FABRICATION OF $BaTiO_3$, $BaSnO_3$ AND Sn-DOPED $BaTiO_3$ COMPACT LAYERS FOR ENHANCED DYE SENSITIZED SOLAR CELL

By

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January 2024

Chairman: **Associate Professor Yusran bin Sulaiman, PhD**
Institute: **Nanoscience and Nanotechnology**

Fossil fuel, non-renewable energy resources used to power up economic necessities need to be replaced with more efficient and renewable resources to reduce greenhouse gases and pollution. Solar energy is a renewable energy that has been studied and developed to explore opportunities to produce efficient and reliable devices to produce electricity. Dye-sensitized solar cells (DSSC) are third-generation solar cells that have an easy fabrication procedure, are low-cost and have good compatibility with flexible substrates. With the aim to replace the commonly used TiO_2 as a photoanode in DSSCs, perovskite oxides have attracted considerable attention to be used in DSSCs. Herein, $BaTiO_3$ (BTO), $BaSnO_3$ (BSO) and Sn-doped $BaTiO_3$ (BTSO) were designed and fabricated as a compact layer in DSSC using a spin-coating method. The experiment was designed using response surface methodology with Box-Behnken design (RSM/BBD), consisting of three independent variables to analyze the optimum deposition condition. The interaction

between these factors was studied and used to identify the best parameters for DSSC performance. FESEM images of the compact layer showed that an ultrathin layer of the perovskite oxide was fabricated on the ITO substrate. FTIR spectra revealed that the distinctive peaks for each compact layer were present. The presence of the thin perovskite oxide on the ITO substrate was further validated using XPS measurement. The addition of those compact layers successfully reduced the leakage current, proofing that the perovskite oxides can be used as a good blocking material to suppress the back electron movement from the transparent conductive oxide to the electrolyte. DSSC with compact layers revealed a higher back charge transfer resistance between metal oxide/dye and electrolyte and longer electron lifetime (τ_e) compared to DSSC without compact layers, illustrating that the recombination effect was decreased and the compact layer aids in the separation of photogenerated charge carriers to increase the photovoltaic performance of the solar cell. The optimized conditions for the compact layers were as follows: $BaTiO_3$ (annealing duration: 3 hours, annealing temperature: 485°C and number of dropcasting: 3 times), $BaSnO_3$ (annealing duration: 2 hours, annealing temperature: 540°C and number of dropcasting: 2 times) and Sn-doped $BaTiO_3$ (annealing temperature: 460°C, percentage of Sn precursor: 41% and number of dropcasting: 3 times). All the perovskite oxide-based compact layers illustrated a good fit towards the quadratic model in the Box-Behnken design with an appreciable coefficient of determination (R^2). All three compact layers attained enhanced photocurrent of 12.02 mA cm^{-2}, 12.40 mA cm^{-2} and 14.70 mA cm^{-2} compared to DSSC without a compact layer with photocurrent of 9.60 mA cm^{-2}. The improvement in the photocurrent was attained due to the increase in the concentration of photogenerated charge carriers, improving the efficiency of the DSSC. DSSC containing the BTO, BSO and BTSO compact layers exhibited enhancement of power conversion efficiency (PCE) of 4.95%, 4.82% and 5.33%, respectively compared to DSSC without a compact layer (3.54%). Thus, BTO, BSO and BTSO compact layers can be used to accelerate the generation and transportation of charge carriers and suppress the recombination.

