SYNTHESIS OF CADMIUM SULFIDE- AND TIN SULFIDE-SENSITIZED ZINC- OXIDE NANORODS AND EFFECT OF NICKEL DOPING FOR PHOTOELECTROCHEMICAL APPLICATIONS

By

TAN HUEY JING

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

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May 2024

Chairman : Professor Zulkarnain Zainal, PhD
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Zinc oxide (ZnO) is a promising semiconducting material for photoelectrochemical (PEC) applications such as water splitting due to its high electron mobility, stability, and favorable band-edge for water oxidation. However, bulk ZnO exhibits high recombination rate of photogenerated electrons and holes and limited visible light absorption owing to its wide band gap. To overcome these barriers, this study developed low-dimensional nanostructures of ZnO, in form of nanoparticles (NPs) and nanorods (NRs). Heterostructures comprised of cadmium sulfide/zinc oxide (CdS/ZnO), tin sulfide/zinc oxide (SnS/ZnO), and tin sulfide/cadmium sulfide/zinc oxide (SnS/CdS/ZnO) extend the absorption range in the solar spectrum, prolong electron lifetime, and enhance PEC performance of ZnO-based photoanodes. The study also examined the effect of nickel (Ni) doping on SnS/CdS/ZnO heterostructures. Synthesis approaches such as sol-gel spin coating, hydrothermal growth, and successive ionic layer adsorption and reaction (SILAR) were utilized to fabricate the proposed nanoheterostructures, followed by parameter optimization. Vertically aligned ZnO NRs demonstrated superior optical absorption and photocurrent generation compared to NPs due to their high aspect ratio and larger surface area. Optimized ZnO NR arrays were then sensitized with CdS forming a core-shell heterostructure. Deposition of CdS resulted in the ultraviolet-visible absorption band edge shifting to a higher wavelength, indicating enhanced visible light harvesting. The type-II interband alignment in the CdS/ZnO heterostructure facilitated electron-hole separation and transfer, boosting the incident light-to-current generation up to 5.44 mA/cm^2. Additionally, polycrystalline tin sulfide (SnS) was also successfully deposited on ZnO NRs, showing an improved photoconversion efficiency (η) from 0.56 % to 1.33 % and photocurrent density (J_{ph}) from 0.48 mA/cm^2 to 1.63 mA/cm^2 due to increased carrier lifetime and efficient charge transfer across the p-SnS/n-ZnO junction. Co-sensitization with a SnS/CdS bilayer further enhanced PEC performance of ZnO, achieving a J_{ph} of 7.50 mA/cm^2. Ni incorporation into the ternary SnS/CdS/ZnO NRs improved film uniformity, crystallinity, and charge transfer kinetics without significantly altering structural properties and absorption behavior. This improvement is attributed to reduced crystallographic defects and electronic structure modulation by Ni doping. In conclusion, the Ni-doped SnS/CdS/ZnO NRs photoanode demonstrates significant potential for enhancing visible light absorption, charge carrier generation and separation, and suppression of electron-hole recombination, thereby significantly improved PEC cell performance.

Keywords: Doping, heterostructures, photoanode, photoelectrochemical cell, zinc oxide

SDG: GOAL 7: Affordable and Clean Energy

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

SINTESIS NANOROD ZINK OKSIDA DIPEKAKAN DENGAN KADMIUM SULFIDA DAN TIMAH SULFIDA, DAN DIDOPKAN DENGAN NIKEL UNTUK APLIKASI FOTOELEKTROKIMIA

