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OPTIMIZED TITANIUM DIOXIDE PHOTOANODE ON FLEXIBLE SUBSTRATES FOR DYE-SENSITIZED SOLAR CELL

SURAYA SHABAN

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By

SURAYA SHABAN

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

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DEDICATION

This dissertation is wholeheartedly dedicated to;
my super-parents, Mrs Hasimah Hassan and Mr Shaban Abdullah,
also, to my benevolent siblings.
Thanks for the continued strength of support and fondness.
I love you.

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

OPTIMIZED TITANIUM DIOXIDE PHOTOANODE ON FLEXIBLE SUBSTRATES FOR DYE-SENSITIZED SOLAR CELL

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April 2020

Chairman : Associate Professor Suhaidi Shafie, PhD
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Dye-sensitized solar cell (DSSC) which is the third-generation solar cell that utilized dye molecules to absorb light and convert it to electrical energy has a promising efficiency with the ongoing research worldwide. Many implementations have been made through various substitution for materials, optimization on electrodes and assembly of the devices. Since the conventional solid-state DSSC is very rigid to the shape, therefore, the flexible DSSC was introduced. In this study, the flexible photoanode electrode was used to replace the FTO/glass as photoanode electrode. The flexible substrate used is indium tin oxide/polyethylene terephthalate (ITO/PET). However due to its character as a polymer, the limit sintering temperature is 150 ºC and it could not withstand the high temperature of 450 ºC, which is needed for the titanium dioxide (TiO_2) photoanode deposited on electrode to remove the binder used inside the paste, ensuring the particles between the TiO_2 is porous and enhance the mobility of electrons generated by N719 dye. Therefore, the free binder TiO_2 paste was introduced and deposited using doctor blade method, sintered at 120 ºC for 24 hours. However, the efficiency obtained is only 0.07 % which is due to due to weak adhesion to the substrate. Another approach of using flexible titanium (Ti) foil substrate is proposed to overcome the intolerance of high temperature of the ITO/PET as it could stand temperature of more than 450 ºC, lightweight and flexible at some degree which is suitable to substitute the ITO/PET. Since it is opaque, the back-illumination technique has been introduced. To improvise the performance, the optimization of the device was done by optimizing the thickness of the TiO_2 photoanode at 15.09 µm and N719 dye loading time for 45 hours. The thickness of the TiO_2 leads to better adsorption of dye N719 to collect photons and transfer the electrons while the thickness of Pt counter electrode optimized at volume of 50 µL with thickness of 10 nm after sintering, enhanced the absorption of lights. The result shows that Ti foil treated with H_2O_2 achieved efficiency of 1.00% exceeded the back illuminated FTO/glass due to the strong

particles interconnect between Ti foil surface and TiO_2 nanoparticles network that enhanced the electron transfer and reduce the electron recombination in the device.

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

FOTOANOD TITANIUM DIOKSIDA YANG DILAKSANAKAN PADA SUBSTRAT FLEKSIBEL UNTUK SEL SURIA TERPEKA PEWARNA

Oleh

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Sel solar peka pewarna (DSSC) merupakan sel suria generasi ketiga yang menggunakan molekul pewarna untuk menyerap cahaya dan mengubahnya menjadi tenaga elektrik dengan cekap melalui penyelidikan berterusan di seluruh dunia. Banyak pelaksanaan telah dilakukan melalui penggantian bahan, pengoptimuman pada elektrod dan pemasangan peranti. Oleh kerana DSSC konvensional kaku bentuknya, maka DSSC fleksibel diperkenalkan. Dalam kajian ini, elektrod fotoanod fleksibel digunakan untuk menggantikan FTO/kaca sebagai elektrod fotoanod. Substrat fleksibel yang digunakan ialah indium tin oxide/polyethylene terephthalate (ITO/PET). Namun kerana sifatnya polimer, had suhu sintering adalah 150 ºC dan tidak tahan dengan suhu tinggi 450 ºC yang diperlukan titanium dioksida (TiO_2) photoanode yang diaplikasikan pada elektrod untuk menghilangkan pengikat yang digunakan di dalam tampalan, memastikan zarah antara TiO_2 berliang dan meningkatkan mobiliti elektron yang dihasilkan oleh pewarna N719. Oleh itu, pes TiO_2 pengikat bebas diperkenalkan dan diaplikasi menggunakan kaedah doctor blade, disinter pada suhu 120 ºC selama 24 jam. Walau bagaimanapun, kecekapan yang diperoleh hanya 0.07% disebabkan oleh lekatan yang lemah pada substrat. Pendekatan lain menggunakan substrat kerajang titanium fleksibel (Ti) dicadangkan untuk mengatasi ketidak-tahanan suhu tinggi ITO/PET pada suhu 450 ºC, ringan dan fleksibel pada sudut tertentu. Oleh kerana fizikal legap, teknik pencahayaan belakang telah diperkenalkan. Untuk meningkatkan prestasi, pengoptimuman peranti dilakukan dengan mengoptimumkan ketebalan fotoanod TiO_2 pada 15.09 µm dan masa pemuatan pewarna N719 selama 45 jam. Ketebalan TiO_2 menyebabkan penjerapan pewarna N719 yang lebih baik untuk mengumpul foton dan memindahkan elektron, sementara ketebalan elektrod penghitung Pt dioptimumkan pada isipadu 50 µL dengan ketebalan 10 nm setelah pensinteran, meningkatkan penyerapan cahaya. Hasil Ti foil yang dirawat dengan H_2O_2 mencapai kecekapan 1.00% melebihi FTO/kaca yang diterangi

