POWER CONVERSION EFFICIENCY ENHANCEMENT USING SILVER NANOPARTICLE AND TITANIUM DIOXIDE COMPOSITE FILM FOR FLEXIBLE TITANIUM FOIL ELECTRODE OF BACK-ILLUMINATED DYE-SENSITIZED SOLAR CELL

By

NUR HAZAHSHA BINTI SHAMSUDIN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

June 2022

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DEDICATION

I dedicate this dissertation to my beloved mom, Zaharah Binti Ibrahim, the woman who sacrifices everything for me unconditionally. You are my source of strength throughout this journey.

Thank you Allah

Thank you ‘

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chair: Prof. Ir. Mohd Zainal Abidin Bin Ab Kadir, PhD
Faculty: Engineering

Power conversion efficiency (PCE) in dye-sensitized solar cells (DSSCs) is a pivotal parameter to gauge the ability to convert light energy into electricity. However, the preference of substrates is among the issues that contribute to a low PCE. While polymer substrates have been exploited as the photoelectrodes for their flexibility and transparency which allows for front illumination, high temperature is required to eliminate the polymer binder from deposited titanium oxide (TiO_2) paste. The polymer substrates are unable to withstand high temperature, and as such affect the PCE with the residue of the polymer binder within TiO_2 film. Herein, a flexible titanium (Ti) substrate, a metal-based material, is used to replace the polymer substrate as the photoelectrode. Inherently, back illumination through counter electrode was opted for instead of front illumination through photoelectrode, owing to the opaque characteristic of metal substrate. However, the longer distance for the light to travel via this route impeded the development of photoelectrons, and thus reducing the power conversion efficiency (PCE). Therefore, the aim of this research is to improve the PCE via back illumination method. Herein, there were two methods proposed so as to increase the PCE. The first method is by coating the counter electrode with a different volume of platinum (Pt) solution at 30 µl, 50 µl, 70 µl and 90 µl. The second method proposed was incorporation of silver nanoparticles (AgNP) prepared via solvothermal into TiO_2 paste. Platinum is integrated in the counter electrode for the optimization of PCE through catalytic activity induced by Pt. It was revealed that 70 µL of Pt solution increased the PCE from 1.248 % to 5.25 %. The incorporation of AgNP into TiO_2 film has been proven to improve the light absorption as compared to solely TiO_2. Solvothermal, a chemical synthesis method was implemented with a varying amount of capping agent of polyvinylpyrrolidone (PVP) from 0.2 to 2.6 g to synthesize the silver nanoparticles. TEM images showed that the higher amount of PVP would result in smaller AgNPs size, while the largest AgNPs resulted from the lower amount of PVP. The absorbance of the AgNPs signified that the absorbance decreased with increasing PVP amount. From the results, the AgNP with 28.6 nm sizes was selected to be incorporated in TiO_2 paste for its low agglomeration and high absorbance value. The concentration of AgNPs was thereafter, varied from 1 to 3 wt.%, incorporated into TiO_2 and followed by subsequent annealing process at 450 °C to fabricate AgNP-TiO_2 film. It was observed that addition of 2 wt.% concentration of AgNPs showed the highest improvement of PCE at 4.691 % from that of TiO_2 film with PCE of 2.35 %. Therefore, coating the counter electrode with Pt solution by volume and adding AgNPs into TiO_2 methods have been carried out by which they have been proven to improve the PCE in DSSC. With this quantification, this study provides a way in addressing a durable and wearable flexible structure of portable electrical solar source in a wide range light intensity environment.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
Sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENINGKATAN KECEKAPAN PENUKARAN KUASA MENGGUNAKAN NANOPARTIKEL PERAK DAN FILEM KOMPOSIT TITANIUM DIOKSIDA UNTUK ELEKTROD TITANIUM FLEKSIBEL BAGI SEL SURIA SOLAR PEKA PEWARNA PENCERNA BELAKANG