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Zink oksida (ZnO) adalah bahan semikonduktor yang berpotensi digunakan sebagai sel fotoelektrokimia (PEC) seperti dalam elektrolisis air kerana mobiliti elektronnya yang tinggi, kestabilan yang baik, dan kesesuaian kedudukan jalur-tenaga. Walau bagaimanapun, ZnO pukal menunjukkan kadar penggabungan semula pembawa cas (elektron dan lubang) yang tinggi dan penyerapan cahaya nampak yang terhad disebabkan oleh jurang jalur yang luas. Untuk mengatasi halangan ini, kajian ini memajukan ZnO berstruktur nano yang berdimensi rendah, dalam bentuk heterostruktur nanozarah dan nanorod yang terdiri daripada kadmium sulfida/zink oksida (CdS/ZnO), timah sulfida/zink oksida (SnS/ZnO) dan timah sulfida/kadmium sulfida/zink oksida (SnS/CdS/ZnO), untuk memperluas julat penyerapan spektrum suria, memanjangkan jangka hayat elektron, dan meningkatkan prestasi PEC. Kajian ini juga meneliti kesan pendopan nikel (Ni) terhadap heterostruktur SnS/CdS/ZnO. Pendekatan sintesis seperti salutan putaran *sol-gel*, pertumbuhan hidroterma, dan penjerapan serta reaksi lapisan ionik berturutan (SILAR) telah digunakan untuk menghasilkan nanoheterostruktur yang dikehendaki, diikuti dengan pengoptimuman parameter. Nanorod ZnO dengan keratan rentas heksagon yang dijajarkan secara menegak menunjukkan penyerapan optik dan penjanaan fotoarus yang lebih baik berbanding nanozarah kerana nisbah aspek yang tinggi dan luas permukaan yang lebih besar. Tatasusunan nanorod ZnO yang dioptimumkan kemudian dipeka dengan CdS membentuk heterostruktur cengkerang-teras. Pengenapan CdS menyebabkan pinggir jalur penyerapan cahaya ultraviolet-nampak beralih ke panjang gelombang yang lebih tinggi, menunjukkan peningkatan penuaian cahaya nampak. Penjajaran antara jalur jenis-II dalam heterostruktur CdS/ZnO ini memudahkan pemisahan dan pemindahan elektron-lubang, meningkatkan penjanaan cahaya kepada arus hingga 5.44 mA/cm^2. Selain itu, polihablur timah sulfida (SnS) yang juga berjaya dienapkan pada nanorod ZnO, menunjukkan peningkatan kecekapan fotopenukaran (η) daripada 0.56 % kepada 1.33 % dan ketumpatan fotoarus (J_{ph}) daripada 0.48 mA/cm^2 kepada 1.63 mA/cm^2 disebabkan peningkatan jangka hayat dan pemindahan pembawa cas yang cekap merentasi persimpangan *p*-SnS/*n*-ZnO. Pemekaan bersama menggunakan dwilapisan SnS/CdS meningkatkan lagi prestasi PEC ZnO, dengan mencapai J_{ph} sebanyak 7.50 mA/cm^2. Penggabungan Ni ke dalam SnS/CdS/ZnO NRs ternari meningkatkan keseragaman filem, kehabluran, dan kinetik pemindahan cas tanpa mengubah sifat struktur dan penyerapan secara signifikan. Peningkatan ini disebabkan oleh pengurangan kecacatan kristalografi dan pengubahsuaian struktur elektronik oleh pendopan Ni. Kesimpulannya, fotoanod Ni-dop SnS/CdS/ZnO NRs menunjukkan potensi

yang signifikan untuk meningkatkan penyerapan cahaya nampak, penjanaan dan pemisahan pembawa cas, serta penindasan penggabungan semula elektron-lubang, dengan itu meningkatkan prestasi sel fotoelektrokimia (PEC) secara ketara.

Kata Kunci: Pendopan, heterostruktur, fotoanod, sel fotoelektrokimia, zink oksida

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LIST OF ABREVIATIONS

ABPE	Applied Bias Photon-to-Current Efficiency
ALD	Atomic Layer Deposition
CB	Conduction Band
CBD	Chemical Bath Deposition
CTAB	Cetyltrimethylammonium Bromide
CVD	Chemical Vapor Deposition
DEA	Diethanolamine
DI	Deionized Water
DOS	Density of States
DSSCs	Dye-sensitized Solar Cells
EDAX	Energy Dispersive X-ray Spectroscopy
EDTA	Ethylenediaminetetraacetic Acid
EIS	Electrochemical Impedance Spectroscopy
FESEM	Field Emission Scanning Electron Microscopy
FTO	Fluorine doped Tin Oxide
FWHM	Full Width at Half Maximum
HER	Hydrogen Evolution Reaction
HMTA	Hexamethylenetetramine
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
IPCE	Incident Photon-to-Current Efficiency
ITO	Indium doped Tin Oxide
J_{ph}	Photocurrent Density
J_{sc}	Short-circuit Current Density

LED	Light Emitting Diode
LSV	Linear Sweep Voltammetry
MBE	Molecular Beam Epitaxy
MO	Metal Oxide
NBE	Near Band Edge
NHE	Normal Hydrogen Electrode
NPs	Nanoparticles
NRs	Nanorods
NSs	Nanosheets
NWs	Nanowires
OER	Oxygen Evolution Reaction
PC	Photocatalytic
PCE	Photoconversion Efficiency
PEC	Photoelectrochemical
PL	Photoluminescence
PLD	Pulsed Laser Deposition
PV	Photovoltaic
PV	Photovoltaic
QDs	Quantum Dots
QDSSCs	Quantum dot-sensitized Solar Cells
RF	Radio Frequency Magnetron Sputtering
SCE	Saturated Calomel Electrode
SCR	Space Charge Region
SILAR	Successive Ionic Layer Adsorption and Reaction
SRH	Shockley-Read-Hall Recombination