pencahayaan belakang kerana partikel yang kuat menghubungkan permukaan foil Ti dan rangkaian nanopartikel TiO_2 yang meningkatkan pemindahan elektron dan mengurangkan pengumpulan semula elektron dalam peranti.

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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 Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

CB	Conduction band
CIP	Cold Isostatic Pressure
CIGS	Copper indium gallium selenide
DC	Direct Current
DI	De-ionized
DSSC	Dye-Sensitized Solar Cells
EDS	Energy dispersive X-ray spectroscopy
FF	Fill factor
FTO	Fluorine-doped tin oxide
FESEM	Field emission scanning electron microscopy
HOMO	High occupied molecular orbital
I-V	Current to voltage
ITO	Indium-doped tin oxide
IPCE	Incident to photon efficiency
LUMO	Lowest unoccupied molecular orbital
PV	Photovoltaic
PEN	Polyethylene Naphthalate
PET	Polyethylene Terephthalate
PEDOT	Poly (3,4-ethydioxythiophene)
TCO	Transparent conductive oxide

CHAPTER 1

INTRODUCTION

1.1 Background

Photovoltaic (PV) is a technology that utilizes semiconductors to transform sunlight (solar radiation) into direct current electricity. There are several peer groups of photovoltaic technologies in solar energy. First-generation of the photovoltaic (PV) mono-crystalline and poly-crystalline silicon, it demonstrates typical efficiency of 47.1 %. The good performance of this solar cell technology as well as high stability is the benefits of this PV Cell. However, lots of energy and expensive production cost is needed [1]. Thin film solar cell is the next group using cadmium telluride (CdTe), copper indium gallium selenide (CIGS) also including amorphous silicon (a-Si), where efficiency at 23.4% was achieved [2]. Since it avoids the usage of silicon wafers, consumption of low amount material was used. High temperature treatments and vacuum involvements during production were implemented associate large energy consumption. Furthermore, low sufficient source for the demand and this is one are price restrain factor. The thin film CdTe solar cell production must be handled carefully as it is toxic for the human being [3]. Third-generation solar cell are mostly polymers, and organic of tiny molecules. It embeds the cost of first and second generation of solar cell which a record-breaker in laboratory efficiency. This type of PV has only some scope in trading application due to its high price. Perovskite solar cell is currently under investigation with great potential with efficiencies record outwit on very small area at 25.2 %. Despite that, vulnerability suffers, and inflexible process happens to Perovskite cell considering should be fabricated in strict uncontaminated area with humidity temperature control [4]. Dye-sensitized solar cell (DSSC) are in the third-generation PV group that exchange light energy to a voltaic energy construct on the dye molecules absorption kindred in wide bandgap of semiconductor film. DSSC received global attention due to its soaring energy conversion efficiency of 12.3% also moderate cost of production [5]. Utmost components key for common DSSC are wide band gap semiconductor, synthetic sensitizer to act as photoanode, catalyst, redox couple, and mechanical support. The photoanode which normally a dye particle coated together with the nano porous metal oxide semiconductor film deposited on the transparent conductive oxide (TCO) glass substrate as the working electrode, platinum (Pt) catalyst deposited on TCO substrate as the counter electrode and redox mediator iodide/tri-iodide (I^-/I_3^-) electrolyte [6]. Metal oxide semiconductor comprise besides titanium dioxide (TiO_2), are zinc oxide (ZnO), and tin oxide (SnO_2). The semiconductor engaged as light absorption and photo-induced charge separation transport are different in functions a DSSC [7]. In an ordinary, lights absorbed by molecule in synthetic dye sensitizer familiarly N719, which conducted to the n-type semiconductor wide band gap surface. The dye sensitizer is DSSC photographic component as when it is sensitized by any visible light, it will be converted to electricity. The dye excites electrons from ground state and then injects the excited electron to the nanocrystalline TiO_2 semiconductor after seized photons from any visible source. TCO glass with Pt coating counter electrode performs catalytic activity of I^-/I_3^- reduction. Electrolyte redox mediators assist to connect and reconstruct the oxidized dye. Most redox mediators with slow recombination rate with the injected electrons chosen are the I^-/I_3^- couple. The electrolyte closes the circuit as the electrons yield to the sensitizer. These electrons create energy and gather them to the load appliances [8].

