SILVER-MONTMORILLONITE MODIFIED TITANIUM DIOXIDE ASSISTED CARBON NITRIDE NANOCOMPOSITES FOR PHOTOCATALYTIC HYDROGEN PRODUCTION THROUGH WATER SPLITTING

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DEDICATION

This thesis is dedicated to my mother, Zariah binti Kosnoh who taught me that even the largest task can be accomplished if it is done one step at a time. It is also dedicated to my father, Mohamad Lazif bin Lasimin who taught me that the best kind of knowledge to have is that which is learned for its own sake.

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ABSTRACT

Photocatalytic hydrogen (H_2) generation is one of the most promising solutions to convert solar power into clean energy to replace non-renewable fossil fuel. The objective of this study is to investigate montmorillonite (MMT) dispersed and silver (Ag)-bridged protonated carbon nitride/titanium dioxide (pCN/TiO_2) Z-scheme heterojunction composite for stimulating photocatalytic H_2 evolution under UV and visible light in different photocatalytic reactor systems. The newly designed MMT-Ag/pCN-TiO_2 composite photocatalysts were fabricated through a sol-gel assisted hydrothermal method and were characterized by X-ray diffraction, Raman spectroscopy, X-ray photoelectron spectroscopy, field emission scanning electron microscopy, energy-dispersive X-ray mapping, transmission electron microscopy, Brunauer–Emmett–Teller, ultraviolet–visible (UV-vis) spectroscopy and photoluminescence spectroscopy. The photocatalytic activity was tested using slurry, fixed bed and monolith photo-reactor systems for continuous H_2 production. Using slurry system, MMT-Ag/pCN-TiO_2 photo-catalyst produced 667 µmol h^{-1} of H_2 which is 8.41 and 9.66 times higher than pCN/TiO_2 and TiO_2 samples, respectively. The efficiency was improved due to formation of heterojunction with faster charges separation, whereas, Ag provides hot photo-generated electrons by surface plasmon resonance and MMT traps electrons for H_2 production. Optimization reveals that the highest production of H_2 was obtained at pH 7, glycerol concentration of 5 wt. % and 0.15 g of catalyst loading using slurry reactor. Furthermore, by applying an engineering approach MMT-Ag/pCN-TiO_2 showed H_2 production rate was increased to 8230 µmol h^{-1} using a monolith reactor, which are 9.01 and 12.34 times higher than fixed-bed and slurry photo-reactors. The monolith honeycomb reactor exhibited a higher apparent quantum yield and space yield of 39.85 % and 54.86 µmol $h^{-1}cm^{-3}$ compared to slurry (22.36 %, 5.13 µmol $h^{-1}cm^{-3}$) and fixed-bed reactors (4.42 %, 6.09 µmol $h^{-1}cm^{-3}$). The superior performance of a monolith reactor was due to higher photon flux utilization, large illuminated surface area and processing volume. The schematic of type II heterojunction and Z-scheme mechanism of MMT-Ag/pCN-TiO_2 were developed and the photocatalytic performance was compared in all types of systems. In conclusion, excellent performance of composite catalyst using a monolith reactor compared to a slurry and fixed-bed reactor for H_2 production would offer a new opportunity in engineering approach for renewable fuels applications.

