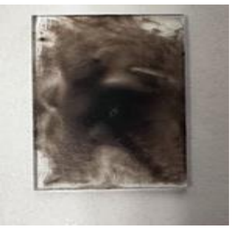
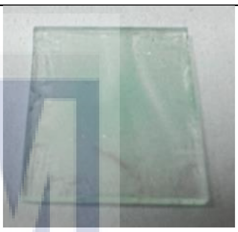
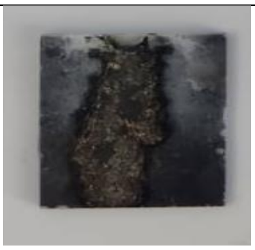
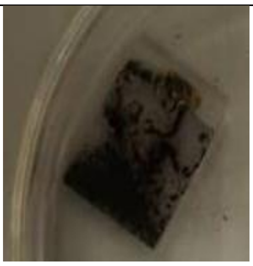
No	Method	Description	Sample
1	Sol gel spin coating	Pure NiO pH 11	
2	Sol gel spin coating	NiO:Cu (0.71g) pH 10	
3	Sol gel spin coating	NiO:Cu (0.071g) pH 10	
4	Sol gel spin coating	NiO:Cu (0.071g) pH 11	
5	Electrodeposition	Pure NiO pH 8	
6	Electrodeposition	NiO:Cu pH 8	

Table 4.2: Non anneal and Annealed.

No	Description	Anneal	Sample
1	Pure NiO pH 11 using sol gel spin coating	No	
		Yes	
2	NiO:Cu (0.71g) pH 10 with sol gel spin coating	No	
		Yes	
3	Pure NiO with electrodeposition	No	
		Yes	

4	NiO:Cu (0.71g) pH 8 with electrodeposition	No	
		Yes	

Table 4.3: Example failed sample.

No	Cause	Due	Sample
1	Not enough electrodeposition time (30s)	Not fully deposit	
2	Not enough electrodeposition time. (5min)	Not fully deposit	
3	Over annealing temperature 700°C with 30min	Crack and not uniform	
4	Over annealing temperature 600°C with 10min	Not uniform	
5	Over annealing temperature 500°C with 10min	Not uniform	

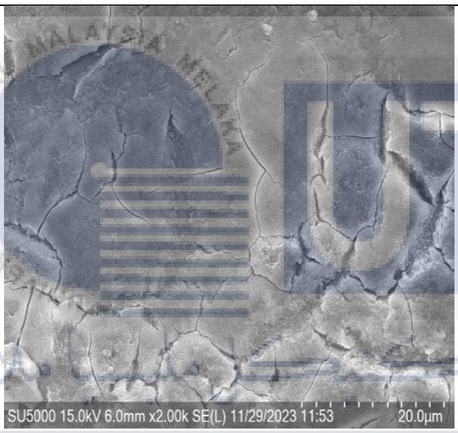
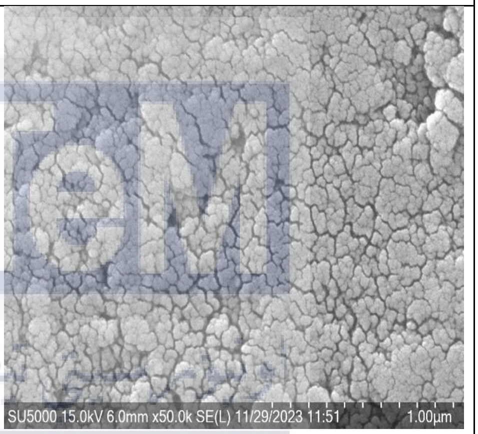
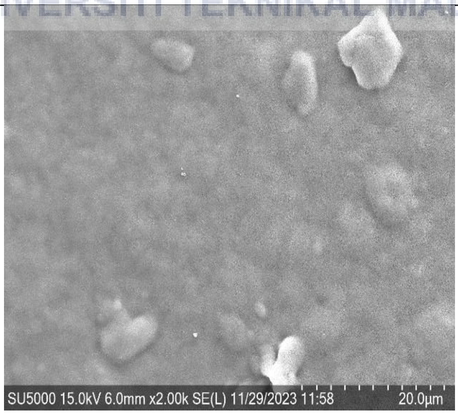
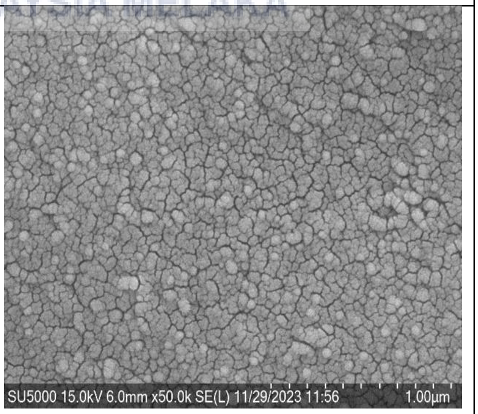
4.2 Scanning Electron Microscopes (SEM) analysis

