BACK ILLUMINATED FLEXIBLE DYE SENSITIZED SOLAR CELL USING TITANIUM FOIL AND PLATINUM- COATED INDIUM TIN OXIDE- POLYETHYLENE TEREPHTHALAT

By

IZAD SYAHRUL ARIFFIN BIN MOHD ISMAIL

Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in fulfilment of the Requirement for the Degree of Master of Science

June 2022

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This thesis is solely dedicated to my beloved parents, late (Allahyarham) Mr. Mohd Ismail K.A Fakir Mohd (may Allah bless him and place him in and place him in the best place) and Mrs. Repiah Uchin, family members and friends for their endless support, love and encouragement.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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Chairman : Prof. Mohd Nizar Hamidon, PhD
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Flexible Dye Sensitized Solar Cell (FDSSC) have attracted great attention due to its ability to install at curved and uneven surface. Therefore, several flexible substrates including plastic and metal substrate have been chosen to replace rigid glass substrate for DSSC application. However, due to the weak thermal stability of plastic Indium Tin Oxide Polyethylene Terephthalate (ITO-PET) substrate that limit the sintering process at 150℃, metal titanium (Ti) substrate was chosen as photoanode substrate due to its high thermal stability as well as good electrical conductivity. The main challenge for metal substrate is the opaqueness of metal substrate that need to be operated under back illumination method which will reduce the light penetration thus lead to the low power conversion efficiency. Consequently, the Platinum (Pt) layer on the ITO-PET for the counter electrode (CE) side need to be properly choose to allow sufficient light penetration under back illumination method. In this study, several experiments have been conducted to improve the efficiency of DSSC until the fabrication of flexible DSSC (FDSSC). During the fabrication of FDSSC, the titanium dioxide (TiO_2) was deposited onto Ti foil using Doctor-Blade method while platinum (Pt) was deposited using sputtering method onto ITO-PET. The highest efficiency of FDSSC with 2.03% has been achieved by applying and pre-treatment of hydrogen peroxide (H_2O_2) Ti foil as photoanode substrate and 7 nm thickness of Platinum (Pt) layer on ITO-PET as CE substrate under back illumination method.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SEL SURIA TERPEKA PEWARNA MUDAH LENTUR MENURUT KAEDAH PENCAHAYAAN BELAKANG MENGGUNAKAN KERAJANG TITANIUM DAN TIMAH INDIUM TEROKSIDA POLIETILENA TEREFTALAT BERSALUT LAPISAN PLATINUM

Oleh

IZAD SYAHRUL ARIFFIN BIN MOHD ISMAIL

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Sel suria terpeka pewarna mudah lentur telah menarik perhatian para pengkaji kerana keupayaannya untuk dipasang pada permukaan yang melengkung. Oleh itu, beberapa substrat mudah lentur termasuk plastik dan logam telah digunakan untuk menggantikan penggunaan substrat kaca. Walaubagaimanapun, disebabkan oleh kadar kestabilan haba yang rendah bagi substrat plastik seperti timah indium teroksida polietilena tereftalat yang mengehadkan proses pembakaran sehingga 150℃, maka substrat logam sepertititanium telah dipilih sebagai substrat fotoanod disebabkan oleh kadar kestabilan haba dan kadar kekonduksian yang baik. Akan tetapi, kadar kelegapan yang tinggi menjadi cabaran utama bagi substrat logam dan perlu dikendalikan di bawah kaedah pencahayaan belakang sekaligus akan membawa kepada pengurangan kadar kecekapan pada sel suria terpeka pewarna mudah lentur. Oleh yang demikian, tempelan lapisan platinum pada timah indium teroksida polietilena tereftalat yang digunakan sebagai substrat elektrod tindak balas perlu dioptimakan untuk membenarkan kadar peratusan penembusan cahaya yang mencukupi. Di dalam kajian ini, beberapa eksperimen telah dijalankan untuk meningkatkan kadar kecekapan sel suria terpeka pewarna mudah lentur. Semasa proses pembuatan sel suria terpeka pewarna mudah lentur, lapisan titanium dioksida ditempelkan pada substrat kerajangtitanium menggunakan kaedah "Doctor-Blade". Manakala lapisan platinum telah ditempelkan pada substrat timah indium teroksida polietilena tereftalat menggunakan kaedah percikan platinum. Kadar kecekapan Sel suria terpeka pewarna mudah lentur tertinggi sebanyak 2.03% telah dicapai menggunakan kerajang titanium dan ketebalan lapisan 7 nm platinum pada substrat timah indium teroksida polietilena tereftalat menggunakan kaedah pencahayaan belakang.

