



UNIVERSITI PUTRA MALAYSIA

**DEVELOPMENT OF TITANIUM DIOXIDE BASED COMPACT LAYERS
AND LIGHT SCATTERING LAYERS FOR ENHANCED DYE-SENSITIZED
SOLAR CELL**

MUHAMMAD NORHAFFIS BIN MUSTAFA

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CELL**

By

MUHAMMAD NORHAFFIS BIN MUSTAFA

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
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Philosophy**

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

DEVELOPMENT OF TITANIUM DIOXIDE BASED COMPACT LAYERS AND LIGHT SCATTERING LAYERS FOR ENHANCED DYE-SENSITIZED SOLAR CELL

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Dye-sensitized solar cells (DSSCs) are the third-generation solar cell that capable of converting solar energy into electrical energy. Titanium dioxide (TiO_2) as a photoanode has faced a lot of drawbacks such as low dye loading capacity, a small range of light scattering, high recombination effect and low charge transport ability that subsequently reduces its power conversion efficiency (PCE). In this work, the enhancement of DSSC performance was studied by the modification of photoanode, specifically on the fabrication of a new compact layer (CL) and light scattering layers (LSLs). A dense, compact and homogenous TiO_2 CL was optimized and prepared using response surface methodology by central composite design (RSM/CCD) and heat treatment assisted electrospinning, respectively. The TiO_2 CL was successfully optimized with less than 5% residual standard error (RSE) and capable of enhancing the PCE up to 76.88% compared with the bare photoanode (1.73%). This is due to an improved electron lifetime (τ_n) and charge collection efficiency (η_c), resulting in a low recombination effect that leads to a higher PCE. Two LSLs were prepared in this study, namely polyvinyl alcohol (PVA/ TiO_2) nanofibers and TiO_2 decorated by graphene quantum dot (TiO_2 -GQD). The PVA/ TiO_2 was prepared using electrospinning while TiO_2 -GQD was prepared via electrodeposition and drop-casting technique. Both PVA/ TiO_2 nanofibers and TiO_2 -GQD LSLs were successfully optimized using RSM/CCD with less than 5% RSE. Upon the addition of TiO_2 -GQD LSL onto the photoanode, the PCE increased up to 5.01% compared to the photoanode with PVA/ TiO_2 nanofibers LSL (4.06%) and bare photoanode (3.06%). This increment is due to the longer τ_n , higher η_c , higher dye loading capacity and higher light reflectance, demonstrating a good light scattering material. Furthermore, a fully flexible photoanode with TiO_2 -GQD LSL has successfully fabricated on indium doped tin oxide/polyethylene naphthalate (ITO/PEN) flexible substrate via electrodeposition and drop-casting technique. The fully flexible DSSC device consisting of photoanode with TiO_2 -GQD LSL

showed an enhanced PCE of 5.18% compared to the bare photoanode (2.65%). The vast enhancement of PCE was due to the increase in the dye loading capacity (more dye can be adsorbed) and light scattering ability (more light can be scattered) upon the addition of TiO₂-GQD LSL. In a nutshell, the introduction of CL and LSLs has successfully increased the DSSC performance.



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**PEMBANGUNAN LAPISAN KOMPAK DAN LAPISAN PENYERAKKAN
CAHAYA BERDASARKAN TITANIUM DIOKSIDA UNTUK SEL SOLAR
BERKEPEKAAN PEWARNA YANG DITINGKATKAN**