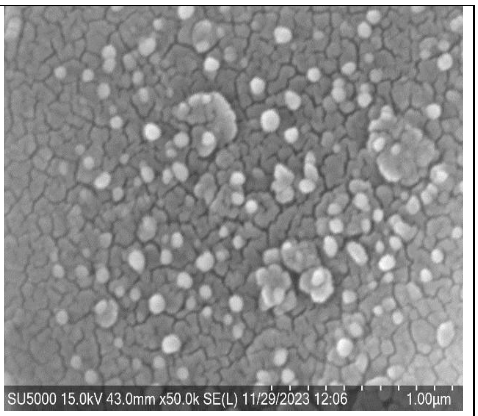
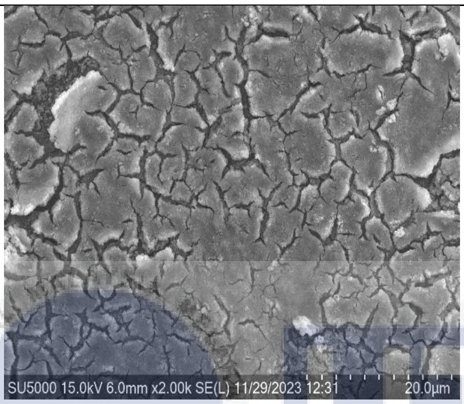
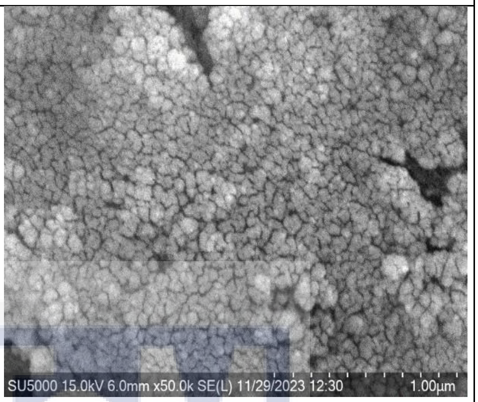
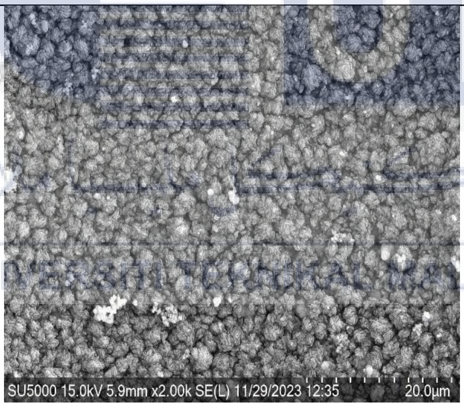
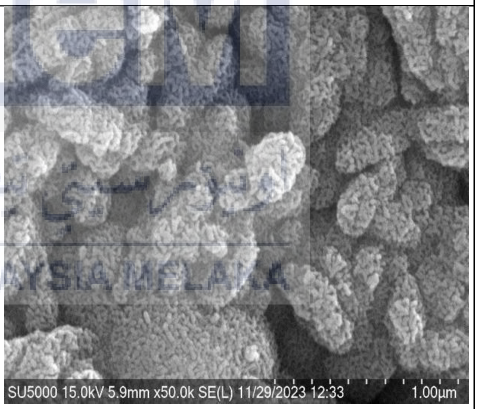
Scanning Electron Microscopy (SEM) is used to utilize an electron beam and scan the sample, this resulting in magnified image. During SEM characteristic, one of the applications called ImageJ software can be used to calculate the average particle size from a SEM picture. However, the minimum scale bar of the SEM picture is 1μm, and this scale still does not clearly display the sample that has been deposit particle. Hence, based on the SEM images in Table 4.4, the particle size in NiO-1 and NiO-2 are kind of similar although NiO-1 uses pH 10 solutions while NiO-2 is using pH-11 solutions. That's mean the difference pH does not affect the particle size so much.