Keywords: Compact layer, Dye-sensitized solar cell, Recombination

SDG: GOAL 7: Affordable and Clean Energy

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

PEMBENTUKAN LAPISAN PADAT BaTiO3, BaSnO3 DAN BaTiO3 DIDOP Sn UNTUK MENINGKATKAN PRESTASI SEL SOLAR BERKEPEKAAN PEWARNA

Oleh

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Bahan api fosil, sumber tenaga yang tidak boleh diperbaharui yang digunakan untuk memperkasakan keperluan ekonomi perlu digantikan dengan sumber yang lebih cekap dan boleh diperbaharui untuk mengurangkan gas rumah hijau dan pencemaran. Tenaga suria adalah tenaga yang boleh diperbaharui yang dikaji dan dibangunkan untuk meneroka peluang menghasilkan peranti yang cekap dan boleh dipercayai untuk menghasilkan tenaga elektrik. Sel solar berkepekaan pewarna (DSSC) ialah sel solar generasi ketiga yang mempunyai prosedur fabrikasi yang mudah, kos rendah dan mempunyai keserasian yang baik dengan substrat yang fleksibel. Dengan tujuan untuk menggantikan TiO_2 yang digunakan secara menyeluruh sebagai fotoanod dalam DSSC, perovskit oksida telah menarik perhatian ramai untuk digunakan dalam DSSC. Dengan ini, $BaTiO_3$ (BTO), $BaSnO_3$ (BSO) dan Sn-didop $BaTiO_3$ (BTSO) telah direka bentuk dan digunakan sebagai lapisan padat dalam DSSC dengan menggunakan kaedah salutan putaran. Setiap kajian telah direka bentuk dengan

menggunakan metodologi permukaan tindak balas dengan reka bentuk Box-Behnken (RSM/BBD), yang terdiri daripada tiga pemboleh ubah bebas untuk menganalisis keadaan pemendapan yang optimum. Keadaan optimum untuk lapisan padat adalah seperti berikut: $BaTiO_3$ (tempoh penyepuhlindapan: 3 jam, suhu penyepuhlindapan: 485°C dan bilangan titisan: 3 kali), $BaSnO_3$ (tempoh penyepuhlindapan: 2 jam, suhu penyepuhlindapan: 540°C dan bilangan titisan: 2 kali) dan $BaTiO_3$ terdop Sn (suhu penyepuhlindapan: 460°C, peratusan prekursor Sn: 41% dan bilangan titisan: 3 kali). Semua lapisan nipis yang berdasarkan perovskit oksida menunjukkan kesesuaian yang baik terhadap model kuadratik dalam reka bentuk Box-Behnken dengan pekali penentuan yang ketara (R^2). Interaksi antara faktor-faktor ini telah dikaji dan digunakan untuk mengenalpasti parameter terbaik untuk menilai prestasi DSSC. Imej FESEM bagi lapisan padat menunjukkan bahawa lapisan nipis perovskite oksida telah dihasilkan di atas substrat ITO. Spektrum FTIR mendedahkan bahawa puncak tersendiri hadir bagi setiap lapisan padat. Kehadiran perovskite oksida nipis pada substrat ITO telah disahkan lagi menggunakan pengukuran analisis XPS. Penambahan lapisan padat tersebut berjaya mengurangkan arus bocor, membuktikan bahawa perovskite oksida boleh digunakan sebagai bahan penyekat yang baik untuk menyekat pergerakan elektron ke arah belakang daripada oksida konduktif lutsinar kepada elektrolit. DSSC dengan lapisan padat mendedahkan rintangan pemindahan cas belakang yang lebih tinggi antara oksida logam/molekul organik pewarna dan elektrolit serta jangka hayat elektron (τ_e) yang lebih lama berbanding DSSC tanpa lapisan padat, menggambarkan bahawa kesan pengabungan semula telah berkurangan dan lapisan padat membantu dalam pengasingan cas terjana disebabkan oleh penjanaan cahaya untuk meningkatkan prestasi sel suria. Ketiga-tiga lapisan padat dapat mencapai arus cahaya yang lebih tinggi sebanyak 12.02 mA cm^{-2} (BTO), 12.40 mA cm^{-2} (BSO) and 14.70 mA cm^{-2} (BTSO) berbanding dengan DSSC tanpa lapisan padat iaitu sebanyak 9.60 mA cm^{-2}. Peningkatan dalam arus cahaya dicapai disebabkan oleh peningkatan dalam kepekatan pembawa cas cahaya yang terjana,meningkatan kecekapan DSSC. DSSC yang mengandungi lapisan padat BTO, BSO dan BTSO masing-masing mempamerkan peningkatan kecekapan penukaran kuasa (PCE) sebanyak 4.95%, 4.82% dan 5.33%berbanding dengan DSSC tanpa lapisan padat (3.54%). Oleh itu, lapisan padat BTO, BSO dan BTSO boleh digunakan utuk mempercepatkan penjanaan dan pengangkutan pambawa cas dan menyekat penggabungan cas yang berlaku antara permukaan logam oksida/bahan organic pewarna/elektrolit.

