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PREPARATION AND PHOTOELECTROCHEMICAL PROPERTIES OF NANOSTRUCTURED MANGANESE-DOPED CADMIUM SULFIDE/ TITANIA FOR SOLAR CELL APPLICATION

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By

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May 2018

Chairman : Zulkarnain Zainal, PhD
Faculty : Science

The outstanding properties of nanocrystalline semiconductor, titanium dioxide (TiO_2) such as low production cost, good chemical stability, non-toxicity and high photocurrent efficiency make it well accepted to mediate solar energy conversion. However, TiO_2 has limitation in absorbing sunlight's visible spectrum due to its wide band gap. Moreover, there are issues such as high proportion of recombination of photogenerated electron-hole pairs which highly influence the photoelectrochemical performance. To overcome these problem inorganic sensitisation using metal chalcogenides were widely explored. CdS attracted so much interest in photoelectrochemical cell applications due to its high absorption coefficient, good direct band gap and excellent conversion efficiency. In this research, the effect of doping of manganese on the photoelectrochemical performance CdS sensitised TiO_2 was studied. The viscous paste of TiO_2 nanopowder was coated on the flourine-doped SnO_2 conductive glass via doctor-blade method, followed by calcination at 500 ^{o}C. Polyester decomposition during calcination was found to be responsible for the formation of high quality crack free strongly adherent without delamination films.

CdS was grown on TiO_2 by successive ionic layer adsorption (SILAR) technique by dipping the TiO_2 electrode alternately in two different solutions containing Cd^{2+} and S^{2-} precursors. SILAR technique has been carried out at varying SILAR cycles, dipping time, pH, concentrations of cationic precursor and annealing temperatures. CdS/TiO_2 prepared by depositing CdS using seven SILAR cycles with one min dipping time was found to show optimum optical absorption with the band gap of 2.238 eV. X-ray diffraction pattern showed that the deposited CdS on TiO_2 were polycrystalline with cubic structure. The CdS/TiO_2 was found to be n-type based on the photoelectrochemical (PEC) results.

Mn-doped CdS was prepared using SILAR technique by introducing manganese salt into the cationic precursor solution in an effort to improve the photocurrent generation. The electrode fabrication was done at different SILAR cycles, manganese concentrations and annealing temperature. XRD patterns showed that the incorporation of manganese in CdS matrix of nanoporous TiO_2 structure shifted the diffraction peak of CdS to lower angle. The optical absorption results reveal that Mn-doping greatly enhanced the light absorption in the visible region. The band gap energy dropped from 2.238 eV to 1.680 eV. The photocurrent of the thin film increase by more than 30 % from 3.613 mA/cm^2 for CdS/TiO_2 to 5.477 mA/cm^2 for Mn-doped CdS/TiO_2. Mn-doped CdS/TiO_2 clearly showed tremendous increased in the photocurrent by more than 60 times compared to TiO_2 nanoparticles. Besides, other transition metals such as chromium, nickel and copper were doped with CdS on TiO_2 nanoparticles to compare the effect on the photoelectrochemical performance. It was found out that Mn doped CdS achieved the highest photoconversion efficiency with the value 4.025 % among the transition metals.