Then for the NiO dope copper sample is tested with different copper weigh which is in NiO+Cu-3 the copper weight 0.071grams is used and for NiO:Cu-4 the copper weight 0.71 grams is used. Hence, based on the SEM results in 50k zoom on NiO:Cu-3 only has a few small white spots on it, this white spot is believed to be a copper material. The 0.071grams copper are dope on NiO on it. However, SEM only can see the surface of the material so further analysis like XRD or EDS can better prove that the copper is really dope with NiO. Meanwhile in NiO:Cu-4 were used 0.7grams of copper that's 10times volume of NiO:Cu-3 copper, so from the 50k zoom SEM there are no having a small white spots only but instead small white sport the white spots are mix with the NiO. So, this proves that the white spot has potential as copper, and it is successfully doped with NiO if too much weight of copper. But too much weight of copper also may affect the performance, durability, and stability of the HTL.

Finally, the NiO:Cu-5 result shown is using electrodeposition method. When synthesis with electrodeposition method can get the better magnified structure image on 50k zoom due to electrodeposition has better deposition rate, firm thickness, and surface than sol gel spin coating method. However, greater thickness also will affect the performance of IPSC. Meanwhile, in 2k zoom also show that there are a few white spots on it, this prove that copper also successfully dope with the NiO. When fabricated IPSC the thinner the layer the better the performance but this proves that electrodeposition method can deposit better on deposition rate, firm thickness, and surface than sol gel spin coating method.

Table 4.4: SEM results

Sample	**2k**	**50k**
NiO-1		
NiO-2		

NiO:Cu -3		
NiO:Cu -4		
NiO:Cu -5		

4.3 Ultraviolet and Visible Absorption Spectroscopy (UV-Vis)

UV-Vis spectroscopy is used to determine the absorption spectrum of a sample. The absorption spectrum was measured by absorbance (a.u) and wavelength (nm). For NiO and NiO:Cu from research paper the UV-Vis has an absorption peak at 300nm for pure NiO. But for NiO:Cu still in studying because of the copper weigh used, crystal structure and etc. But having a higher absorption means that the material is good at absorbing light energy. Besides that, the optical energy band gap (eV) is calculated using Origin software to calculate and plot. The bandgap for NiO is around 3.2eV while NiO dope copper has not specific value yet because it still in research. In the research paper have mentioned that the bandgap for doped NiO will decrease when the doping ratio of copper is increased and the of the change of crystallite sized during doping.