Kata Kunci: Lapisan padat, Penggabungan semula, Sel solar berkepekaan pewarna

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LIST OF SYMBOLS

Symbol		Unit
ΔE_P	Peak to peak separation	mV
FF	Fill factor	%
f_{max}	Maximum frequency	Hz
I_P	Anodic peak current	mA cm^{-2}
I_{sc}	Short circuit current	mA
J_{sc}	Short circuit current density	mA cm^{-2}
PCE	Power conversion efficiency	%
R^2	Coefficient of determination	
R_{ct}	Charge transfer resistance	Ω cm^2
R_s	Series resistance	Ω cm^2
V_{oc}	Open circuit voltage	V
η	Charge collection efficiency	%
τ_e	Electron lifetime	ms

LIST OF ABBREVIATION

AM	Air mass
ANOVA	Analysis of variance
BBD	Box-Behnken design
BSO	Barium stannate ($BaSnO_3$)
BTO	Barium titanate ($BaTiO_3$)
BTSO	Sn-doped barium titanate
CCD	Central composite design
CNT	Carbon nanotube
CV	Cyclic voltammetry
DFT	Density-functional theory
DSSC	Dye-sensitized solar cell
EDA	Ethylenediamine
EDX	Energy dispersive X-ray spectroscopy
EIS	Electrochemical impedance spectroscopy
FESEM	Field-emission scanning electron microscopy
FTIR	Fourier-transform infrared spectroscopy
FTO	Fluorine-doped tin oxide
ILCT	Intra-ligand charge transfer
J-V	Current density-voltage
MLCT	Metal-ligand charge transfer
MOF	Metal-organic framework
N719	Ruthenizer 535-bis TBA

NIR	Near infrared
OSC	Organic solar cell
PEG	Poly-ethylene glycol
PSC	Perovskite solar cell
P-V	Power-voltage
PVA	Poly-vinyl alcohol
Rgo	Reduced graphene oxide
RSM	Response surface methodology
TCO	Transparent conductive oxide
TDIP	Titanium di-isopropoxide bis-acetylacetonate
TTIP	Titanium tetra-isopropoxide
UV	Ultraviolet
Vis	Visible
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Research background