STH	Solar-to-Hydrogen Conversion Efficiency
TEM	Transmission Electron Microscopy
TFSCs	Thin-Film Solar Cells
UV-Vis	Ultraviolet-visible spectroscopy
VB	Valence Band
V_{fb}	Flat-band Potential
V_{oc}	Open Circuit Voltage
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The rapid industrial revolution and global population growth have heightened concerns about greenhouse gas emissions and climate change that predominantly addressed by oil, coal, and fossil fuel combustion to meet the escalating energy demands. However, a continuous consumption of fossil fuels not only depletes limited resources but also emitting carbon dioxide (CO_2) into the environment, posing risks to both ecosystems and human health. Considering the worldwide energy consumption forecast of nearly 50 % grow by 2050 (Energy Information Administration, 2021), it is imperative to accelerate the transition to clean energy sources such as nuclear, solar, hydro and wind power, to ensure sustainable energy supply for the future. According to International Energy Agency (IEA), the most significant progress is accounted to hydropower growth which supplied 17 % of global electricity generation in 2020, making it the largest renewable energy source after coal and natural gas. However, the environment degradation and climate change can negatively impact the water flow and reservoir supply, rendering hydropower generation unreliable (Borowski, 2022). Solar energy emerges as a promising substitute in tackling the energy crisis and environmental challenges. Solar radiation is inexhaustible, and the solar cell technologies could be potentially scaled up due to the immense solar energy (3×10^{24} J) or solar power (89300 TW) reaching Earth's surface annually. With an energy conversion device to collect and transform a 8-hours incoming sunlight radiation into technically accessible energy, it is potentially fulfil the 2 weeks' worldwide usage (Jin et al., 2021; Zhang et al., 2019).

Extensive research has so far established different series of photovoltaic (PV) systems to efficiently harvest and store the renewable solar energy into electricity. These PV technologies include the traditional silicon-based solar cells, thin-film solar cells (TFSCs), dye-sensitized solar cells (DSSCs), quantum dots-sensitized solar cells (QDSSCs), organic and polymeric solar cells, perovskite solar cells, and the emerging fourth-generation hybrid inorganic cells as shown in Figure 1.1. A key issue on the operation of photovoltaic cells is highly dependent on the amount of sunlight trapped and an appropriate storage system is needed to ensure the all-time electricity supply. Such an off-grid approach for example, the application of lithium-ion battery or supercapacitor to store the converted electricity, is still considered expensive, space-consuming and short-lived (Dias & Mendes, 2018). In this context, solar driven water splitting process that mimics the nature of photosynthesis is potentially to ease the solar energy storage by generating chemical fuels. For instance, hydrogen (H_2) is an abundant and flexible energy carrier, exhibiting 3- to 4-fold higher mass energy density compared to gasoline (Jiang et al., 2017). Owing to its transportability, environmental-friendliness (free of hydrocarbon, carbon monoxide, and carbon dioxide emission), and better efficiency compared to traditional combustion-based engines, hydrogen can be directly applied as transportation fuel or utilized in food industry, chemicals manufacturing, and power generation (Dias & Mendes, 2018; Stępień, 2021).