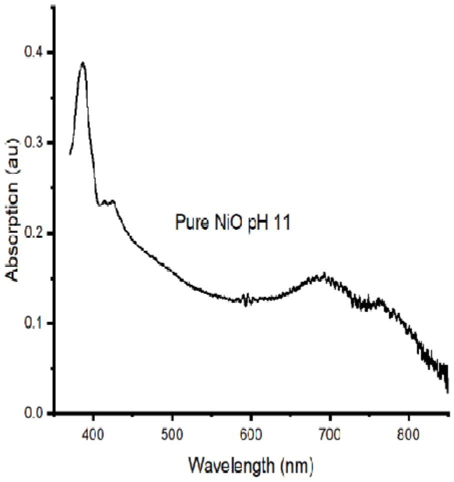
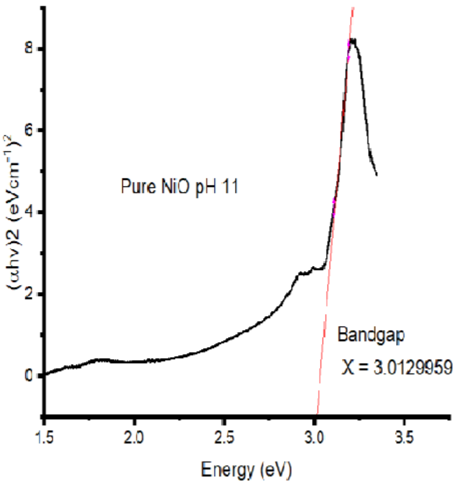
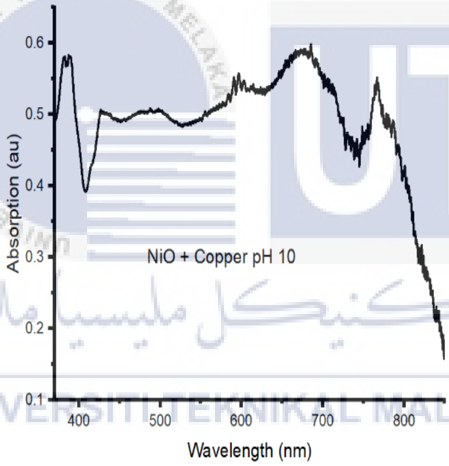
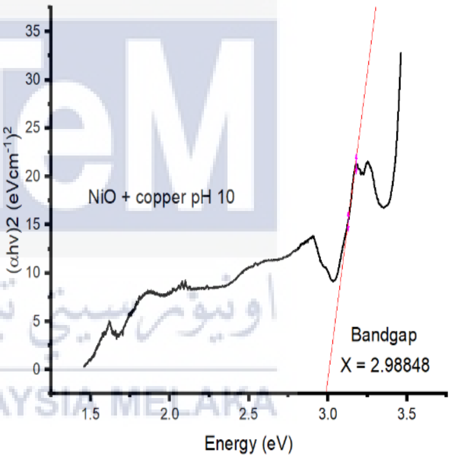
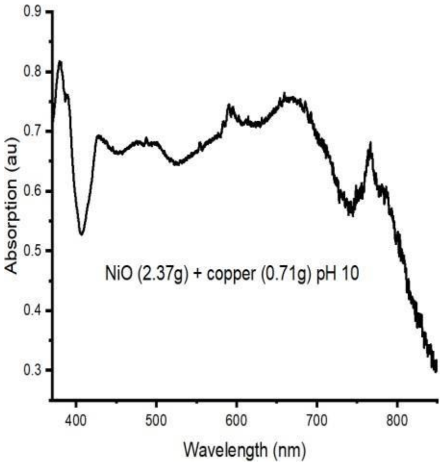
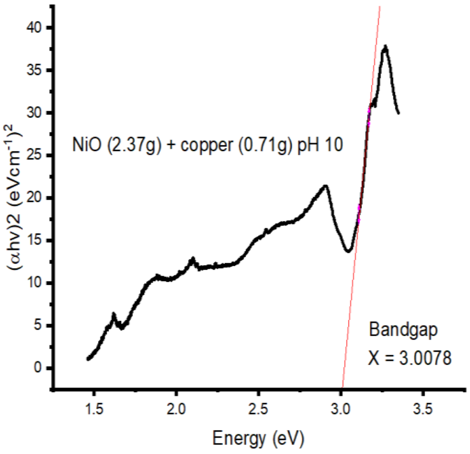
In Table 4.5 NiO-1, NiO:Cu-2, NiO:Cu-3, NiO-4A, NiO:Cu-5A, NiO-6A, NiO:Cu-7A, shows the comparison of the absorption and bandgap of each sample. This table can be separated into 3 part, the first part include NiO-1, NiO:Cu-2, NiO:Cu-3 that are no annealed thin film. From the absorption for each absorption, which is 0.4 au, 0.58 au, 0.82 au, Meanwhile the band gap values were 3.01 eV, 2.99eV, 3.0eV. As the result, the NiO-1 has the lowest absorption but the NiO:Cu-2 and NiO:Cu-3 has better absorption value than NiO-1. This prove that the doped NiO will increase the absorbing light energy compare pure NiO.