Oleh

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Kecekapan penukaran kuasa (PCE) dalam sel suria pemeka pewarna (DSSC) ialah parameter penting untuk mengukur keupayaan untuk menukar tenaga cahaya kepada elektrik. Walaubagaimanapun, pemilihan substrat menjadi antara isu yang menyumbang kepada permasalahan PCE yang rendah. Walaupun substrat polimer telah dieksploitasi sebagai fotoelektrod untuk fleksibiliti dan ketelusannya yang membolehkan pencahayaan hadapan, suhu tinggi diperlukan untuk menghapuskan pengikat polimer daripada semikonduktor titanium oksida (TiO_2) yang terdapat pada substrat tersebut. Substrat polimer tidak dapat menahan suhu tinggi, dan oleh itu menjejaskan PCE dengan sisa pengikat polimer dalam filem TiO_2. Di sini, substrat titanium (Ti) fleksibel, bahan berasaskan logam, digunakan untuk menggantikan substrat polimer sebagai fotoelektrod. Secara semulajadi, pencahayaan belakang melalui elektrod kaunter telah dipilih dan bukannya pencahayaan hadapan melalui fotoelektrod kerana ciri substrat logam yang legap. Walau bagaimanapun, jarak yang lebih jauh untuk cahaya bergerak melalui laluan ini menghalang pembangunan fotoelektron, dan dengan itu mengurangkan kecekapan penukaran kuasa (PCE). Oleh itu, tujuan penyelidikan ini adalah untuk menambah baik PCE melalui kaedah pencahayaan belakang. Di sini, terdapat dua kaedah yang dicadangkan untuk meningkatkan PCE. Kaedah pertama ialah dengan menyalut elektrod pembilang dengan isipadu larutan platinum (Pt) yang berbeza daripada 30 μl, 50 μl, 70 μl dan 90 μl. Kaedah kedua yang dicadangkan ialah penggabungan AgNP yang disediakan melalui solvotermal ke dalam TiO_2. Platinum disepadukan dalam elektrod kaunter untuk pengoptimuman PCE melalui aktiviti pemangkin yang disebabkan oleh Pt. Telah didedahkan bahawa 70 μL larutan Pt meningkatkan PCE daripada 1.248 % kepada 5.25 %. Penggabungan AgNP ke dalam filem TiO_2 telah terbukti meningkatkan penyerapan cahaya berbanding dengan TiO_2 semata-mata. Solvotermal, kaedah sintesis kimia telah dilaksanakan dengan jumlah agen penutup polivinilpirolidon (PVP) yang berbeza-beza daripada 0.2 hingga 2.6 g untuk mensintesis nanozarah perak. Imej TEM menunjukkan bahawa jumlah PVP yang lebih tinggi akan menghasilkan saiz AgNP yang lebih kecil, manakala AgNP yang terbesar terhasil daripada jumlah PVP yang lebih rendah. Penyerapan AgNP menunjukkan penyerapan menurun dengan peningkatan jumlah PVP. Daripada keputusan, AgNP dengan saiz 28.6 nm telah dipilih untuk dimasukkan ke dalam TiO_2 untuk penggumpalan rendah dan nilai penyerapan yang tinggi. Kepekatan AgNPs selepas itu, diubah daripada 1 hingga 3 wt.%, dimasukkan ke dalam TiO_2 dan diikuti dengan proses pembakaran seterusnya pada 450 °C untuk menghasilkan filem AgNP-TiO_2. Diperhatikan bahawa penambahan 2 wt.% kepekatan AgNPs ke dalam TiO_2 menunjukkan peningkatan tertinggi PCE sebanyak 4.691 % daripada TiO_2 dengan PCE hanya sebanyak 2.35 %. Oleh itu, cara salutan elektrod kaunter dengan larutan Pt mengikut isipadu dan menambah AgNPs ke dalam TiO_2 telah dijalankan yang mana ia telah terbukti dapat meningkatkan PCE dalam DSSC. Dengan kuantifikasi ini, kajian ini dapat menyediakan cara dalam menangani struktur fleksibel yang tahan lama dan boleh pakai bagi sumber suria elektrik mudah alih dalam persekitaran cahaya yang lebih malap.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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PE	Photoelectrodes
PCE	Power conversion efficiency
Jsc	Current density
FF	Fill factor
Voc	Open circuit voltage
AgNPs	Silver nanoparticles
TW	terawatt
CE	Counter electrode
DSSC	Dye sensitized solar cell
FTO glass	Fluorine-doped Tin Oxide soda lime glass
TCO	Transparent Conductive Oxide
ITO	Indium-doped tin oxide
PET	Polyethylene terephthalate
PEN	Polyethlene naphthalate
LUMO	lowest unoccupied molecular orbital
wt%	Weight percent
TiO_2	Titanium dioxide
$NaBH_4$	sodium borohydrate
Fe_2O_3	Iron (III) oxide
AgNPs	Silver Nanoparticles
AuNPs	Gold Nanoparticles
Ti	Titanium