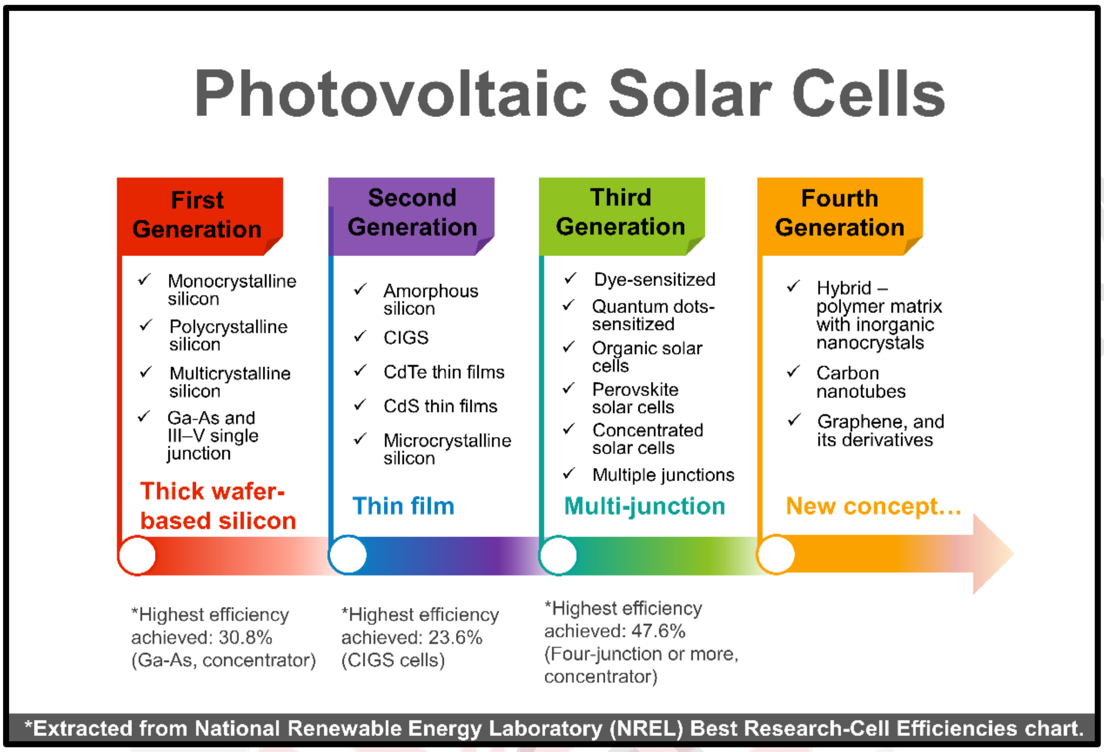

Figure 1.1: Classification of photovoltaic solar cells
(Source: Pastuszak & Węgierek, 2022)

From pass investigations, hydrogen generation can be readily occurred *via* diverse water splitting procedures which are electrolysis of water, photoelectrochemical (PEC) and photocatalytic routes, radiolysis, photobiological, and thermochemical production (Ahmed & Dincer, 2019; Joy et al., 2018; Saraswat et al., 2018; Tee et al., 2017). Radiolysis and biolysis produce unwanted nuclear waste and carbon dioxide, respectively (Joy et al., 2018; Tee et al., 2017). Electrolysis of water is costly as it requires copious amounts of electricity supply to initiate dissociation of water (Stępień, 2021) while thermochemical water splitting utilizes an extremely high thermal energy (>2500 K) to split the water molecules into hydrogen and oxygen (O_2) (Tee et al., 2017). Therefore, photoelectrochemical (PEC) and photocatalytic (PC) water splitting are more promising to be employed for hydrogen production. These two strategies are analogous where photoactive semiconductor is primarily required to allow light harvesting and ensure the facile separation and transport of photogenerated carriers (Ning & Lu, 2020; Si et al., 2017; Wu et al., 2020). According to Wu and co-workers (2020), a conduction band with more negative energy than water reduction potential and a valence band with more positive energy than water oxidation potential are the decisive necessity for realizing the overall water splitting. However, some semiconducting materials (such as CdSe, MoS_2, WO_3, and Fe_2O_3) are incapable of producing H_2 or O_2 through photocatalytic water splitting process due to the inappropriate conduction band (CB) or valence band (VB) position(s) (Rozhkova & Ariga, 2015). In addition, most semiconductors could easily suffer from carrier recombination that resulted in low photocatalytic performance. This circumstance can be overcome when an external bias is applied to the photoelectrochemical (PEC) electrodes to compensate the potential deficiency and assist the charge separation and diffusion mechanism (Rozhkova & Ariga, 2015). In comparison to photocatalytic based setup, PEC system is also advantageous with direct product separation by generating O_2 and H_2 gases at different electrodes (Jin et al., 2021).

1.2 Brief history and development of PEC cell using classic semiconductor materials

The photoelectrochemical (PEC) water splitting is straightforward and sophisticated in concept, with the solar-to-hydrogen (STH) conversion efficiency highly dependent on every single process, including light absorption;

charge generation, separation, and transfer; and electrode/electrolyte interfacial reaction (Chen et al., 2021). Developing a robust and stable photoanode is therefore essential, as the water oxidation reaction is always the bottleneck for efficient solar-to-hydrogen power generation (Amano, 2021). Since the pioneer discovery by Fujishima and Honda in 1972, where a rutile TiO_2 single crystal photoanode in contact with an electrolyte achieved photoelectrochemical water splitting (Ahmed & Dincer, 2019), PEC cells with photoanode fabricated from single metallic or bimetallic materials have garnered significant interest.