Oleh

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Ogos 2020

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Sel solar berkepekaan pewarna (DSSCs) ialah generasi ketiga sel solar yang mampu menukarkan tenaga solar kepada tenaga elektrik. Titanium dioksida (TiO_2) sebagai fotoanod telah mengalami banyak masalah seperti kapasiti pemuatan pewarna yang rendah, julat penyerakkan cahaya yang kecil, kesan penggabungan yang tinggi dan keupayaan pengangkutan caj yang rendah dan seterusnya mengurangkan kecekapan penukaran kuasanya (PCE). Dalam kajian ini, peningkatan prestasi DSSC dikaji dengan pengubahsuaian fotoanod, khususnya pada penghasilan baru lapisan padat (CL) dan lapisan penyerakkan cahaya (LSLs). CL TiO_2 yang padat dan homogen dioptimumkan dan disediakan masing-masing dengan menggunakan metodologi permukaan tindak balas dengan reka bentuk komposit pusat (RSM/CCD) dan elektroputaran dibantu rawatan haba. CL TiO_2 berjaya dioptimumkan dengan baki ralat standard (RSE) kurang dari 5% dan mampu meningkatkan PCE sehingga 76.88% berbanding dengan fotoanod pengosong (1.73%). Hal ini disebabkan oleh peningkatan jangka hayat elektron (τ_n) dan kecekapan pengumpulan caj (η_c), menghasilkan kesan penggabungan yang rendah yang membawa kepada PCE yang lebih tinggi. Dua LSL disediakan di dalam kajian ini iaitu nanofiber poli(vinil alkohol)/titanium dioksida (PVA/ TiO_2) dan TiO_2 dihiasi dengan titik kuantum grafin (TiO_2 -GQD). Nanofiber PVA/ TiO_2 telah dihasilkan melalui elektroputaran manakala TiO_2 -GQD telah dihasilkan melalui elektroenanapan dan kaedah penyalutan titis. Kedua-dua LSL nanofiber PVA/ TiO_2 dan TiO_2 -GQD telah berjaya dioptimumkan menggunakan RSM/CCD dengan RSE kurang dari 5%. Setelah penambahan TiO_2 -GQD LSL ke atas fotoanod, PCE telah meningkat sehingga 5.01% berbanding fotoanod dengan PVA/ TiO_2 nanofibers LSL (4.06%) dan fotoanod pengosong (3.06%). Peningkatan ini disebabkan oleh τ_n yang lebih lama, η_c yang lebih tinggi, kapasiti pemuatan pewarna yang lebih tinggi dan

pantulan cahaya yang lebih tinggi, menunjukkan ciri-ciri bahan penyerakan cahaya yang baik. Selanjutnya, fotoanod fleksibel sepenuhnya dengan TiO_2 -GQD LSL telah berjaya disediakan pada substrat fleksibel indium timah oksida/polietilena naftalat (ITO/PEN) melalui elektroenapan dan kaedah penyalutan titis. Peranti DSSC yang fleksibel sepenuhnya terdiri daripada fotoanod dengan LSL TiO_2 -GQD menunjukkan peningkatan PCE sebanyak 5.18% berbanding dengan fotoanod pengosong (2.65%). Peningkatan PCE yang sangat besar adalah disebabkan oleh peningkatan kapasiti pemuatan pewarna (lebih banyak pewarna boleh dijerap) dan kemampuan penyerakan cahaya (lebih banyak cahaya boleh diserakkan) setelah penambahan LSL TiO_2 -GQD. Kesimpulannya, penambahan CL dan LSL telah berjaya meningkatkan prestasi DSSC.



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“Some people arrive and make such a beautiful impact on your life, you can barely remember what life was like without them”

- Anna Taylor -

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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LIST OF SYMBOLS

Symbol	Meaning	Unit
OCP	Open circuit voltage	V
P_{in}	Power input	$mW.cm^{-2}$
P_{max}	Maximum power	mW
J_{sc}	Short circuit current	$mA.cm^{-2}$
V_{oc}	Open circuit voltage	V
R_{ct}	Charge transfer resistance	Ω
R_s	Series resistance	Ω
CPE	Constant phase element	F
Z'	Real impedance	Ω
Z''	Imaginary impedance	Ω
E_{pp}	Peak to peak separation	V
η_c	Charge collection efficiency	%
PCE	Power conversion efficiency	%
τ_n	Electron lifetime	ms
λ	Wavelength	nm
RSE	Residual standard error	%
FF	Fill factor	%
J_{max}	Maximum current	mA
V_{max}	Maximum voltage	V