Al	Aluminium
Cu	Copper
AgNPs-TiO_2	The incorporation of silver nanoparticles into titanium dioxide
ZnO	Zinc Oxide
Nb_2O_5	Niobium Oxide
Zn^{2+}	Zinc ion
I^{3-}/I^-	triiodide/iodide
QS	quasi solid-state
SS	solid-state
PAN	poly(acrylonitrile)
PVC	poly(vinylchloride)
PEO	poly(ethyleneoxide)
HTM	hole-transporting material
TBP	4-tert-butylpyridine
TBA	tert-Butyl alcohol
PVP	poly(vinylpyrrolidinone)
DI	deionized water
Ni-Se	nickel selenide
In	Inconel
StSt	Stainless steel
SPR	Surface plasmonic resonance
$AgNO_3$	Silver Nitrate
EG	Ethylene Glycol

H_2O_2	Hydrogen peroxide
$TiCl_4$	Titanium tetrachloride
HNO_3HF	Mixture of nitric acid and hydrofluoric acid
Pt	Platinum
AgCl	Silver chloride
EDP	Electrodeposition
CI	chemical ionization
CVD	Chemical Vapor Deposition
DC	Direct Current
RF	Radio Frequency
C	Carbon
N	Nitrogen
Fe	Ferum
S/m	Siemens per meter
l	litre
mg	milligram
eV	Electron volt
TEM	Transmission electron microscopy
FESEM	Field emission scanning electron microscope
UV-Vis	Ultraviolet visible near infrared
XRD	X-ray diffraction
IPCE	Incident photon to current conversion efficiency
TGA	Thermogravimetric analysis

CHAPTER 1

INTRODUCTION

1.1 Research Background

Past research led by the energy researchers' team from Stanford University conducted a roadmap study related to the high performance of renewable energy consisting of sunlight, wind, geothermal, and hydropower in consuming just the 11.808 TW from the global energy demand. To do so, they gathered data covering all energy authorities in 139 countries with the predicted 100 % conversion up to 2050 of the aforementioned renewable energies [1]. Their roadmap provided strong shreds of evidence to support the proposition with optimum electricity consumption while casting new light over the world's worst scenarios such as global warming, air pollution, unpredictable energy expenses market, as well as burden climate and healthcare expenditures

As such, photovoltaic (PV) energy has been thrust into the limelight for years to come. Figure 1.1 gives the upshot of a global inspection commissioned by the National Renewable Energy Laboratory (NREL) to form an up-to-date view of the produced efficiencies by PV research experts across various technologies of PV in experimental scale-based over 45 years. There is a clear divide between the Silicon-based PV, thin films, and the surge of PV evolution through the cells made of organic and inorganic cells, DSSCs, and the perovskite PV-types when it comes to power conversion efficiency (PCE) performances. Among the diverse PV technologies developed, the highest PCE was emphasized by the multijunction cells, aside from crystalline Silicon PV cells. Even though the efficiency of the PV evolution or familiarly called third generation PV is the lowest PCE amongst others, the success rate of the evolved PV generation appears to be between (12 - 28) % compared to (14 - 24) % for the thin-film technologies. In the third generation PV cells, perovskite holds the PCE of 28%, whereby the PCE of DSSCs was around (10 - 13) % [2].

Amidst a well-performed plateau of PV energy, the excessive costs incurred by solar PV including manufacturing, transportation, assembling, installation, and maintenance are increasing yearly. Even worse, the complex procedures using high amounts of energy to manufacture the final product of cells, higher recombination losses due to the morphological structures of the grain boundaries which hinder the continuous flow of electrons in the cells, and the large losses of material are other baffling issues faced by Silicon cell [3], [4].

Subsequent disclosure of Flexi DSSCs was discovered by a similar group of researchers who developed the first DSSCs in 1993 [5]. Astoundingly, the finding was accidental but unveiled fresh insights into the flexible architectural role of DSSCs, thus garnering momentum on DSSCs application from lab maneuvers to commercial trade. This momentum has been grasped by third-generation-based PV companies of GreatCell Solar, GCell, and 3GSolar. DSSCs have been classified into two categories, and both categories are manufactured from Transparent Conductive Oxide (TCO) material but differ based on the coating layers. The first category known as a solid-state based DSSCs consists of transparent soda-lime glass layered with Fluorine Tin Oxide (FTO) or Indium Tin Oxide (ITO), while the second type is layered with polymer poly(ethylene terephthalate) (PET) or poly(ethylene naphthalate) (PEN) and termed as flexible based DSSCs. In some cases, the metal based substrates are also introduced in the flexible category. The solid-state is designed for extreme and harsh circumstances in wide geographical areas, whilst the criteria for flexibility are meant to be designed in any size or shape relevant to the incessant obligation of performance such as transparent, conductive, good transmittance, and low sheet resistances.