A conventional DSSC is composed of two electrodes, the anode and the cathode. These electrodes are made from a specific glass that has a TCO coating on one side. The TCO material is a thin layer of fluorine-doped tin oxide, also called FTO. The transparency of the substrate allows sunlight to enter the cell while its conductive surface collects charges. The anode is the positive terminal of the solar cell. It essentially bears a continuous network of sintered TiO_2 nanoparticles. This porous network offers an inner surface that is a thousand times greater than the equivalent flat area, and acts like a “light sponge” in which sunlight can get trapped [9]. TiO_2 is a white semiconductor that is not sensitive to visible light. The TiO_2 nanoparticles must be sensitized with a layer of dye molecules that absorbed light in the visible spectrum. Some natural dyes can be employed, but the most efficient dyes were synthesized after intense scientific investigation. The negative terminal of the solar cell, also called the cathode, is coated with a catalytic material for electron transfer. In most cases, this catalyst is either Carbon or Platinum. Since a very small quantity of catalyst is needed, the electrode remains transparent. The space left between the two electrodes is filled with an electrolyte that ensures charge transportation through a redox couple. I^-/I_3^- in a nitrile solvent is typically used for this purpose. Eventually, the two electrodes are sealed together to prevent the electrolyte solvent from evaporating. However, the assembly can remain open when simplicity is preferred over durability [8, 9]. DSSC have attracted much attention for the last more than a decade since they were developed by Grätzel in 1980s because of their low-cost, environment friendliness and high conversion efficiency of solar energy into electrical energy compared to silicon PV that has higher efficiency compared up to 30%. The fabrications of DSSC are much easier and have good performance under 1 sun condition [10].

Flexible DSSC, based on the substrates of indium tin oxide (ITO) coated polyethylene terephthalate (PET), substituting for rigid glass substrates, are regarded as one possible breakthrough in the field of DSSC regarding their commercialization, because flexible DSSC have presented great advantages of low cost of production and wide application [11]. Conductive plastic substrates, such as ITO/PET can be processed by a continuous process like roll-to-roll production for porous nanocrystalline film coating, therefore, greatly decreasing the production cost of the solar cell. In addition, it is light weight, having portable character. ITO coated plastic (usually polyethylene naphthalene (PEN) and PET) is the most used flexible substrate, which has been attracted much attention because of its flexibility, lightweight and low cost [12]. There are various organic binder-free pastes for low temperature sintering for plastic DSSC [13].

Comparative studies of flexible DSSC on plastic [12] and [13] tabulated in Table 1.1. The sintering temperature for TiO_2 paste for plastic DSSC is 120 °C [14]. Some alteration made to the common DSSC regarding to the substrates as it has excellent electrical conductivity, metal materials were chosen. Aside, its bendability and pliable, at high thermal treatment stability at 450 °C [15] matched to the plastic, surface resistivity lower than ITO/PET, plus as inexpensive production cost puts metal a good substitute [16]. The implementation of metal as working electrode substrates of DSSC could minimize the internal resistance on substrate yet helps the productivity of the solar cell and mark down the cost of the device [17]. Various metals are carried out nowadays as substitution inclusive of W, StSt, Ni, Ti, Co, Pt, Cu, Al, and Zn [18].