ABSTRAK

Penjanaan fotopemangkinan hidrogen (H_2) adalah salah satu jaminan penyelesaian untuk menukarkan tenaga suria kepada tenaga bersih bagi menggantikan bahan api fosil yang tidak boleh diperbaharui. Tujuan penyelidikan ini adalah untuk mengkaji serakan montmorillonite (MMT) dan perak (Ag)-menjambatani karbon nitrida diprotonkan/titanium dioksida (pCN/TiO_2) Z-skema komposit heterosimpang untuk merangsang perkembangan fotopemangkinan H_2 di bawah sinaran UV dan nampak dalam sistem reaktor fotopemangkinan yang berbeza. Foto-mangkin komposit MMT-Ag/pCN-TiO_2 yang baharu disediakan melalui kaedah sol-gel berbantukan hidrotermal dan dicirikan oleh pembelauan sinar-X, spektroskopi Raman, spektroskopi fotoelektron sinar-X, mikroskop elektron imbasan pancaran medan, pemetaan serakan tenaga sinar-X, mikroskop elektron transmisi, Brunauer–Emmett–Teller, spektroskopi ultralembayung-nampak (UV-vis) dan spektroskopi fotoluminesen. Aktiviti fotopemangkinan diuji menggunakan sistem reaktor buburan, lapisan-tetap dan monolit bagi penghasilan H_2 yang berterusan. Menggunakan sistem buburan, foto-mangkin MMT-Ag/pCN-TiO_2 menghasilkan 667 μmol h^{-1} H_2 di mana 8.41 dan 9.66 kali lebih tinggi masing-masing daripada sampel pCN/TiO_2 dan TiO_2. Kecekapannya bertambah baik disebabkan oleh pembentukan heterosimpang dengan pemisahan caj yang lebih pantas, sedangkan, Ag memberikan foto-janaan elektron panas oleh permukaan plasmon resonans dan MMT memerangkap elektron untuk menghasilkan H_2. Pengoptimuman mendedahkan bahawa H_2 tertinggi diperolehi pada pH 7, kepekatan gliserol pada 5 wt. % dan 0.15 g muatan mangkin menggunakan reaktor buburan. Tambahan pula, dengan menggunakan pendekatan kejuruteraan MMT-Ag/pCN-TiO_2 menunjukkan kadar penghasilan H_2 telah meningkat ke 8230 μmol h^{-1} menggunakan reaktor monolit, 9.01 dan 12.34 kali lebih tinggi daripada fotoreaktor lapisan-tetap dan buburan. Reaktor sarang lebah monolit menunjukkan lebih tinggi hasil kuantum ketara dan hasil ruang pada 39.85% dan 54.86 μmol $h^{-1}cm^{-3}$ berbanding dengan reaktor buburan (22.36%, 5.13 μmol h^{-1} cm^{-3}) dan lapisan-tetap (4.42%, 6.09 μmol h^{-1} cm^{-3}). Prestasi unggul reaktor monolit disebabkan oleh penggunaan fluks foton yang lebih tinggi, keluasan besar permukaan yang diterangi dan isipadu pemprosesan. Skema heterosimpang jenis II dan mekanisma Z-skema MMT-Ag/pCN-TiO_2 telah dihasilkan dan prestasi fotopemangkinan telah dibandingan untuk semua jenis sistem. Kesimpulannya, prestasi cemerlang mangkin komposit menggunakan reaktor monolit berbanding reaktor buburan dan reaktor lapisan-tetap bagi penghasilan H_2 akan menawarkan peluang baharu bagi pendekatan kejuruteraan untuk aplikasi bahan api yang boleh diperbaharui.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ALD	-	Atomic Layer Deposition
AQE	-	Apparent Quantum Efficiency
AQY	-	Apparent Quantum Yields
BET	-	Braunauer-Emmer Teller
BJH	-	Barrett-Joyner-Halenda
CB	-	Conductance Band
CPD	-	Contact Potential Difference
CPSI	-	Channels Per Square Inch
DET	-	Direct Electron Transfer
DRS	-	Diffuse Reflectance Spectra
EDX	-	Energy-dispersive X-ray
FESEM	-	Field Emission Scanning Electron Microscopy
GHG	-	Greenhouse gas
LSPR	-	Localized Surface Plasmon Resonance
MFC	-	Mass Flow Controller
MMT	-	Montmorillonite
PL	-	Photoluminescence
PS I	-	Photosystem I
PS II	-	Photosystem II
SEM	-	Scanning Electron Microscopy
SPR	-	Surface Plasmon Resonance
TEM	-	Transmission Electron Microscopy
UV	-	Ultra-Violet
VB	-	Valance Band
XPS	-	X-ray Photo-electron Spectroscopy
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

e^-	-	Electron
h^+	-	Hole
E_g	-	Energy Band Gap
λ	-	X-ray Wavelength
ΔH	-	Change in enthalpy of Reaction
ϕ	-	Work Function Value of Metal
h	-	Planck's Constant
c	-	Speed of Light.
N_A	-	Avogadro Constant
E_c	-	CB Minimum Energy Level Position
E_v	-	VB Maximum Energy Level Position
k_B	-	Boltzmann Constant
T	-	Absolutely Temperature
N_c	-	Effective Densities of State in CB
N_v	-	Effective Densities of State in VB
n	-	Electrons Concentration
p	-	Holes Concentration