Classic metal-oxide semiconductor materials such as TiO_2, WO_3, Fe_2O_3 and $BiVO_4$ are among the most common semiconducting materials utilized in PEC cell technology due to their non-toxicity, good stability, earth-abundance, and favorable band structures. Figure 1.2 displays the theoretical and reported photocurrent densities for these metal oxides under one-sun illumination. Of particular concern is the poor absorption, slow reaction kinetics, and sluggish photogenerated carriers transport that possibly limited their PEC performance (Kalanur et al., 2020; Najaf et al., 2021; Tayebi et al., 2019). Despite having higher electron mobility and lower recombination rate compared to TiO_2 (Sharma et al., 2018), ZnO, with its intrinsic wide band gap of 3.37 eV could absorb only the ultraviolet (UV) light which is merely 5 % of the solar spectrum (Cao et al., 2019; Wibowo et al., 2020). It has thus remained challenging to rely on pristine ZnO-based photoanode for high-performance PEC water splitting even though it possesses suitable band edges position meeting the thermodynamic requirement for water splitting (Yang et al., 2017). To date, the highest reported photocurrent density for a ZnO single-crystalline photoanode is 1.84 mA/cm^2 (Zhang et al., 2016) while the highest STH efficiency achieved using a multijunction monolithic PEC cell incorporating III-V semiconductors is 19.3 % (Cheng et al., 2018). Indeed, an efficiency target of 25.0 % is required for a PEC water splitting system to meet the renewable energy market competitiveness (Vilanova et al., 2024). Continuous investigation into PEC cell photoelectrodes is crucial for achieving this efficiency target and advancing the field of solar-to-hydrogen energy conversion.

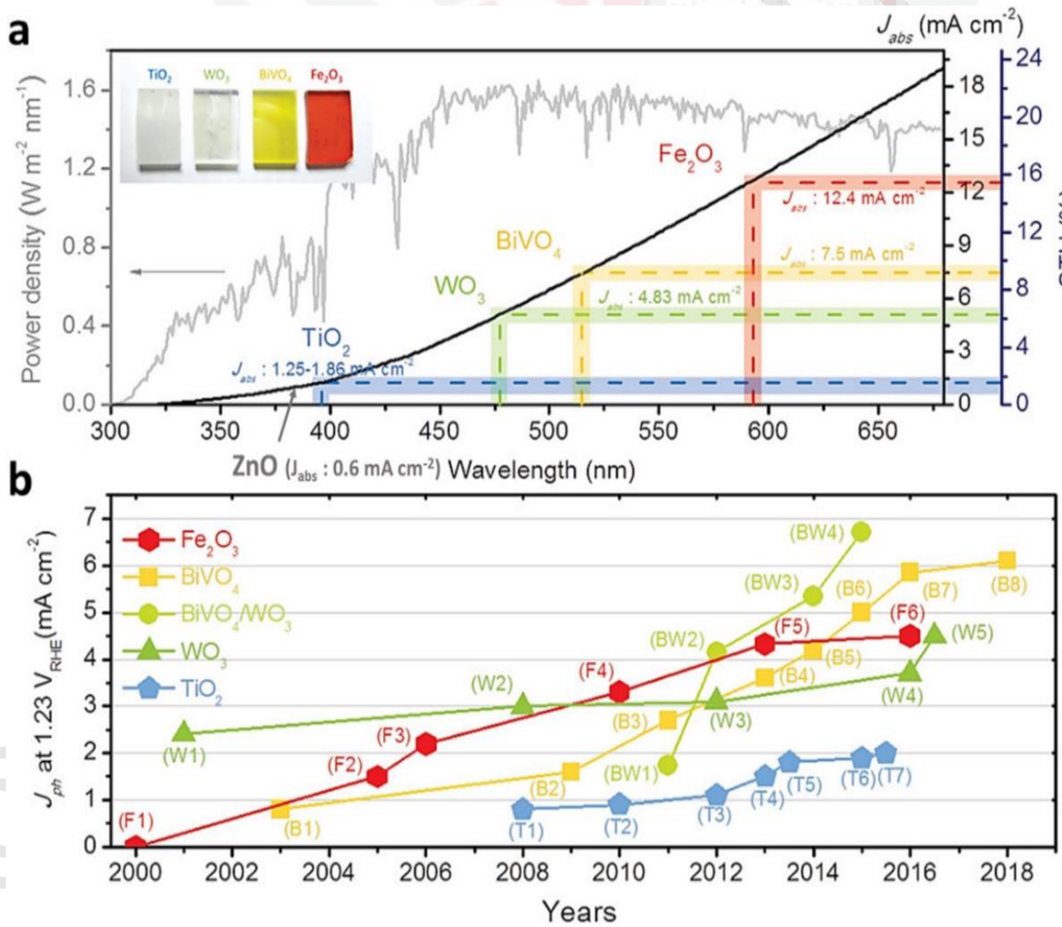

Figure 1.2: (a) Theoretical solar-to-hydrogen efficiency and (b) Reported photocurrent density of TiO_2, WO_3, Fe_2O_3, and $BiVO_4$ photoanodes under 1 Sun irradiation from PEC water oxidation
(Source: Kim & Lee, 2019; Liu et al., 2014)