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

PENYEDIAAN DAN PENCIRIAN FOTOELEKTROKIMIA MANGAN-DOP KADMIUM SULFIDA/TITANIA BERSTRUKTUR NANO UNTUK APLIKASI SOLAR SEL

Oleh

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Kelebihan semikonduktor nanokristal titanium dioksida (TiO_2) adalah kerana kos pengeluarannya yang rendah, kestabilan kimia yang baik, tidak toksik dan keberkesanan fotoelektrokimia yang tinggi telah menjadikannya diterima baik sebagai bahan pengantara dalam pernukaran tenaga solar. Walaubagaimanapun, TiO_2 mempunyai had dalam menyerap spektrum cahaya nampak kerana luang tenaga jalur yang besar. Selain itu, terdapat isu seperti perkadaran yang tinggi dalam penggabungan semula elektron-lubang selepas fotogenerasi amat mempengaruhi prestasi fotoelektrokimianya. Untuk mengatasi masalah ini pemekaan tak organik dengan menggunakan logam kalkogenida diterokai secara meluas. CdS telah menarik banyak minat dalam aplikasi sel fotoelektrokimia disebabkan oleh pekali penyerapan yang tinggi, luang tenaga jalur terus yang baik dan keberkesanan penukaran yang sangat baik. Di dalam kajian ini, kesan pendopan mangan ke atas prestasi fotoelektrokimia CdS/TiO_2 dikaji. Campuran TiO_2 yang likat daripada serbuk nano TiO_2 telah disalut ke atas kaca kondukif SnO_2 terdop florin melalui kaedah pangacuan pita, diikuti dengan rawatan haba pada suhu 500 ^oC. Faktor yang memberi kesan kepada ciri-ciri fotoelektrokimia seperti kaedah pengendapan, nombor lapisan pengendapan dan rawatan $TiCl_4$ telah dikaji. Penghuraian polyester ketika rawatan haba bertanggungjawab dalam pembentukan filem yang berkualiti tinggi, tidak mudah retak dan melekat kuat tanpa pengelupasan.

CdS telah diendapkan di atas TiO_2 dengan menggunakan kaedah tindakbalas penjerapan lapisan ion berturut (SILAR) dengan elektrod TiO_2 direndamkan berselang-seli di dalam dua larutan yang berlainan yang mengandungi Cd^{2+} dan S^{2}. Teknik SILAR telah dilakukan dengan membezakan kitaran SILAR, masa rendaman, pH, kepekatan kation dan suhu rawatan haba. CdS/TiO_2 disediakan dengan mengendapkan CdS sebanyak tujuh lapisan SILAR dalam satu minit masa rendaman dan didapati sampel ini menunjukkan penyerapan optik yang optimum dengan nilai jalur ruang optik 2.238 eV. Keputusan pembelauan sinar-X menunjukkan pengendapan CdS di atas TiO_2 merupakan

polikristal yang berstruktur kubus. CdS/TiO_2 merupakan semikonduktor jenis n berasaskan keputusan ciri-ciri fotoelektrokimia (PEC).

Mn didopkan dengan CdS telah disediakan melalui teknik SILAR dengan memperkenalkan garam mangan ke dalam larutan kation dalam usaha meningkatkan kadar fotoarus. Fabrikasi elektrod telah dilakukan dengan membezakan kitaran SILAR, kepekatan mangan dan suhu rawatan haba. Corak pembelauan XRD menunjukkan gabungan mangan di dalam matriks CdS dan liang TiO_2 yang berstruktur nano telah mengalih puncak pembelauan CdS ke sudut yang lebih rendah. Serapan optik mendedahkan bahawa Mn banyak meningkatkan serapan cahaya pada kawasan nampak. Tenaga jalur ruang didapati telah jatuh dari 2.238 ke 1.680 eV. Prestasi fotoelektrokimia lapisan nipis meningkat lebih daripada 30 % daripada 3.613 mA/cm^2 untuk CdS/TiO_2 ke 5.477 mA/cm^2 untuk Mn-dop CdS/TiO_2. Mn dop CdS/TiO_2 jelas menunjukkan peningkatan yang besar di dalam fotoarus melebihi 60 kali ganda berbanding dengan nanopartikel TiO_2. Di samping itu, logam peralihan yang lain seprti kromium, nikel dan kuprum juga didopkan dengan CdS di atas nanozarah TiO_2 untuk membandingkan kesan prestasi fotoelektrokima. Didapati Mn terdop CdS mencapai kecekapan fotopenukaran yang paling tinggi dengan nilai 4.025 % berbanding logam peralihan yang lain.