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Natural resources such as coal and petroleum products as a source of energy are nearly exhausted [1]. The reduction of fossil fuel reserves has prompted substantial research efforts toward the usage of hydrogen (H_2) as an environmentally friendly energy carrier for the post fossil fuel regime [2]. It is generally agreed that H_2 may be the best option for tackling the triple issues of exhaustion, pollution and climate change effects [3]. One of the technologies for H_2 production is photo-catalytic water splitting, since it entails photonic energy, which is the most abundant energy resource on the Earth [4]. Previous research states that solar based H_2 generation by photo-catalysis provides near zero global warming and air pollutants [5] and can be stored easily [6]. Therefore, H_2 is considered as a possible important energy in future, since it is free from toxic and it can produce high energy content from natural resources such as light (photon) energy and water, which are clean, long lasting sources of energy, and renewable resources [7].

The pioneer work of photo-catalytic for H_2 production from water splitting using TiO_2 semiconductor photo-catalyst was conducted by Fujishima [8]. Since then, the photo-catalyst development has become great attention from researchers to improve the photo-catalytic performance. Based on the previous research outcomes, the efficiency of H_2 production via photo-catalytic water splitting is relatively low as the activity and stability of semiconductors is not much appreciable. However, H_2 production via photo-technology can be improved using modified semiconductors and introducing sacrificial reagents such as alcohols as electron donor [9].

Among all, titanium dioxide (TiO_2) with band gap 3.2 eV is a recognized photo-catalyst and it has been extensively studied because of numerous advantages such as low cost, high photochemical stability and non-toxic. On the other hand, wide band gap limits its applications under visible light and faster charges recombination rate lowers its photo-catalytic activity [10]. Coupling TiO_2 with visible light semiconductors can narrowing the band gap with faster charges separation, thus could enables enhanced photo-catalytic activity.

Among the low band gap semiconductors, polymeric graphitic carbon nitride (g-C_3N_4) has attracted more attentions as metal-free polymeric semiconductor in photo-catalytic water splitting. It is a visible light responsive with lower band gap and low cost semiconductor. It can be synthesized from cheap precursors such as melamine and urea by simple thermal approach. In addition, g-C_3N_4 has numerous advantages such as high thermal and chemical stability and appropriate band structure (2.7 eV) to absorb visible light irradiation [11]. Among the limitations, g-C_3N_4 has low surface area and small active sites for interfacial (photon) reaction, moderate oxidation reaction of water to H^+ and low charge mobility which disrupt the delocalization of electrons. Hence, the coupling or/and doping g-C_3N_4 with other elements can overcome its limitations. Among the other alternatives, coupling g-C_3N_4 with TiO_2 to develop type II heterojunction could be promising to get enhanced H_2 production during photo-catalytic water splitting under visible light irradiations. Tan et al. [12] fabricated g-C_3N_4/TiO_2 composite and 10.8 times higher efficiency for H_2 production than bulk g-C_3N_4. Similarly, Li et al. [13] reported significantly improved H_2 production over g-C_3N_4/TiO_2 composite compared to TiO_2 under visible light. In another work, g-C_3N_4/TiO_2 photo-catalysis reported by Yan and Yang [14] found very efficient for good activity for H_2 generation due to faster separation of electron-holes pairs.

In addition, protonation of g-C_3N_4 (pCN) has been considered as an effective method to affect surface charge properties which could be utilized to construct assembled catalysts to improve the efficiency of g-C_3N_4. The protonation effects the electrical surface by lowering the valance band (VB) that is good for water oxidation under visible light and helpful in charges separation [15]. Thus, combination of pCN with TiO_2 to form heterojunction can alter the electronic structure and improves the efficiency of photo-catalyst with higher reduction ability.