1.3 Problem Statement

Zinc oxide (ZnO) is a low-cost material with a wide range of applications, including solar cells, transistors, sensors, photocatalysis and photoelectrochemical systems. Its versatility stems from its well-defined conduction and valence bands, making ZnO a particularly promising material for photoanode fabrication. This is crucial in facilitating the transfer of holes to water, thereby enhancing the separation of electron-hole pairs and improving the efficiency of PEC water splitting. Nevertheless, the band gap energy of ZnO which is larger than 3.1 eV inhibited incident visible light absorption. This represents an important drawback in ZnO-based photoelectrodes since the entire solar spectrum consists of 42 % of visible light (400 nm $< \lambda <$ 800 nm) (Amano, 2021). Besides that, the undesired high photoinduced carrier recombination rate in ZnO bulk semiconductor and at ZnO/electrolyte interface (Ma et al., 2020) also emerged as an obstacle in achieving the theoretical PEC efficiency.

Over the years, numerous attempts have been made to address the deficient PEC performance of ZnO. Nanoscale control could produces different low dimensional nanostructures of ZnO including nanoparticles, nanorods, nanowires and nanotubes to simultaneously enhance the light capturing and carrier transport abilities compared to their bulk counterpart (Chen et al., 2021). Other than that, introduction of plasmonic materials (e.g., Au, Ag and Pt) and element (metal or non-metal) doping into ZnO are also attractive means to broaden the absorption spectral range and tune the optical and electronic properties of fabricated photoelectrodes (Ma et al., 2020; Ros et al., 2020).

Considering few successful strategies capable of obtaining high enough photocurrent generation with only a single component photoanode, surface modification on ZnO nanomaterials *via* sensitization and layered structure formation has open a whole field of opportunities (Amano, 2021). For example, dye sensitization on ZnO is promising to significantly expand the light absorption into visible and even the infrared regions of solar spectrum depending on the type of adsorbed dye (Tyona et al., 2018) while at the same time, a vectorial charge separation reaction can be observed in dye-sensitized photoelectrochemical cells as the photoexcited electrons in dye molecules are injected into ZnO at the semiconductor/dye interface (Ngidi et al., 2019) and the oxidized dye is later regenerated by the coupled water oxidation catalysts (WOCs) (Zhang et al., 2019). In parallel to the typical build-up of dye sensitized ZnO photoelectrodes, creation of a heterogeneous junction constructed with narrow-band gap chalcogenide photosensitizer is considered a more direct method to induce an alternative absorption channel that effectively utilize the full solar spectral energy as well as better charge carrier separation mechanism (Consonni et al., 2014) by injecting the electrons into lower lying conduction band of ZnO to prolong the electrons lifetime.

Among the reported inorganic chalcogenide materials, PbS (Mehrabian et al., 2020), CdS (Nan et al., 2017), ZnS (Wang et al., 2018), Bi_2S_3 (Al-Zahrani et al., 2019), Ag_2S (Holi et al., 2019) and CdSe (Li et al., 2016) are the most common sensitizers employed in bilayer or multilayer ZnO-based photoanodes fabrication (Jin et al., 2021; Yoo et al., 2019). In spite of the high toxicity of cadmium (Cd) element, cadmium sulfide (CdS) still remained as an ideal candidate to be coupled with ZnO due to its visible light absorption ability, high carrier mobility, facile synthesis and more importantly, favorable energetics compatible to ZnO in constructing an efficient type-II heterostructure that able to hamper the quick recombination rate of photogenerated charge carriers in pure CdS semiconductor (Biswas et al., 2019), and regulate the photo-corrosion and poor photostability of CdS in aqueous solution upon irradiation (Jiang et al., 2017; Peng et al., 2023). Recently, tin chalcogenides such as tin (II) sulfide (SnS) have also attracted much attention from heterojunction solar cell research community ascribed to their non-toxicity, high absorption coefficient and fascinating electrical properties (Huang et al., 2017). To date, most studies in nanostructured SnS synthesis have only focused on their applications as photocatalyst (Mittal et al., 2020), SnS-based photovoltaic device (Cho et al., 2020; Voznyi et al., 2020) or SnS-based photocathode for PEC system (Cheng et al., 2018; Lee et al., 2020). Furthermore, the best performing SnS-based solar cell devices achieved only 4.36 % photoconversion efficiency using expensive atomic layer deposition (ALD) technique (Sinsermsuksakul et al., 2014). Literature reviews have drawn the possibility of binary SnS/TiO_2 (Shiga et al., 2016) and SnS/CdS (Fu et al., 2018) electrodes construction but no studies have examined the effectiveness of ternary SnS/CdS/ZnO NRs/ITO photoanode for PEC application. Moreover, rare empirical research has adequately explained on how the transition metal (e.g., Mn, Fe, Ni, Cu) doping modifies and correlates the electrical, optical, physical, and photoelectrochemical characteristics of SnS thin films. In this work, ZnO nanorods (NRs) arrays, CdS nanoparticles (NPs) and SnS nanoparticles (NPs) are synthesized and assembled into binary or ternary heterostructures through inexpensive spin coating, hydrothermal, and successive ionic layer adsorption and reaction (SILAR) methods. To the best of our knowledge, Ni-SnS/CdS co-sensitized ZnO NRs photoanode is first time introduced, and the comparison with undoped sample for use in photoelectrochemical (PEC) cells is presented.