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May Allah ease and bless all of us with His blessings.

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LISTS OF ABBREVIATIONS

Al	Aluminium
AM 1.5	Air mass coefficient
ALD	Atomic layer deposition
CB	Conduction band
CE	Counter electrode
CO_2	Carbon dioxide
CIP	Cold isostatic pressure
CNT	Carbon nanotubes
COOH	Carboxylate group
CVD	Chemical vapour deposition
DSSC	Dye sensitized solar cell
e^-	Electron
E_{redox}	Redox potential of the electrolyte
EDX	Energy Dispersive X-ray Spectroscopy
EPD	Electrophoretic deposition
EIS	Electrochemical impedance spectra
eV	Electron-voltage
FDSSC	Flexible dye sensitized solar cell
FESEM	Field Emission Scanning Electron Microscope
FF	Fill factor
FTO	Fluorine doped tin oxide

GNP	Graphene nanoplatelet
GQD	Graphene quantum dots
H_2	Hydrogen
HOMO	Highest occupied molecular orbital
H_2O_2	Hydrogen peroxide
H_2PtCl_6	Hexachloroplatinic acid hydrate
H_2PO_3	Phosphonate group
I_3^-/ I^-	Triiodide/ iodide
IPCE	Incident-photon-to-current efficiency
I	Light intensity to the detector after transmittedthrough the sample
I_s	Incident photon flux density
I_o	Light intensity before transmitted through sample
I_{mp}	Optimum current
ITO-PET	Indium tin oxide coated polyethylene terephthalate
ITO-PEN	Indium tin oxide coated polyethylene naphthalate
I-V characteristics	Current-voltage characteristics
J	Joules
J_{sc}	Short circuit current
LUMO	Lowest unoccupied molecular orbital
$MeCpPtMe_3$	Trimethyl (methylcyclopentadienyl) platinum
N_{CB}	Total number of electrons states in the CB
NIR	Near infrared region

n-type	Excess of electrons
NH_4F	Ammonium fluoride
$NaBH_4$	Sodium Borohydrate
O_2	Oxygen
PANI	Polyaniline
PPy	Polypyrrole
p-type	Excess of holes
P_{mp}	Optimum power
P_{in}	Power input by sunlight
Pt	Platinum
PV	Photovoltaic
QDSC	Quantum Dot solar cell
R_{ct}	Charge transfer resistance
R_s	Series resistance
R_{sh}	Shunt resistance
R_t	Electron transport resistance
R_{sheet}	Substrate sheet resistance
Si	Silicon
StSt	Stainless steel
SnO_2	Tin dioxide stannic oxide
T	Transmittance
TiO_2	Titanium dioxide
TBP	4-*tert*-butylpyridine

TCO	Transparent conductive oxide
Ti	Titanium
UV	Ultra violet
UV-Vis	Ultraviolet-visible
V	Volts
VB	Valence band
V_{mp}	Optimum voltage
V_{oc}	Open circuit voltage
wt	Weight
Xe	Xenon
ZnO	Zinc Oxide
η	Power conversion efficiency
λ	Incident photon wavelength
kT	Thermal energy
q	Unit charge
c	Electron number in conduction band (CB)

CHAPTER 1

INTRODUCTION AND BACKGROUND

1.1 General Overview

In the beginning of the 21st century, the increase of energy consumption is expectedas the world population continues to expand. However, conventional fossil energywas too limited in order to meet growing needs, with drawbacks such as air and water pollution, as well as releasing unsustainable levels of carbon dioxide (CO_2) causing environmental effects, especially global warming and environmental destruction [1], [2]. Thus, renewable energy resources have received a lot of attention recently as one of the most promising green energy resources which hold tremendous potential in replacing the reliance on conventional energy resources [3].

Solar energy has already been used for a long period of time in human history as a heat source with 3×10^{24} Joules (J) of annual solar radiation striking the surface of the earth [4], [5]. Solar cells, on the other hand, made a significant difference by converting sunlight into electricity via the photovoltaic effect which was discovered by French physicist, Edmond Becquerel in 1839 [6].

The photovoltaic effect is a combination between *n-type* (excess of electrons) and *p-type* (excess of holes) silicon materials forming a *p-n* junction. When these two materials are in contact with each other, free electrons and holes are able to diffuse into the opposite materials, hence creating an internal electric form between the positive charge on the n-type side and negative charge on the p-types side (refer Figure 1.1). Several other types of first- and second-generation solar cells based on inorganic materials were successfully developed and inspired by this structure.