Fossil fuels are made up of decomposed plants and animals from millions of years ago. These fossil fuels have been responsible for powering up the economy for over 150 years. However, fossil fuels are non-renewable energy which need hundreds of millions of years to form. Besides, upon burning, fossil fuel produces harmful substances, including carbon dioxide, sulfur dioxide, nitrogen oxide and particle pollution. Carbon dioxide is a well-known greenhouse gas that traps heat in the earth's atmosphere. This greenhouse gas potentially increases the effect of global warming on the surface of the earth. The greenhouse gas absorbs sunlight that has bounced off the earth's surface, which supposedly needs to escape to space. Greenhouse gases are the main factor that leads to climate change, causing the thinning of icebergs, extremely hot weather, and stress to the ecosystem. Thus, the need to replace this non-renewable source with a renewable source is required. Solar energy is an abundant, renewable, and cleanest source of energy. Solar and wind energy were reported to contribute about 14.7% and 11.4%, respectively to global power generation (IEA, 2023). Solar energy is harnessed to convert it into electrical energy, powering houses, businesses, schools, and hospitals. With the goal of transitioning to renewable energy, solar cells are crucial for converting light energy into electrical energy with low cost and high efficiency. Despite the benefits associated with solar energy, there are also challenges to implementing solar energy as the main source of energy. Some of the main challenges are the cost of installation, sensitivity toward high temperatures and lower lifespan. Different types of solar cells have been established in the market, which include monocrystalline solar panels, polycrystalline solar panels, thin film: amorphous silicon solar panels, and concentrated photovoltaic cells. However, research has been continuously expanding in recent years, bringing opportunities for new photovoltaic technologies. Solid-state photovoltaic devices have predominantly been used to construct robust large-scale solar electricity production. Conventional solid-state photovoltaic technologies utilize semiconductors and silicon to produce efficient and reliable commercialized solar devices. Pure semiconductor materials such as silicon and germanium have electrical properties which are in between conductor and insulator. By controlling the number of impurities in the pure semiconductor materials, the conductivity of the material could be improved. Various impurities, either donors or acceptors can be added to the pure semiconductor material to produce free electrons or holes, respectively. The p-n junction with built-in electric fields is generated when n-type material (semiconductor doped with donors) and p-type material (semiconductor doped with acceptor) are assembled to produce photogenerated electron-hole pairs. The development of dye-sensitized solar cells with the use of molecular components has challenged the use of conventional solid-state solar cells, recording efficiencies of up to 12%. Dye-sensitized solar cells (DSSC) are the photovoltaic device that belongs to the emerging photovoltaics. The performance of DSSC is dependent on the architecture of the device and varies according to technological design criteria. The integration of new solar cell technologies into structures such as building, greenhouses, airplanes, sails, textile, or interior applications that requires flexible and semi-transparent devices is the current focus of the research. In the last decade, the amount of research being conducted and published on DSSCs has grown tremendously. This type of solar cell offer dvantages, which include easy fabrication procedure, low manufacturing cost and excellent compatibility with flexible substrate.

1.2 Problem statement

DSSCs mainly depend on the components in the device, where photoanode is the main active material that plays a pivotal role in ensuring light energy is converted to electrical energy. Various materials have been explored as possible photoanodes in DSSCs. A typical photoanode has high electron mobility, and a large surface area to ensure systematic charge transport, improving the charge collection efficiency. Titanium dioxide, TiO_2 has been used primarily as an active material in photoanode to anchor dye and transport electrons towards the conducting substrate. TiO_2 is well known for its large band gaps with suitable band edge levels, which is important for charge injection and extraction, and the long lifespan of photo-excited electrons. Furthermore, TiO_2 also has high resistance towards photo corrosion, low cost of fabrication and is non-toxic to the environment. However, TiO_2 suffers from the capacity to absorb light in the visible range (Pitre, Yoon, & Scaiano, 2017) and has low electron mobility (Ahmad, Rahim, & Pandey, 2018). Despite the great advantage of TiO_2 as a photoanode in DSSCs, the drawback in the material must be resolved to produce efficient solar cells. To overcome the drawback, different methods were explored to improve the properties of the photoanode, such as interfacial engineering, light scattering application, compact layer application, doping and introduction of composite.

A compact layer is a thin and homogenous layer added before the mesoporous TiO_2 layer to minimize undesirable charge recombination between the electron and hole which produces dark current and degrades the power conversion efficiency. Besides, the compact layer is added to prevent the electrolyte from being in contact with the TCO substrate. Different materials have been used as compact layers such as titanium dioxide, niobium pentoxide, zinc oxide, and cadmium oxide. Perovskite oxides are considered possible materials used as compact layers in DSSC. Recently, perovskite oxides have attracted a lot of interest as promising materials in optoelectronic applications. The compositional and structural flexibilities of perovskite oxide can be tuned by controlling the oxidation state of metal elements and the concentration of oxygen vacancy in perovskite oxide (J. Li et al., 2023). The ratio of the precursor metallic element can be controlled to modify the structure of the material according to the suitability of the application. Due to the remarkable ferroelectric properties, non-toxicity, low cost, and increased electron mobility characteristics of the material, perovskite oxides have been considered as a compact layer. The ferroelectric material changes the polarization in the electric field by regulating ion and charge transport (L. Yang et al., 2018). The enhancement in the separation of charge carriers, reduces charge recombination, improving the performance of the device.