LIST OF ABBREVIATIONS

AACVD	Aerosol assisted chemical vapor deposition
ATR	Attenuated total reflection
BET	Brunauer–Emmett–Teller
CLs	Compact layers
CO ₂	Carbon dioxide
D	Dye molecules
D ⁺	Hole of dye molecules
DSSCs	Dye-sensitized solar cells
e ⁻	Electron
EIS	Electrochemical impedance spectroscopy
Eu ³⁺	Europium ion
F ⁻	Fluoride
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
FTO	Fluorine doped tin oxide
GO	Graphene oxide
GQD	Graphene quantum dot
GW	Gigawatts
HF _s	Hollow fibers
IEA	International energy agency
IRENA	International renewable energy agency
ITO	Indium doped tin oxide
ITO/PEN	Indium doped tin oxide/polyethylene naphthalate
LSL	Light scattering layer
MRs	Micro rods

Nb	Niobium
Nb ₂ O ₅	Niobium pentoxide
NFs	Nanofibers
NiTDP	Nickel doped titanium dioxide powder
NOAA	National oceanic and atmospheric administration
NRs	Nanorods
PCE	power conversion efficiency
PEI	Polyethyleneimine
P-TiO ₂	Popcorn-like titanium dioxide
PV	Photovoltaic
PVA/TiO ₂	Polyvinyl alcohol/titanium dioxide
R	Reduced redox species
R ⁺	Oxidized redox species
RGO	Reduced graphene oxide
RSM/CCD	Response surface methodology with central composite design
SnO ₂	Tin oxide
TCMS	Titanium dioxide core-shell microspheres nanostructures
TCO	Transparent conductive oxides
TDIP	Titanium diisopropoxide
THNR	Titanium dioxide hierarchical nanorods
Ti ⁴⁺	Titanium (IV)
TiCl ₃	Titanium trichloride
TiCl ₄	Titanium tetrachloride
TiO ₂	Titanium dioxide
TiO ₂ -GQD	Titanium dioxide decorated by graphene quantum dot
TMMS	Titanium dioxide smooth microspheres surface
TMSR	Titanium dioxide rough microspheres surface

TNA	Titanium dioxide nanotube arrays
TNNW	Titanium dioxide nanowires with nanoscale whiskers
TNW	Titanium dioxide nanowires
TTIP	Titanium tetraisopropoxide
TW	Terawatts
USA	United State America
WER2-O	Titanium dioxide nanocrystalline polygons with rods
W-TiO ₂	Worm-like titanium dioxide
WWF	World wide fund
XRD	X-ray diffraction
Yb	Ytterbium
YbF ₃ :Eu ³⁺	Europium ion doped ytterbium fluoride
ZnO	Zinc oxide

CHAPTER 1

INTRODUCTION

1.1 Background of study

In the last few decades, the demand for energy supply increases rapidly as the population around the world keep increasing. According to the International Energy Agency (IEA), the demand for energy is expected to increase by 27% or 3743 million tons of oil equivalent, globally from 2017 to 2040. The problem arises because the mainstream energy supply still depends on non-renewable energy such as petroleum, natural gas and charcoal. This type of energy is limited and causes pollution. One of the main concerns regarding the pollution caused by the use of non-renewable energy is global warming, where carbon dioxide (CO₂) emission is the main cause of this problem. The global CO₂ emission has increased significantly from 2 billion tons per year in 1900 to over 36 billion tons per year in 2015, increasing the average global temperature (Le Quéré *et al.*, 2018). According to National Oceanic and Atmospheric Administration (NOAA), the earth experienced its second warmest year on the record in 2019 (0.95 °C) which is only 0.04 °C less than the earth's highest temperature rise in 2016. According to the World Wide Fund (WWF), global warming will cause the species extinction, coastal erosion, coral bleaching, oceans acidifying and extreme weather event. Besides, global warming will lead to the rise in sea level due to the melting of glaciers and ice caps that increase the volume of water in the ocean. As a result, low-lying island and coastal cities will be drowned and disappeared.