1.4 Objectives of Study

This research involves fundamental investigation on nanoarchitecture, design of semiconductor heterostructures, and adoption of impurity doping strategies for high-performance thin film photoelectrochemical electrodes fabrication. The project's focus is to study the ability of ZnO nanorods-based photoanode in environmental-friendly energy conversion, which is promoted by visible light active materials sensitization and electron rich transition metal doping. Below are the clearly defined specific objectives to be addressed in this research:

1. To synthesize and optimize
    i. crystalline and uniform ZnO nanoparticles (NPs) seed layer by sol-gel spin coating approach
    ii. high aspect-ratio one-dimensional ZnO nanorods (NRs) by hydrothermal method
    iii. CdS/ZnO NRs, SnS/ZnO NRs, and ternary SnS/CdS/ZnO NRs *via* successive ionic layer adsorption and reaction (SILAR) process

iv. electrical properties of SnS/ZnO NRs and SnS/CdS/ZnO NRs photoanodes by introducing nickel (Ni) dopant at different concentrations

2. To determine the structural, optical, and morphological properties of nanostructured ZnO, CdS/ZnO, SnS/ZnO, Ni-SnS/ZnO, SnS/CdS/ZnO and Ni-SnS/CdS/ZnO heterostructures using XRD, UV-Vis and FESEM techniques

3. To evaluate and compare the photoelectrochemical performance and photoconversion efficiency of PEC cells with different fabricated photoanodes: ZnO NRs/ITO, CdS/ZnO NRs/ITO, SnS/ZnO NRs/ITO, Ni-SnS/ZnO NRs/ITO, SnS/CdS/ZnO NRs/ITO, and Ni-SnS/CdS/ZnO NRs/ITO

1.5 Scope of Study

This work covers the development of low-cost nano-heterostructures photoelectrodes for photoelectrochemical application. In particular, the effects of CdS and SnS incorporation on morphological, structural, optical, and electrical properties of ZnO NRs arrays were investigated extensively. The experimental conditions during spin coating, hydrothermal, and SILAR processes were controlled under series of optimizations as the particles' nucleation rate and growth kinetics in solution-based approaches always have a direct impact on the size and properties of synthesized nanomaterials (Nikam et al., 2018), which in turn affecting the photoelectrochemical cells performance. Detailed discussion was conducted based on the characterization analysis results to understand the role of CdS NPs, SnS NPs and Ni dopant in tuning the light harvesting and photocurrent generation efficiencies of ZnO NRs. Figure 1.3 illustrates the workflow and overview of the project. The thesis is arranged into eight chapters. Chapter 1 provides the general introduction on solar power and its trending applications in photovoltaic solar cell technology, and renewable hydrogen fuel production which can be done by implementing at least an effective semiconductor for photoelectrochemical cell invention. It also serves as the foundation for the discussion on weakness of ZnO semiconductor, the importance, and objectives of this work. Chapter 2 highlights the working principles of photoelectrochemical cell and the evolving research practices in modifying the performance of ZnO-based PEC cells. Literature review on the employed techniques for related materials and heterostructures synthesis is also reported to give readers new insights into ongoing heterojunction PEC cells evolution. Chapter 3 presents all sample preparation procedures with the adjusting variables, and the fundamentals of applied characterization methods. Chapters 4 - 8 outline the characterization data analysis and results discussion for ZnO NPs seed; ZnO NRs growth; CdS-sensitized ZnO NRs; SnS-sensitized ZnO NRs and Ni-doped SnS-sensitized ZnO NRs; SnS/CdS co-sensitized ZnO NRs and Ni-doped SnS/CdS co-sensitized ZnO NRs, respectively. Chapter 8 also compares and summarizes the optical, morphological and photoelectrochemical properties changes of the above photoelectrodes. Chapter 9 concludes the research findings with some recommendations of future studies related to the proposed photoelectrodes.