Metals are not as flexible as ITO-PET or ITO-PEN, most likely because they have standard ductility that limits their flexibility. However, metals can withstand intense heat temperatures soaring up to 500°C and are resistive to corrosion that is crucial for stability preservation purposes, in particular during electrolyte instillation. The most pervasive metals used are stainless steel (StSt) and titanium (Ti), other than copper (Cu), zinc (Zn), Inconel (In), and Tungsten (W). Although the highest PCE of flexible DSSCs was exhibited by StSt in contrast to the PCE of Ti as photolectrodes in a previous study [6], the conclusive findings [7], [8] disclosed that Ti leads ahead StSt when it comes to their stability aptitude.

Surface plasmon resonance (SPR), acting as the optical application in DSSCs, is proven to enhance the scattering effects of light into the metal nanostructure in the cell. This energy arises from the oscillation of electron density which becomes excited when being hit by polarized UV light. The process merely occurs at the surface of the metal nanoparticles. In this case, the absorbed light will be intensified and directed in terms of its intensity. Thus, the dye as the photoactive material would increase its efficiency in absorbing the sunlight. Commonly, the engagement of LSPR is found in boundless applications such as biomedical, sensing, medical, energy, and catalysis [9]–[11]. For more definitive preferences in this research, the plasmonic nano-additive metal particles are used as doping substances in DSSCs. Noble metals including Copper(C), Gold (Au), and Silver (Ag) can affect the SPR performance through their morphological properties such as shapes, sizes, and environment of the utilized metals nanoparticles [11].

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This study is concerned with the development of back-illuminated DSSCs with flexible metal electrode using various volume of Pt solution on counter electrode. By combining the advantageous of metal substrate with AgNPs-TiO2, the PCE of back-illuminated DSSCs could be enhanced. There is no shadow of doubt that, this research work able to contributes towards many fields such as the photodetector, telecommunication, optoelectronics and chemical based sensor areas.