The advantages of first- and second-generation solar cells rely on their high efficiency of 13% to 25% and 10.2% to 21.7% respectively [7], [8]. However, despite its high efficiency among the solar cell group, the manufacturing process for first- and second-generation solar cells remains the main challenge especially when involving harmful, toxic and scarce materials.

Therefore, third generation solar cells have become a main focus and are expected to be a significant research and development in the fast-growing solar cell area. Dye sensitized solar cell (DSSC) is one of the most promising third generation solar cell groups. DSSC enables the ease of fabrication process without using the complex procedures, high temperature or high vacuum processes. Interestingly, DSSC is based on the photoelectrochemical process which is identical to the photosynthesis in green plant leaves.

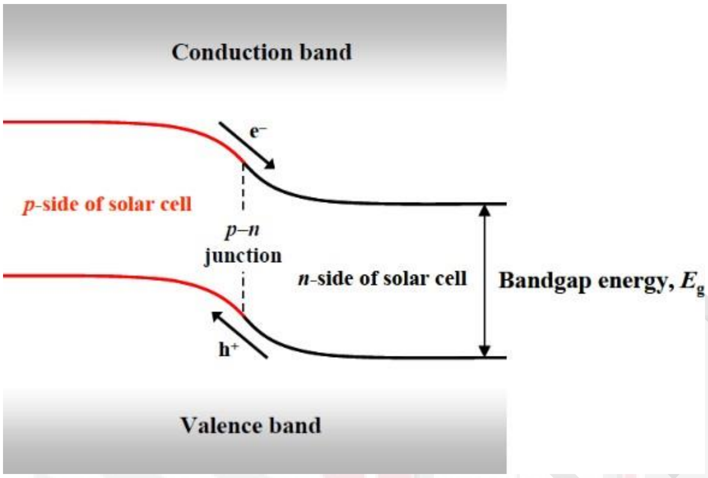

Figure 1.1 : Electron-holes transportation in *p-n* junction [9]

Concurrently, both first and second-generation solar cells are still limited by the theoretical value of maximum conversion efficiency of 33.7% established by Shockley-Queisser due to *p-n* junction. The limitation occured when an absorbed photon generates an electron-hole pair, while other photons with excess energy arewasted as heat. The limitation has led to research and development of third generation solar cells [10].

1.2 Third Generation Solar Cell

Third generation solar cells are based on nanomaterial approaches not relying on conventional *p-n* junctions and have been a practical and promising method for the efficiency improvement in comparison to first- and second-generation solar cells.Three types of solar cells which highly interested by researchers are:

1. Organic solar cell
2. Quantum dot solar cell (QDSC)
3. Dye sensitized solar cell (DSSC)

DSSC is one of the most promising approaches in third generation solar cell groups. Basically, DSSC is composed of photoanode side attached to the transparent conductive oxide (TCO) with 12 µm thick semiconductor layer (usually titanium dioxide with 10 – 20 nm diameter nanoparticles) anchored with molecular dye (normally N719 Ruthenium dye) via carboxylate ligands which absorbs sunlight. The photoanode was then sandwiched together with a counter electrode (CE) side containing a catalyser layer (normally platinum). A redox electrolyte is placed between a dye sensitized photoanode and catalyst coated CE.The details of DSSC working principle as well as DSSC components will be presented in the next chapter, while the basic layout of DSSC is presented in Figure1.2.

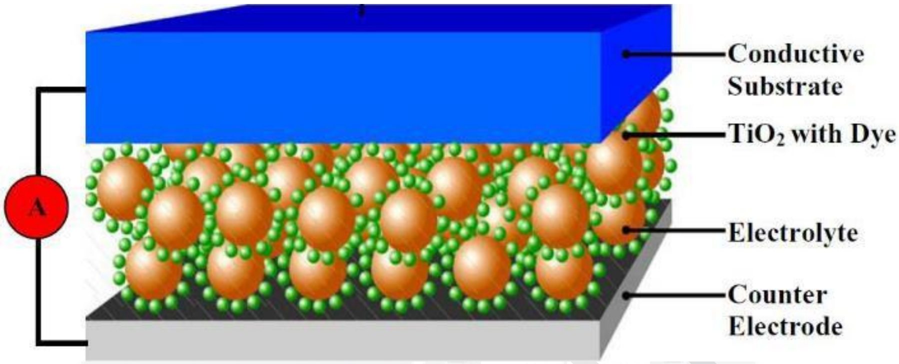

Figure 1.2 : Basic layout of DSSC [9]

Figure 1.3 illustrates the operating principle of DSSC and the operation principles of DSSC generally can be divided into 4 basic steps:

1. Absorption
2. Electron injection
3. Dye regeneration
4. Iodide regeneration

As light is illuminated onto the photoanode side, a photon is absorbed by a dye molecule. Due to the *absorption process,* photoexcitation occurred, thus promotingthe electron (e^-) to the excited state (S*) from the ground state (S^o/S^+) of the dye,resulting in an oxidized dye. Consequently, an electron is injected into the conduction band of the semiconductor layer through the *electron injection* process. The injected electron is then transported through the network of semiconductor layers and flowing through the external circuit. The *iodide regeneration* process takes place at the CE as the electron reaches the CE coated with a catalyst layer, where the triiodide ion (I_3^-) receives an electron and promotes it to an iodide ion (I^-). Concurrently, the oxidized dye is reduced to its ground state (S^o/S^+) as it receives an electron to replace the lost electron by the redox iodide/triiodide (I^-/I_3^-) coupled in electrolyte (*dye regeneration*).

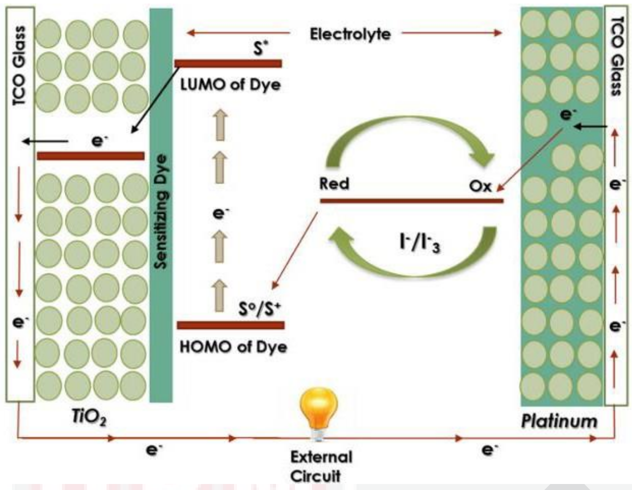

Figure 1.3 : DSSC operating principle [11]

Recently, flexible DSSC (FDSSC) has attracted considerable attention due to the competitive advantage of fabrication on flexible substrates which increased the requirements for the broader application and easy installation on curved surfaces.Therefore, various flexible substrates, including indium tin oxide coated polyethylene terephthalate (ITO-PET) and indium tin oxide coated polyethylene naphthalate (ITO-PEN) substrates as well as metal sheets have been used for FDSSC fabrication.

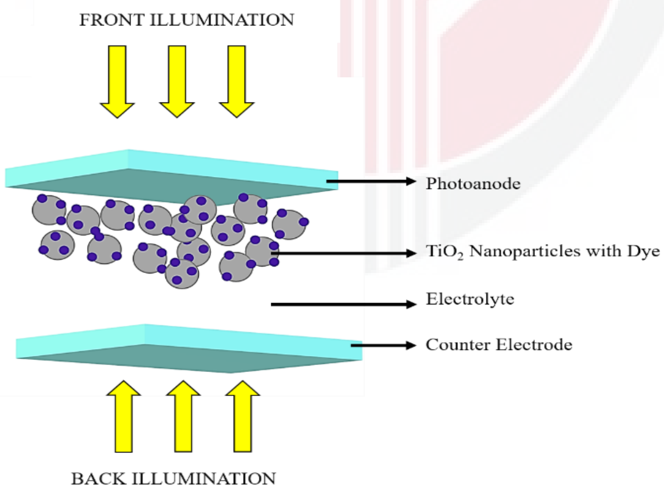

Figure 1.4 : Front illumination and Back illumination

Metal substrate has been used to overcome the limitation since the weak thermal stability of plastic substrate that limits the sintering process to as low as 150℃, Higher sintering temperature (450℃) is required to remove the organic binder from the TiO_2 paste to achieve a good electrical contact at the interface between the TiO_2 film and the substrate as well as to enhance the mechanical strength of the TiO_2 semiconductor layer. Owing to its excellent properties of low sheet resistance (R_{sheet}), high thermal stability, abundance and flexibility to install at various angles, metal foil has been chosen widely as a flexible substrate. Despite their excellent properties, the main challenge of metal substrates is their opaqueness which required to be illuminated from CE side (back illumination) instead of photoanode side (front illumination) as illustrated in Figure 1.4.

Besides, the DSSC technology disadvantage is low power conversion efficiency especially when dealing with flexibility and back illuminations method. The best-known conversion efficiency in FDSSC is only at 8.02% which focuses on the improvement of the semiconductor oxide (photoanode side) using Titanium (Ti) foil substrate [12]. However, this is relatively low as compared to rigid and inflexible DSSC (14.3%) [13]. Therefore, this research will concentrate on improving the efficiency of FDSSC using back illumination methods for future commercialization.