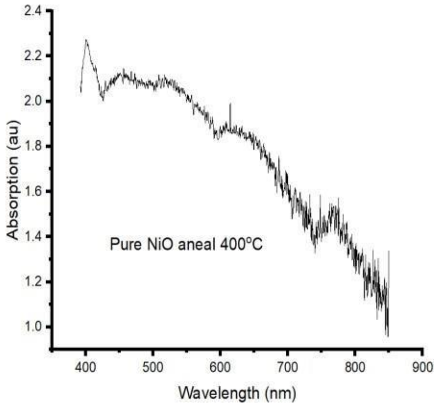
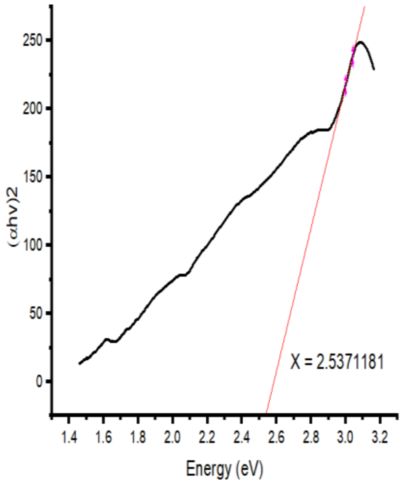
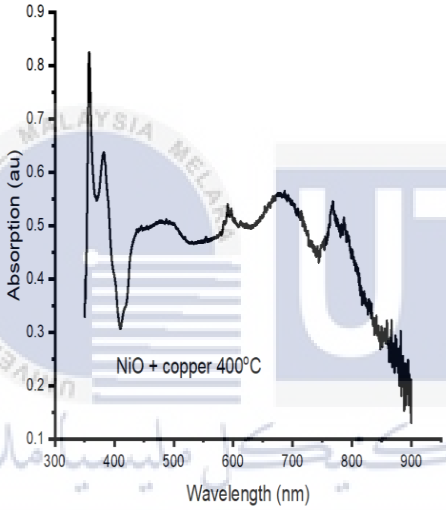
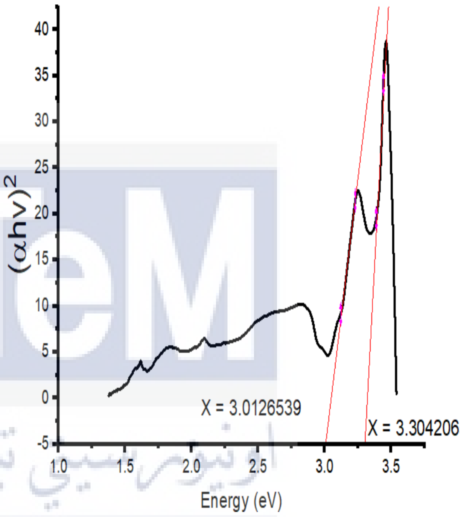
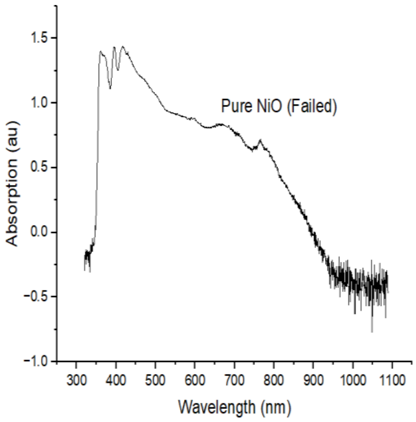
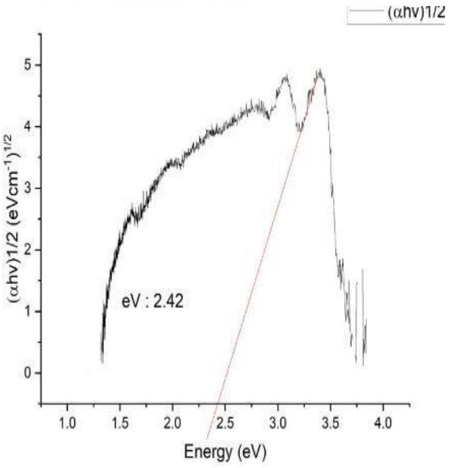
Besides that, the second part consists of NiO-4A, NiO:Cu-5A, that are annealed with 400°C on thin firm. The result for NiO-4A was increase from 0.4 au to 2.3 au, which is a problem because the peak absorption for NiO is only 1.0 au but it surpasses the value so the NiO-4A sample has error procedure during annealing process that makes the result increase rapidly. However, the NiO:Cu-5A sample are success since after annealing the value increase from 0.8 au to 0.85 au. While the bandgap value for NiO-4A also decreased from 3.01 to 2.53eV which is very horrible and the NiO:Cu-5A has increased from 2.99 eV to 3.01eV and there is second increment to 3.3eV.