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I certify that a Thesis Examination Committee has met on 8 May 2018 to conduct the final examination of Wardatun Nadrah binti Mohd Amin on her thesis entitled "Preparation and Photoelectrochemical Properties of Nanostructured Manganese-Doped Cadmium Sulfide/Titania for Solar Cell Application" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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4CT	4 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles
5CT	5 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles
7CT	7 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles
9CT	9 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles
11CT	11 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles
7CT20	7 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles for 20 s dipping time
7CT40	7 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles for 40 s dipping time
7CT60	7 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles for 60 s dipping time
7CT80	7 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles for 80 s dipping time
7CT100	7 cycles successive ionic layer adsorption reaction of cadmium sulphide on titanium oxide nanoparticles for 100 s dipping time
CA	Citric acid
CBD	Chemical bath deposition
DSSC	Dye-sensitized solar cell
E_c	Conduction band
EDX	Energy Dispersion X-ray
EG	Ethylene glycol
E_f	Fermi level
E_V	Valence band
FESEM	Field Emission Scanning Electron Microscopy

FTO	Fluorine doped Tin Oxide
HRTEM	High Resolution Transmission Electron Microscopy
hν	Photon energy
I.I	Impact Ionization
JCPDS	Joint Committee of Power Diffraction Standard
LSPV	Linear Sweep Photovoltammetry
QDSSC	Quantum dot-sensitized solar cell
QD	Quantum dot
SILAR	Successive ionic layer adsorption reaction
TNT	Titania nanotubes
UV-VIS	Ultraviolet visible
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 General introduction

The rapid industrialization and high population growth are the two major factors which contribute to the global energy crisis and therefore accelerate the research on renewable energy. Over the past few decades, solar energy, biomass and geothermal energy are emerging as alternative energy sources. Out of the mix, solar energy is the renewable and clean type of energy that offers an answer to the increasing concern of global warming and greenhouse effect (Kabir et al., 2018). It is also definitely an abundant resource with rapid declining conversion cost and thus attracts many researchers for its use in various theoretical and experimental studies (Jun et al., 2014).

Solar cell is highly regarded as an alternative renewable energy candidate in the current market. Solar cells based on silicon wafer so-called first generation solar cell technology is the most popular solar cells in the market which can reach solar cell efficiency as high as 29% (Blakers et al., 2013). Moreover, they are expensive and more at risk to lose some of their efficiency at higher temperature (Ubani et al., 2017). Alternatively, solar cell utilized with inorganic thin film is the second generation solar cell which offer cost reduction in manufacturing procedure but this type of cell exhibits relatively lower than 14% efficiency (Jun et al., 2013). Chronologically, the invention of the third-generation of high–efficiency thin film solar cell is to reduce cost by significantly increasing efficiencies by 15-20 % (Conibeer, 2007).

High efficiency thin film solar cell technology is a reliable technology to compete with the silicon wafer solar cell which now makes up to 90 % of the global market. It satisfied minimum material usage with acceptable efficiency to allow high market penetration of solar electricity (Lee et al., 2017). Therefore, studies on high efficiency thin film semiconductor materials such as ZnO (Pietruszka et al., 2015), TiO_2 (Peng et al., 2013) and carbon based species (Uddin et al., 2013) have attracted immense interest of worldwide researchers in order to reduce cost and enhance the capability in the solar cell technology. Semiconductor materials capable to incorporate with a small band gap sensitizing agent such as metal chacolgenides and dyes which are responsive to visible light. The sensitizing agents improved the photocurrent by the electron injection properties of the semiconductor materials. Moreover, the low band gap of inorganic material is able to suppress the recombination of photogenerated electron-hole pairs in wide band gap semiconductor materials which eventually exhibit higher performance of photoelectrochemical cell (Zhang et al., 2017) .

Many methods has been employed to deposit inorganic metal chacolgenide such as chemical bath deposition (Chen et al., 2010), successive ionic layer adsorption reaction (SILAR) (Badawi et al., 2016), electrochemical deposition and hydrothermal (Song et al., 2018) methods. The low cost SILAR technique is one of the effective ways due to its ability to control nanocrystal size and distribution of the deposited metal chacolgenide by parameter optimization such as precursor concentration and number of SILAR cycles. In this study, CdS as a sensitizer was modified by introducing Mn and deposited on TiO_2 nanoparticles for high performance photoelectrochemical cell via SILAR method.