Among various perovskite oxides, $BaTiO_3$ (BTO), $BaSnO_3$ (BSO) and Sn-doped $BaTiO_3$ (BTSO) are promising candidates which can be used as compact layers in DSSCs. BTO is the first polycrystalline perovskite oxide exhibiting ferroelectricity properties (Yogamalar, Kalpana, Senthil, & Chithambararaj, 2018). BTO exhibits unique properties given to its structure, such as chemically and mechanically stable, high dielectric constant, and Curie temperature (120°C). Besides, BSO shows the advantage of having high charge carrier mobility, faster dye sensitizing capability and high stability at higher temperatures. The doping mechanism introduced in the BTO structure helps to improve ferroelectricity properties and electron mobility, enhancing the performance of the device (Shi, Xie, Gu, & Zhu, 2015). Thus, the addition of the perovskite oxide in DSSCs is believed to deliver significant enhancement towards the photovoltaic performance such as photocurrent, open-circuit voltage, and photon-to-current efficiency.

Response surface methodology (Ondersma & Hamann, 2013) is a mathematical and statistical modeling technique used to design and optimize experiments (Ondersma & Hamann, 2013). The main objective of RSM is to minimize the number of experiments related to the response by applying appropriate statistical techniques based on factorial design such as central composite design (CCD) or Box-Behnken Design (BBD). RSM is able to search parameters that influence the measurement and evaluate the importance of the process parameters and quality of results in the experiments. (Darvishmotevalli, Zarei, Moradnia, Noorisepehr, & Mohammadi, 2019). RSM investigates the approximation relationship between input and output variables and identifies optimal operating conditions for a system under the study of the factor region that satisfies the operating requirement (Goren, Recepoğlu, & Khataee, 2022). The optimization of the perovskite oxide-based compact layer was implemented by using response surface methodology and Box Behnken design (RSM/BBD). RSM/BBD is a collection of statistical techniques that helps to optimize the performance of a system by finding the optimal values of input factors.

1.3 Objectives

The aim of this research is to prepare and investigate the photovoltaic performances of perovskite oxide-based compact layer for DSSC. The objectives of this research are:

1. To optimize the preparation of $BaTiO_3$, $BaSnO_3$ and Sn-doped $BaTiO_3$ as compact layers in DSSC using response surface methodology/Box-Behnken design to produce the highest power conversion efficiency of the device.
2. To evaluate the physicochemical properties of compact layers using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), and X-ray photoelectron spectroscopy (XPS), Ultraviolet-visible spectroscopy (UV-Vis) and photoluminescence (PL)
3. To assess the electrochemical performance of DSSC by using $BaTiO_3$, $BaSnO_3$ and Sn-doped $BaTiO_3$ as compact layers in DSSC.
4. To evaluate the photovoltaic performance of $BaTiO_3$, $BaSnO_3$ and Sn-doped $BaTiO_3$ as compact layers in DSSC.

1.4 Scope of study

This study focused on enhancing the photovoltaic performance using BTO, BSO and BTSO as compact layers in DSSC. The BTO, BSO and BTSO compact layer was fabricated using spin-coating. The spin-coating parameters, i.e. number of drop-casting times, annealing temperature and annealing duration and percentage of Sn precursor were optimized using response surface methodology and Box Behnken design. Sn dopant was introduced in $BaTiO_3$ due to better conductivity to enhance electron mobility and unique absorption properties that can improve the light harnessing range. The solar cell devices with BTO, BSO and BTSO compact layers were assembled, and its photovoltaic performance was further evaluated using electrochemical measurements such as current density-voltage (J-V), power-voltage (P-V) measurement, cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS).

1.5 Organization of study

This thesis consists of five chapters. Chapter 1 discusses the background of research on sources of renewable energy, the problem statement, and research objectives. Chapter 2 reviews the fundamentals of solar cells and recent progress in photoanodes and methods used to fabricate compact layers. Chapter 3 elaborates on materials and general procedures executed in the research. Chapter 4 describes the preparation, optimization, and application of BTO, BSO and BTSO as compact layers in DSSCs. Chapter 5 provides the overall summary of the findings, conclusions, and prospects for future work in this study.

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