Table 1.1 : Comparative studies of flexible DSSC on plastic

	Zhang, D. et al [12]	Yamaguchi, T. et al [13]
Article Title	Highly Efficient Plastic-Substrate Dye-Sensitized Solar Cells with Validated Conversion Efficiency of 7.6%	Organic-Free Anatase TiO_2 Paste for Efficient Plastic Dye-Sensitized Solar Cells and Low-Temperature Processed Perovskite Solar Cells
Efficiency	8 % with 0.25cm^2 cell Efficiency of 7.6 % with 1.111cm^2 cell	7.51 %
Novelty	Combines press method (without heat treatment) with TiO_2-water paste	Pure anatase TiO_2 nanoparticles and organic-free TiO_2-sol
Working Electrode	Plastic substrate ITO-PEN	Plastic substrate ITO-PEN
Surface Treatment	ITO-PEN film was examined using UV-O_3 treatment also on TiO_2 film	The electrodes were exposed to UV-irradiation from a 300W mercury lamp for 30 min (the temperature of the electrodes was detected to be around 90□C during the UV-irradiation)
Measurement Intensity	100mW/cm^2 (AM 1.5).	100mW/cm^2 (AM 1.5).
Tio_2 Paste	10 wt% nanocrystalline TiO_2 and water	Anatase TiO_2 colloid
Deposition Method	Combination of doctor blading and press method TiO_2 paste was coated onto an ITO-PEN film by the doctor-blade method using polyimide tape Press Method (without heat treatment)	Doctor-blade technique
Active Area	0.25 cm^2 and 1.111 cm^2	0.25 cm^2
Thickness of TiO_2	6-10 μm It had been difficult to fabricate TiO_2 films thicker than 8 mm due to exfoliation of the TiO_2 film.	11±0.2 μm
Sintering	After air-drying at room temperature, the TiO_2 photoelectrode was cut into desired shapes and then pressed for 1 min at room temperature	Drying at ambient temperature
Dye	N719	0.5mM N719
Sealant	Surlyn film (thickness:30 μm)	Surlyn film (thickness: 25 μm)
Counter Electrode	Conductive plastic film	Pt/ITO/PEN counter electrode
Electrolyte	Iodide Triiodide	Iodide Triiodide

(Source : Zhang, D. et al [12] and Yamaguchi, T. et al [13])

1.2 Problem Statement

TiO_2 paste for solid state is not suitable for ITO/PET or ITO/PEN substrate for low temperature sintering as the plastic-substrates have thermal instability at the high temperatures (450-550°C) which are required for sintering of the porous TiO_2 photo-electrodes of the DSSC. Attachment of low temperature processed TiO_2 layer to the PET is very weak which may detach in dye solution during dye immersion process. This thermal treatment eliminates organic residues and induces electrical contact between the particles and the substrate. Unfortunately, they should be treated below 150°C [19]. As mentioned above, most of plastic substrates will be deformed when sintering temperature is more than 150°C because the plastic-based electrode cannot withstand the high temperature [20]. When practical processing temperatures exceed 150 °C, the ITO/PET and ITO/PEN substrates start to deform, and even melt at 235 °C [21]. Therefore, low temperature sintering technique must be proposed to overcome the deformation of ITO/PET.

Metal substrate mostly used for its toleration to thermal process and the approachable method to use is backside illumination [22], but it is not optimal in DSSCs since the platinized counter electrode slightly reflects light while iodine in the electrolyte absorbs photons at lower wavelengths (400 – 600 nm) [23]. The use of iodine electrolyte is needed to complete the DSSC even it shows some disadvantages such as sealing problems and long-term durability, solvent evaporation and leakage of volatile organic solvent [24]. The reduction of absorption of light passing through the counter electrode affected the performance of the dye as the light received on TiO_2 photoanode is lessen. Optimization on thickness of TiO_2 photoanode is bring out to attributed to more dye absorption, which leads to more photon absorption, resulting in a higher photocurrent response of the cells. [25] Several optimizations on thickness of Pt, thickness of TiO_2, dye-loading time must be considered to overcome the problems.

Efficiency results for single optimized parameters may have resulted in varies output performance from all parameters when combined. Therefore, to analyse DSSC performance in term of power conversion efficiency from assembled cell optimized parameters, single test should be done for each parameter and then performance of assembled device combining all parameters would be analysed.

1.3 Objectives of the study

The aim of this research is to achieve high-efficiency flexible DSSC based on back-illuminated approach. The activities to achieve this target are;

(a) To optimize parameters and the suitable process for TiO_2 deposition on flexible photoanode electrode.
(b) To analyse the thickness of Platinum (Pt) catalyst deposited on counter electrode, thickness of TiO_2 and N719 dye-loading time on photoanode electrode for optimum light harvesting.
(c) To analyse DSSC performance in term of power conversion efficiency from assembled cell based on proposed flexible photoanode and optimized parameters.

1.4 Scope and limitation

This research work was carried out to study the performance of flexible photoanode to for DSSC. Two type of substrate were used namely, ITO coated PET and Ti foil.