Therefore, a lot of efforts have been given to counter the energy and pollution crisis that occurs throughout the world by shifting the source of energy supply from non-renewable towards green renewable energy sources. The International Renewable Energy Agency (IRENA) reported that global renewable energy continues to undergo a positive growth by additions of 171 gigawatts (GW) power in 2018. The increase in the production of power is mainly boosted by solar and wind energy. Other renewable energies such as bioenergy, geothermal, hydropower and ocean also contribute to the positive growth in the development of renewable energy worldwide. Among renewable energies, solar energy is the most promising renewable energy resource due to the unlimited supply of sunlight, facile fabrication process and high power generation efficiency (Shaikh *et al.*, 2017). Solar cells or photovoltaic (PV) cells produce electricity by harnessing the energy from the photon of sunlight. The produced energy is called solar energy and the discovery of the PV effect has begun as early in 1839 by Alexandre Becquerel. Solar energy is classified into four different generations. The first generation of solar cells is the most common solar cell available in the industry which is made of single and multi-crystalline silicon. However, due to high fabrication costs, the second generation of solar cells was introduced which is made of thin-film solar cells. The thickness of the solar cells of this generation

was reduced to nanometers in order to reduce the fabrication cost. In the meantime, the third generation such as dye-sensitized solar cells (DSSCs), quantum dots solar cells, organic solar cells and perovskite solar cells have gathered numerous attention due to the simple fabrication process and high power conversion efficiency (PCE). The emerging solar cells or fourth-generation solar cells consist of a combination of inorganic and organic materials to boost the PCE and lower the cost of the solar cells (Jayawardena *et al.*, 2013).

1.2 Problem statements

DSSCs are the third-generation solar cell that capable of converting sunlight into electrical energy where the source of electrons comes from the dye compared with the conventional solar cell where the main source of the electrons comes from the semiconductor (Chander *et al.*, 2015). Compared to other types of solar cells, DSSCs have attracted numerous attention due to their low fabrication cost, simple experimental design and moderate PCE. A complete DSSC consists of four main components which are photoanode, counter electrode, dye and electrolyte. Among them, photoanode plays a crucial role in the production of high PCE. Titanium dioxide (TiO_2) is the most common material used as a photoanode in DSSCs due to its high surface area and high porosity which is effective for the dye adsorption process (Kim *et al.*, 2012b). However, TiO_2 as the photoanodes in DSSC has faced a lot of drawbacks such as a small range of light scattering, high recombination effect and low charge transport ability that reduces its PCE.

The recombination effect is the process where the amount of photocurrent and voltage produced is reduced due to the unnecessary recombination. There are three possible routes of recombination in DSSC which are (i) the recombination between electrons of excited dyes and hole of dyes, (ii) the recombination between electrons of TiO_2 with the oxidized redox species and (iii) the recombination between the electrons of transparent conductive oxides (TCO) substrate and the redox electrolyte (Gregg *et al.*, 2001). The first and second recombination routes can be prevented using suitable types of dyes and suitable types of electrolytes, respectively while the third route of recombination can be overcome by introducing a compact layer in between the porous TiO_2 nanoparticles and TCO substrates. The compact layer must be thin, compact and conductive to reduce the recombination effect and facilitates the electron-hole regeneration process. The compact layer is mostly made of metal oxides or carbon-based materials because both materials are conductive and capable to form a thin and compact film. However, metal oxides are more preferable compared to carbon-based materials because the former can sustain high heat treatment during the preparation of photoanode. In this study, TiO_2 compact layer was successfully prepared and optimized using a heat treatment assisted electrospinning and response surface methodology with central composite design (RSM/CCD), respectively.