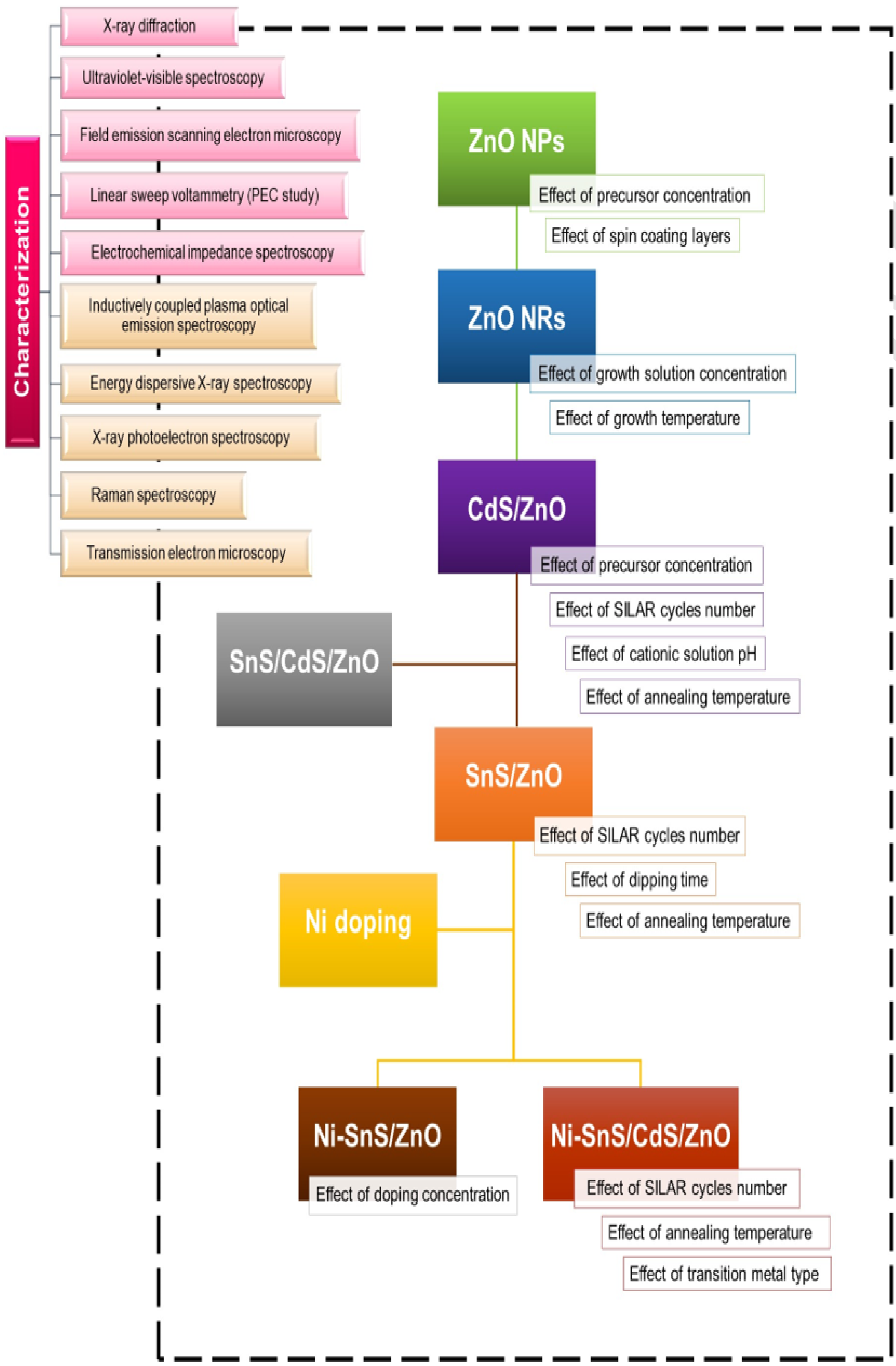

Figure 1.3: Research flow and overview of study

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