1.2 Problem Statements

In recent years, many semiconductor-sensitized material has been reported for solar cell application (Zhu et al., 2000). TiO_2 is one of the semiconductors that has been widely used to deliver high efficiency photoelectrochemical cell due to its good chemical stability, low cost production, high corrosion resistance, high photocatalytic activities and good charge transport which play an important role in the solar cell performance (Bhat et al., 2017). However, TiO_2 has limitation in absorbing sunlight's visible spectrum due to its wide bandgap that restricts the photoactivation to only ultraviolet region.

One of the alternative ways to extend the optical absorption of TiO_2 is by coupling with organic dyes. Dyes as sensitizers have attracted high attention due to their low fabrication cost and high efficiency, flexibility in colour, shape and transparency (Golobostanfard et al., 2014). Dyes used in DSSCs are extremely efficient at converting absorbed photons into free electron in the titanium oxide layer. However, the photocurrent is limited to the amount of photons that can actually be absorbed by the dye. Typically dyes have poorer absorption compared to silicon, meaning that fewer of the photons in sunlight are available for current generation in comparison to silicon.

As an alternative to dye, inorganic metal chalcogenides have been attracted high attention among researchers owing to their great stability, good absorption over wider wavelength range and multiple exciton generation that leads to the high production of power efficiencies (Xu et al., 2013; Yang et al., 2011). Many of inorganic semiconductor materials have been used in order to enhance the photoelectrochemical performance such as CdTe (Bao et al., 2017), CdSe (Jun et al., 2014) and PbS (Shi et al., 2015). All these unique characteristics of the inorganic metal chacogenides have raised interest among researchers in renewable energy research field. Another alternative way to enhance the performance is by introducing metal ions dopant to create long-lived charge carrier and reduce the electron-hole recombination (Wu et al., 2015).

Besides, the difficulty in incorporating inorganic semiconducting material into TiO_2 mesoporous matrix to obtain a well-covered monolayer on inner surface of TiO_2 electrode also influence the efficiency of photoelectrochemical cell. It is an important component which require suitable surface area for effective inorganic semiconductor material loading and eventually increase the high amount of electron-hole pair generation upon the excitation. Many studies in DSSCs using TiO_2 paste in the photoanode and some preparations are not suitable for highly efficient inorganic solar cell due to the small size of inorganic semiconductor which is less than 10 nm. In order to overcome such problem, high viscosity paste can be prepared by mixing commercial TiO_2 powder with citric acid (CA) and ethylene glycol (EG). High quality thin film with large number of pores was produce which created from the decomposition of polyester from CA and ethylene glycol EG.

1.3 Background of research

The outstanding properties of Mn doped CdS on TiO_2 nanoparticles are studied extensively as one of the potential electrodes in photoelectrochemical cell. In this study, three major parts were adopted to enhance the photoelectrochemical performance. The first part was conducted through the preparation of TiO_2 electrode that plays important roles in photoelectrochemical performance due its good charge transport properties (Park et al., 2013). The second part was by deposition of semiconductor materials on TiO_2 nanoparticles by using SILAR method that demonstrates excellent photocurrent density of 25 times higher than TiO_2 nanoparticles. The third part was carried out by modification of CdS by introducing Mn dopant that shows remarkable photocurrent density of 60 times higher than TiO_2.

Prior to the deposition of inorganic semiconductor materials, TiO_2 coating parameter was done to find the optimum condition for TiO_2 coating. Parameter such as type of TiO_2 paste, coating method and coating layer are varied throughout the study. The overview of the study presented in Figure 1.1. In this study, CdS was deposited on TiO_2 as light harvesting materials by employing SILAR method which responsible to the enhancement of the photocurrent due to its good characteristics such as tunable energy band gap, multiple exciton generation and better charge separation and transport (Hu et al., 2018). Moreover, modification of CdS was done by incorporating Mn as a dopant with CdS that produces dramatic changes in the electrical properties and photoelectrochemical performance.