(a) Since this research focus only on study of flexible substrates for photoanode, the counter electrode is set to remain as FTO coated glass as reference to the conventional DSSC.
(b) This research also utilized liquid electrolyte although the stability of fabricated DSSC may reduce due to corrosion since the quasi solid-state electrolyte is not available in the market.
(c) The attachment of Ti foil and Surlyn polymer spacer is weak which affected the stability of fabricated DSSC due to electrolyte leakage or short circuit.

1.5 Thesis outline

This research report is divided into five chapters.

Chapter one is the introductory part which discusses the background of the study. This chapter also elaborates on the problem statements, objectives of the study based on the study of DSSC, together with the scope and limitation of the research.

Chapter two presents the literature review on concept of the PV and extends the frame of its generation. Next, it focuses on the architecture of DSSC and the development of fabrication made to the flexible DSSC. The scope of literature review covers the related researches on flexible DSSC, mechanism and preparation of flexible photoanode. Literature review studies on the subject matter to provide various useful inputs in fabrication process methodology.

Chapter three explains the details of method to fabricate the DSSC. This chapter begins through the elaboration on the research performed. The procedure was divided into three parts, the preparation of photoanode, counter electrode and the assembly of the DSSC device. Lastly, the techniques for DSSC measurement setup to evaluate performance of the device according to the established standard characterization for solar cell efficiency.

Chapter four covers the analysis of the conventional and flexible DSSC from the experimental results. The discussions for the pre- and post-treatment on substrates has been carried out. This chapter concludes the investigation made of the results from various flexible photoanode of DSSC. Completing the chapter with thorough explanation on the analysis performed on the DSSC.

Chapter five is a summary of the outcome from the proposed research and further potential future work of DSSC. This chapter concludes the research and fabrication of the DSSC. Conclusions achieved are initially proposed research methodology and the collected experimental results. Finally, this chapter proposed recommendations and future works of DSSC.

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BIODATA OF STUDENT

Suraya Shaban was born in Kuala Lumpur, Malaysia in 1991. She received her certificates of her Diploma and Bachelor degree from Universiti Kuala Lumpur – British Malaysian Institute (UniKL BMI) in Engineering Technology (Hons) in Medical Electronics on 2012 and 2016 respectively. Since 2017, she has been with the Institute of Advanced Technology (ITMA), Universiti Putra Malaysia (UPM), where she is currently a Master student furthering her Master in Science (MSc). Her main areas of research interest are Energy Engineering specialized in Dye-Sensitized Solar Cell (DSSC). Suraya is one of the IEEE Student members, IEEE Women in Engineering (WiE), IEEE Young Professionals and IEEE Electron Device Society (EDS).

LIST OF PUBLICATIONS

Publication in Index Journal

Shaban, S., Shafie, S., Pandey S.S., Lokman, M.Q., Ahmad, F., Hamidon, M.N., & Sharif, N.F.M., "*Back-Illuminated Dye-Sensitized Solar Cell Flexible Photoanode on Titanium Foil*," ASM Sc. J., Vol. 12, Special Issue 4, 2019 for ICSE2018, pp.29-3, 2019 (Scopus Index)

Conference Proceedings

Shaban., S, Shafie, S., Pandey, S.S., "*Effect of Dye Loading Time to The Efficiency of Dye-Sensitized Solar Cell*," 7th International Symposium on Applied Engineering and Sciences (2019), 11 – 12 November 2019, Engineering Faculty, Universiti Putra Malaysia (UPM), Malaysia.

Shaban, S., Shafie, S., Mustafa, M.A., Pandey, S.S., Ahmad, F., Hamidon, M.N., Lokman, M.Q., Sharif, N.F.M., *"Efficiency Performance Effect of TiO_2 Thickness Deposited on FTO Coated Glass Photoanode,"* 4th IEEE International Circuit and System Symposium (ICSyS 2019), 18-19 September 2019, Hotel Istana, Kuala Lumpur, Malaysia.

Shaban, S., Shafie, S., Lokman, M.Q., Ahmad, F., *"Preparation of TiO_2 Photoanode on Flexible ITO Coated PET for Dye Sensitized Solar Cell,"* International Symposium on Advanced Materials and Nanotechnology (i-SAMN 2018), 15-16 August 2019, The Everly, Putrajaya, Malaysia.

Shaban, S., Shafie, S., Sulaiman, Y., Ahmad, F., Lokman, M.Q., & Sharif, N. F. M. *Flexible Photoanode on Titanium Foil for Back-Illuminated Dye Sensitized Solar Cells*. IEEE International Conference on Semiconductor Electronics (ICSE 2018), pp.197-200.15 – 17 August 2018, Pullman Kuala Lumpur City Centre Hotel & Residence

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