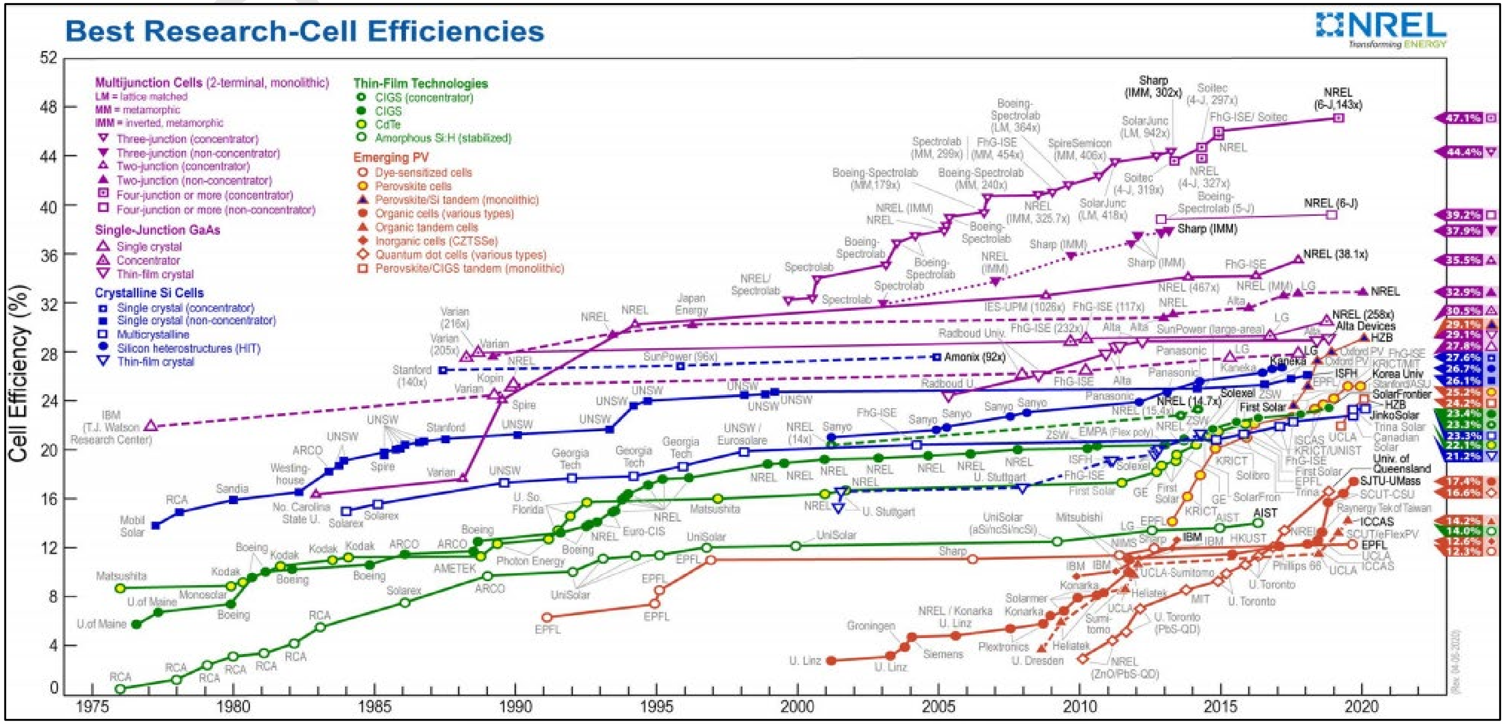

Figure 1.1: The trend of efficiency produced by energy researchers within 1975 -2020 as commissioned by the National Renewable Energy Laboratory (NREL) [2]

1.2 Problem Statement

Back illumination ensues from the solidity and opacity properties of Ti metal substrate as photoelectrodes. It occurs when light is exposed and transferred from the back of the DSSCs cell which is the counter electrode. This leads to the developed photoelectrons being stunted by lower luminous irradiation as a consequence of longer medium-light travel from the counter electrode to the TiO_2 semiconductor. To optimize the loss of back-illuminated performances, previous efforts and research were endeavored in modulating variable circumstances subjected to cathode electrodes playing a pre-eminent role during the light irradiation. This includes investigating the impact of Pt deposition on the counter electrode by manipulating different methods of Pt deposition [12], the deposition parameters [13], thermal annealed temperature [14], and Pt concentration [15]. Unlike the aforementioned past research, the current study employs the variation of Pt volume solution by using the spin coating method. The most common Pt deposition method is sputtering and chemical reduction [16]. Although sputtering provides technologically advanced privileges, especially the ability to deposit into a wide area with multiple controlled parameters, it necessitates a costly and high energy-consuming setup via a vacuum system. As chemical reduction deals with low-temperature heating and the use of sodium borohydrate ($NaBH_4$) as a reagent, spin coating is another alternative of coating Pt, favored for its uniformity, simplicity in the perspectives of preparation and fabrication as well as rein-condition of parameters [17], [18].

A preference of silver (AgNPs) plasmonic impurities doped TiO_2 is therefore implied to improve the deteriorating performance of TiO_2 on the afflicted issues. Instead of other plasmonic metal NPs such as copper(C), aluminium (Al), and gold (Au), AgNPs are known greatly for having the highest conductivity, which allows for more charge transfer pathways into TiO_2 to the external circuit. It also offers a degree of consistency in terms of corrosivity, flammability, and chemical traits. The plasmonic AgNPs excites its light intensity which helps the dye to further elongate the wavelength spectrum potency to the visible range of wavelength [19], [20].

Through years of research, metal plasmonic NPs-TiO_2 doping studies were mostly used for FTO glass substrates. According to a previous study [21], the increase of PCE of from 5.36% to 6.99% of TiO_2 could be observed only when TiO_2 was incorporated with the AgNPs via the chemical reduction technique. The conductivity was attributed to the competent interfacial movement of charge carriers by the AgNPs. Other studies have also reported that the AgNPs enhanced the PCE to 6.06 % [22] and 4.86 % [23] after doping with TiO_2. The solvothermal process on AgNPs on the other hand initiates the electron sink effect inside the photoanode to record the PCE of 6.56% [24]. The exploitation of AgNPs was also disclosed via ion implantation [25]. The implantation of AgNPs-TiO_2 was potent with PCE of 5.59 % contributed from the LSPR light absorption shift to the visible range that facilitates the photoelectron generation.

Nevertheless, there is less disclosed research on the doping of plasmonic nanoparticles-TiO_2NPs on flexible and back-illuminated DSSCs.