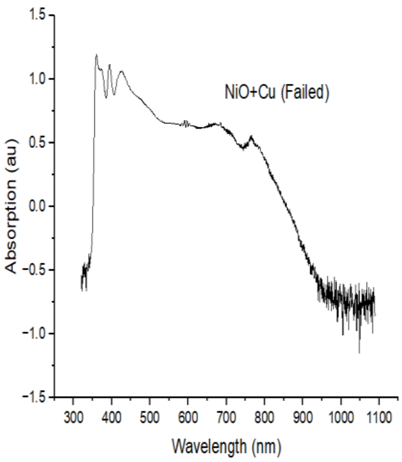
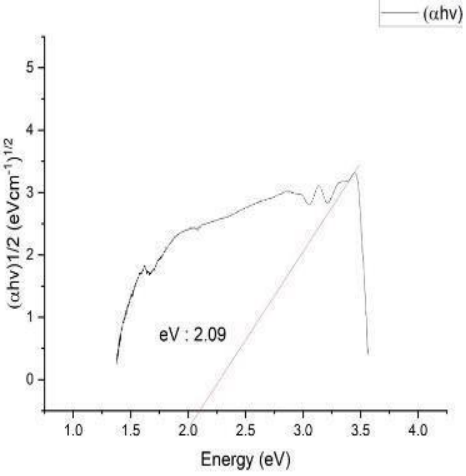
The third part shows NiO-6A, NiO:Cu-7A sample which are failed samples. As the result show that the absorption is reach out of the standard au range (1.0 au) for NiO and NiO dope Copper which is around 1.4 au respectively. The bandgap also drops until 2.42eV and 2.09 eV which does not meet the standard (3.0eV). From this result the NiO-6A, NiO:Cu-7A can prove that the sample are have failed NiO and NiO dope copper.

Finally, in this UV-Vis result the pure NiO meets the standard value from other research paper and by having a NiO doped copper the doping can increase the absorption light and bandgap values. In the other hand, the annealing process also can improve the sample but need to study further on the anneal temperature and time used, so that the sample can be controlled to make sure the values are achieved.

Table 4.5: Absorption and Bandgap of Sample

Sample	Absorption	Bandgap
NiO-1		
NiO:Cu -2		
NiO:Cu -3		

NiO-4A		
NiO:Cu -5A		
NiO-6A		

NiO:Cu -7A		

4.4 Summary

In this chapter 4, the result is discussed and analysis by scanning electron microscopes (SEM) and Uv-Vis test. SEM images reveal the formation of NiO and NiO:Cu nanoparticles of spherical forms, prove that electrodeposition offered superior deposition rate and uniformity compared to the sol-gel spin coating method, despite its more complex setup. Meanwhile, UV-Vis spectroscopy test confirms that the absorption light energy and bandgap value for NiO:Cu is better than pure NiO. This proves that NiO:Cu has better performance that pure NiO.

CHAPTER 5

CONCLUSION AND FUTURE WORKS

In this chapter, two sections are discussed. The first section reviewed and concluded this project. The second section provides suggestions on future enhancements for this project.

5.1 Conclusion

In conclusion, the first objective of this project is to synthesis the pure NiO and NiO doped copper successfully deposited. As the deposition method has been involve both pure NiO and NiO doped copper was using sol gel with spin coating method and electrodeposition and each method has their own advantages and disadvantages. The second objective of this project is to characteristic the pure NiO and NiO doped copper. Following that, several characterizations of the test are performed, in SEM results showed that electrodeposition offered superior deposition rate and uniformity compared to the sol-gel spin coating method, despite its more complex setup. In UV-Vis analysis test confirms that the absorption light energy and the bandgap of pure NiO outperformed NiO:Cu in terms of performance and stability due to the effects of doping, charge electron carriers, and lattice distortions. It can be concluded that, from these characteristics and analysis results shows that NiO:Cu still has better performance than pure NiO material as the HTL. However, there is still a lot of room for improvement in the preparation of NiO doped copper and make it better. Lastly, it is hoped that these results will give an opportunity to produce a complete IPSC with excellent performance.

5.2 Future Work

For future work and recommendation, the project focus will be on fabricating a whole IPSC device. For expected fabrication devices, the pure NiO can sandwich with FTO/NiO/Perovskite/ZnO/Ag and for dope ITO/NiOxCu/Perovskite/ZnO/Ag. Next, study and research of the different NiO dope copper doping levels were also conducted because different doping levels has been identifying that the doping level can affects the performance without compromising other material in IPSC. For synthesis, new methods can be tested, such as physical vapor techniques like Pulsed Laser Deposition (PLD) or sputtering, will also be undertaken. Furthermore, in characterization techniques the more advanced test been used like X-ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM), and scanning tunneling microscopy (STM) will be employed to scrutinize the surface composition, morphology, and electronic structure of IPSC with NiO and doped NiO device.

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