1.3 Problem Statement

Due to the limitation of flexible plastic substrates such as ITO-PET, they are only restricted to low sintering temperature which limited the sintering process to 150℃. Poor interparticle connectivity, and poor adhesion between interparticle and substrate has resulted in a high probability of semiconductor layer cracks leading to low power conversion efficiency. Thus, leaving only a metal substrate to be used as a photoanode substrate with the ability to withstand a high temperature sintering method, where high temperature sintering process is crucialfor excellent electron collection transportation purposes and substantially to remove additives in semiconductor oxide paste as well as to provide good interconnection between nanoparticles to provide excellent electron collection transportation

Despite of the advantages owned by metal substrate; all kind of metal substrates suffer from low optical transmittance due to its opaqueness which require the solarcell to be operated under back illumination method that will slightly decrease the performance of FDSSC because of the limitation of sunlight penetration through the CE side. Consequently, the thickness of the Pt catalyst layer needs to be properly chosen in order to increase the efficiency of the FDSSC under the back- illumination method.

Normally, DSSC is made from rigid FTO glass substrate that can only be installed at a fixed angle which makes it challenging in order to achieve its maximum exposure of the sunlight as it falls at different angles throughout the day. Therefore,orienting it at a certain angle is the crucial requirement to maximize its exposure to direct sunlight. For this reason, FDSSC is selected due to its ability to be flexedat various angles without being put into calculation of the tilt angle in order to achieve the maximum exposure to the sunlight in which will contribute to the increment in power conversion efficiency.

1.4 Research Objectives

The main aim of this work is to propose and fabricate flexible DSSC (FDSSC) with improved efficiency. Several steps in order to improve the FDSSC efficiency are stated as below:

1. To investigate back illumination based DSSC utilizing Ti foil as photoanode to the I-V characteristics performance.
2. To study the effect of the back-illumination method to the I-V characteristics performance at different thickness of sputtered ITO- PET platinum (Pt) layer in order to improve the power conversion efficiency of DSSC under back illumination method.
3. To study the effect of flexibility of fabricated FDSSC at varying angles (from 0° to 60°) towards the I-V characteristics performance.

All the necessary assumptions, theories, statistics, calculations, relevant discussionand analysis are included in this project and expected to propose some inspirationsto overcome the problem in improving DSSC efficiency.

1.5 Scope of Work

This study will concentrate on the photoanode and CE substrate of the DSSC. Meanwhile, N719 photosensitizer and electrolyte are kept constant using the manufacturing process as described in [14] and [15] respectively.

As for the photoanode component, this research work will focus on the semiconductor deposition method which will involve the use of Doctor-Blade method and titanium dioxide (TiO_2) semiconductor material to produce a size of 1cm x 1 cm working area. Both fluorine doped tin oxide (FTO) glass and Ti foil will be used as rigid and flexible photoanode substrate respectively.

Besides that, the thickness of the TiO_2 layer will not be considered as one of the components of this research due to difficulty in controlling the thickness using the Doctor-Blade method. Thus, the thickness will be controlled using adhesive tape for photoanode masking. The minimum thickness of adhesive tape is 63 µm and the thickness of TiO_2 semiconductor layer after the sintering process is estimated between 10 µm to 15 µm [16]. Meanwhile, a sputtering method will be used in the CE part for Pt deposition as a catalyst layer. Similar to the photoanode component, FTO glass and ITO-PET will be used as rigid and flexible CE substrates respectively. As for the fabricated FDSSC, flexibility study will concentrate on bending tests only due to sturdiness of Ti foil.

1.6 Thesis Organization

This dissertation is structured and organized into five chapters. Chapter one provides a general overview of the list of energy that has been used globally including a background of FDSSC. Problem statement and research objectives of this dissertation are also presented in this chapter

Chapter two provides literature review on the basic of operation principles of PV devices including FDSSC material, manufacturing methods, emphasis on the alternative substrate of TCO glass and necessary information are presented.

Chapter three presents the detailed explanation on FDSSC material chosen for eachFDSSC constituents consisting of photoanode and CE substrate, N719 dye and electrolyte solution as well as Pt layer. General overview of each measurement technique as well as detailed material that has been used in this research project ispresented in this chapter.

Chapter four is the main part in this research project report. Comparison study of front illumination and back illumination performance as well as optimization of back illumination method. The performance comparison of the bending test effect in I-V characteristics varies with different angles (from 0° to 60°). Additionally, all details analysis and discussion are presented in this section.

Chapter five gives an overall conclusion for this research work along with future work recommendation provided.

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