This study revolves around the idea of acquiring high temperature resistance and inexpensive flexible and back-illuminated DSSCs with better PCE performance. To do so, the merit plasmonic effect of the solvothermal produced from AgNPs as an additive material is exploited into TiO_2NPs film. The beneficial impacts of AgNPs which were mostly applied in FTO glass and front illuminated, was therefore exploited in this study that used flexible and back-illuminated structures of DSSCs. Before the doping process, different volumes of Pt solution are coated on the counter electrode by spin coating for optimization purposes. Therefore, an improvement must be done to improve the light absorption in photoanode which can be fulfilled by exploiting of LSPR in AgNPs of the photoanode.

1.3 Motivation

The low-temperature sintering for plastic and polymer photoanode substrate appears to be troublesome, in flexible DSSCs. Therefore, this has allured the current study to focus on Ti metal, instead. Unfortunately, the downside of Ti metal is the opacity feature that is unable to absorb light from the front which leads to back-illuminated circumstances. Therefore, the amount of Pt coated on CE provides an impact in optimizing the light penetration and catalytic activity into the CE during the process of light irradiation for back-illuminated performances. Hence, the various volume of Pt solution was investigated towards the back-illuminated DSSCs. For front illumination DSSCs, the optimum Pt volume of Pt solution is 50 µl. Therefore, for back illumination, the range selection is varied between 10 µl to 100 µl. Meanwhile, the synthesis of AgNPs via physical route commonly involves high energy and cost, despite the assurance of high purity in material production. In order to provide a simple and size-controlled product without a high degree of thermal requisites procedures, the preparation of customized AgNPs through the solvothermal method was executed.

1.4 Aim and Objectives

The research aims to improve the power conversion efficiency (PCE) performances of back-illuminated DSSC. This can be achieved by few objectives as highlighted below:

1) To investigate the effect of Platinum catalyst deposition by spin coating under 30 µl to 90 µl volumes of Pt solution towards the back-illuminated DSSC to improve light penetration of the counter electrode.

2) To synthesize the Silver nanoparticles (AgNPs) between 20 nm to 40 nm using the solvothermal method to be utilized in DSSC photoanode for improvement of light absorption.
3) To investigate on current-voltage (I-V) characteristics of back-illuminated DSSCs by embedding the synthesized AgNPs into TiO_2NPs based on a variation of concentration.

1.5 Scope of Research

This research work focuses on the development of flexible DSSCs, which was limited to the following circumstances:

- A Titanium foil with a thickness of 0.2 mm was tested as photoelectrode with the optimized film thickness at two layers Scotch tape thicknesses.
- The counter electrode (CE) is FTO coated glass substrate with a catalytic layer of Pt. This is due to the limitation of equipment for Pt deposition on ITO-PET substrates. The ranges of Pt volume solution at 30 µl, 50 µl, 70 µl and 90 µl were measured by micropipette under spin coating method.
- Method used to obtain Silver Nanoparticles (AgNPs) was the Solvothermal method, which was then embedded with TiO_2 nanoparticles under different concentrations. This research only conducted on the preliminary implementation of a composite AgNPs-TiO_2 film on the Ti foil photoelectrodes based DSSCs in performing the PCE performances. It also does not scrutinize the analysis and effect in regards to oxidation as well as corrosion of AgNPs resulting from the injected non-aqueous iodide/triiodide electrolyte.
- The experimental works are conducted Functional Nanotechnology Devices Laboratory (FNDL) and Nanomaterials Processing and Technology Laboratory (NPTL), Institute of Nanoscience and Nanotechnology (ION2), UPM Serdang.

1.6 Thesis Outline

The thesis is structured into five (5) chapters, which outlines as follows as:

Chapter 1 introduces the background of this research, the declaration of the research problem, and its objectives. The limitation of research activities is also asserted.

Chapter 2 presents the inclusive fundamental of Dye-Sensitized Solar Cells (DSSCs) and defines the Power Conversion Efficiency (PCE) Performances which includes Current Density (Jsc), Open Circuit Voltage (Voc), Fill Factor

(FF). The flexible DSSCs in particular of polymer, plastic and metal-based anodes are also reviewed. For metal-based anodes, Titanium is highlighted as the main preference. The review also comprises the surface modification of TiO_2 via TiO_2 nanostructured and Localized Surface Plasmon Resonance (LSPR) that exerted metal nanoparticles, specifically AgNPs as a doping substance.

Chapter 3 provides the chemicals and methods utilized to synthesize the AgNPs and the methodology of cell construction. The characterizations in verifying the optimized studies are also described.

Chapter 4 discusses the PCE and analysis of characterization from the experimental results.

Chapter 5 concludes the final finding of the research as well as suggestions for further extending of the research.

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