Furthermore, TiO_2 as the photoanode also suffers from a small range of light scattering and low charge transport and this problem can be overcome by introducing a light scattering layer (LSL) on top of the photoanodes. The LSL helps to trap more sunlight, resulting in more excitation of electrons and producing more photocurrent and voltage that leads to a higher PCE. The LSL must be conductive and larger (>250 nm) compared to the porous TiO_2 nanoparticles (10-30 nm) to enhance the light scattering effect. The large LSL is important to reflect the incident sunlight back to the sensitized TiO_2 film, resulting in an increase in the excitation of electrons and produce more photocurrent. In this study, two types of LSL were introduced i.e. polyvinyl alcohol/titanium dioxide (PVA/TiO_2) nanofibers and titanium dioxide decorated by graphene quantum dot (TiO_2 -GQD) LSL.

In addition, typical glass substrate based DSSC devices are rigid and inflexible, therefore the application is limited to flat surfaces such as rooftop and window. In order to overcome this problem, a fully flexible DSSC device made of TiO_2 nanoparticles with TiO_2 -GQD LSL on flexible plastic substrate indium doped tin oxide/polyethylene naphthalate (ITO/PEN) were introduced.

1.3 Objectives of research

The objectives of this research are:

1. To prepare and optimize the TiO_2 compact layer using heat treatment assisted electrospinning and response surface methodology.
2. To evaluate the effect of concentration of PVA and volume of titanium tetraisopropoxide on the DSSC performance of TiO_2 -PVA nanofibers as a light scatterer.
3. To optimize and evaluate the DSSC performance of TiO_2 decorated by GQD as a light scattering layer.
4. To develop and assess a fully flexible DSSC made of TiO_2 -GQD as a light scatterer.

1.4 Scope of study

This study focused on the preparation and optimization of the compact layer and LSL to enhance DSSC performance. The TiO_2 compact layer was prepared by heat treatment assisted electrospinning to overcome the recombination effect. The preparation of the TiO_2 compact layer was optimized using response surface methodology with central composite design (RSM/CCD). The first LSL was made of PVA/TiO_2 nanofibers that were successfully prepared via electrospinning and optimized using RSM/CCD, respectively. The second LSL i.e. TiO_2 -GQD was prepared via electrodeposition of TiO_2 and drop-casted of GQD. The preparation of TiO_2 -GQD LSL was optimized using RSM/CCD. The GQD was introduced as

an LSL due to unique photoluminescence properties that can broaden the light-harvesting range from the ultraviolet range to near infra-red range. A fully flexible DSSC device also was introduced as to widen the application of DSSCs due to its attractive traits such as flexible, lightweight, thin and capable to generate moderate PCE.

1.5 Organization of chapter

This thesis consists of 9 chapters and constructed as follows. Chapter 1 describes the background of the study, problem statements, objectives of research and scope of the study. Chapter 2 contains a comprehensive review of photoanodes for DSSCs where a detailed explanation of the compact layer and LSL is discussed. Chapter 3 discusses the optimization of the PVA/TiO₂ compact layer using RSM/CCD while, Chapter 4 elaborates on the characterization and DSSC performances of the PVA/TiO₂ compact layer. Chapters 5 elaborates the preparation and optimization of PVA/TiO₂ nanofibers as an LSL using electrospinning and RSM/CCD, respectively. The DSSC performances of PVA/TiO₂ nanofibers are discussed in Chapter 6. The optimization (RSM/CCD) and DSSC performance of TiO₂ decorated by GQD as a light scatterer are studied in Chapters 7 and 8 respectively. Chapter 9 reports the preparation and characterization of fully flexible DSSC consisting of TiO₂ decorated by GQD as an LSL. The last chapter describes the conclusions and recommendations to improve the DSSC performances.

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LIST OF PUBLICATIONS

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