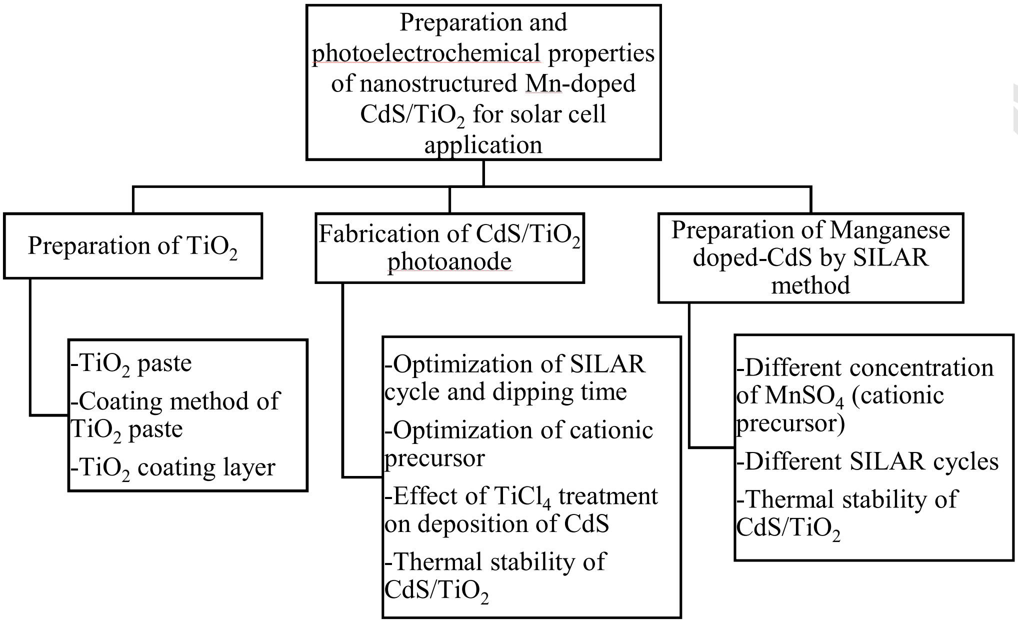

Figure 1.1: Overview of the approach of preparation of photoelectrochemical properties of nanostructured Mn doped CdS/TiO_2 for solar cell

1.4 Objectives

This research aims to explore, optimize and develop Mn-doped CdS on TiO_2 nanoparticles with excellent photoelectrochemical performance. In order to achieve this main objective, this project was divided into five specific objectives:

1) To prepare high quality TiO_2 photoanode from the viscous TiO_2 paste by doctor-blade method.
2) To deposit CdS nanoparticles and Mn-doped CdS nanoparticles on the TiO_2/FTO photoanodes via successive layer adsorption and reaction (SILAR) method.
3) To optimize study parameter of the deposition of CdS and Mn-doped CdS on TiO_2/FTO photoanodes.
4) To determine the photoelectrochemical and optical properties of CdS and Mn-doped CdS on the TiO_2 nanoparticles.
5) To determine the photoconversion efficiency of the CdS and Mn, Cr, Ni and Cu doped CdS for solar cell application.

1.5 Structure of Thesis

This thesis consists of 5 chapters. This chapter, Chapter 1, provides the introduction, problem statements and objectives. The outline structure of the remaining part of the thesis is as follow.

Chapter 2 is focused on the background of literature on the previous work of photoelectrochemical cell. A comprehensive literature on TiO_2 as a photoanode and fabrication of metal chalcogenide was discussed intensively covering the properties until method of preparation. An overview on the effect of dopant to photoelectrochemical performance also will be presented.

Chapter 3 focuses on the research methodology and particularly, the source of the materials and chemicals used will be explained in detail. The complete methodology of preparation of TiO_2 nanoparticles photoelectrode and fabrication of CdS and Mn doped CdS on TiO_2 nanoparticles will be also discussed in this chapter. In addition, characterizations of samples are described.

Chapter 4 demonstrates the experimental results for deposition of different types of TiO_2 nanoparticles, different deposition methods, and various coating layers and different calcination temperatures. The optical and photoelectrochemical properties of CdS/TiO_2 and Mn doped CdS/TiO_2 will be discussed intensively in this chapter. In addition, the crystal structure, morphology, elemental composition and photoefficiency are discussed comprehensively.

Finally, Chapter 5 presents all the key findings of this project and provides conclusion on the observations obtained beyond previously published work. Recommendations are also given for further studies regarding potential applications.

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