Furthermore, loading metals on pCN/TiO_2 can further enhance the charges separation to retard the recombination. There have been several reports on doping semiconductor with noble metals such as silver (Ag), gold (Au) and platinum (Pt) [16-18]. Among them, Ag is more attractive because of low cost and can improve the efficiency under visible light due to surface plasmon resonance (SPR) [19]. Patra and Gopinath [20] reported that Ag doping TiO_2 increased electrons density and SPR effect to enhance H_2 production. Similarly, improved H_2 production due to SPR effect of Ag/TiO_2 was reported by Rather et al. [21]. Therefore, it can be suggested that Ag loading to TiO_2 can further improve the H_2 production due to SPR effect. Besides, Ag metal can act as mediator for Z-scheme system. Previously, Gong et al. [22] revealed Z-scheme of Ag_2CrO_4/Ag/g-C_3N_4 composites with Ag as mediator, thus exhibits superior photo-catalytic activity toward 2,4-DCP degradation under visible light irradiation. Similarly, Huang et al. [23] reported excellent activity and stability of indirect Z-scheme of Ag/AgBr@$CoFe_2O_4$ photo-catalyst with Ag as a solid-state electron mediator for degradation of phenol. Although, Ag has been successfully reported as mediator in different applications, yet it has not been reported using Ag-pCN/TiO_2 composite for H_2 production. Furthermore, performance of Ag-pCN/TiO_2 can also be improved by loading with low-cost, green and natural materials such as clay minerals.

Clay minerals are heterogeneous and environment friendly materials with lamellar structure with advantages such as non-corrosive, low-cost, abundant, resistance to chemicals and high thermal stability [24]. Among the clay mineral, montmorillonite (MMT) has been widely used as co-doping with semiconductors due to high cations exchange capacity and excellent electron trapping ability, which can be adjusted by treating with acid to exchange the cations in the interlayer space [25,26]. Since, MMT has 2D interface, it is predicting to have large specific surface area with dense active surface groups to trap electrons efficiently. Moreover, it is abundance and cheap to make MMT a promising matrix support for semiconductors in order to prevent the aggregation and form a new innovation to improve the performance in catalytic composites [27]. Previously, MMT-loaded Fe/TiO_2 has been reported for CO_2 reduction to fuels [28]. Koci et al., [35] reported CO_2 reduction over ZnS/MMT with enhanced photo-activity. Thus, MMT dispersed g-C_3N_4/TiO_2 heterojunction composite would be helpful to avoid recombination of charges by trapping electron from $g\text{-}C_3N_4/TiO_2$ within MMT structure. Although, MMT has been successfully reported in different applications, yet it has not been reported using MMT-Ag/pCN-TiO_2 composite for H_2 production.

Recently, the formation of Z-scheme photo-catalytic system, analogous to artificial photosynthesis, is one of the latest strategies to improve photo-catalytic performance as compared to using single semiconductor photo-catalyst. Commonly investigated Z-scheme systems have three classifications that are with shuttle redox mediators, without electron mediators, and with solid-state electron mediators [29]. These systems can enhance the efficiency of photo-catalyst performance, since it effectively increases the visible light absorption, accelerates the separation and transportation of charge carriers.

In addition, surface modification such as catalyst structure and morphology can improve performance due to increasing surface area and efficient charge carrier separation [30]. The configuration of semiconductors has been designed and investigated in the form of nanoparticles, nanosheets, nanotubes and nanowires [31]. Therefore, semiconductor photo-catalyst selection and modification has great potential to narrow the band gap, utilizing visible light and promoting charge separation towards selective H_2 evolution. Furthermore, photo-catalytic efficiency can be achieved in the presence of sacrificial reagents such as reforming alcohols, which play roles as electron donor and hole scavenger, since the oxidation potential of alcohol is lower than reduction of H^+ to H_2. From the previous research, glycerol is more helping for the generation of H_2 than using methanol and ethanol. Bahruji et al. [32] investigated 20 different sacrificial reagents and proved increasing H_2 production rate in order triols > diols > primary (1°) alcohols > secondary (2°) alcohols > tertiary (3°) alcohols. The production of H_2 also depends on the location of physical properties of alcohols like number of α-H or OH atoms and alcohols polarity [33].

The design and selection of photo-reactor is another engineering approach which contributes significantly in evolution of H_2 during photo-catalysis. Since, the effectiveness of photo-catalytic activity depends on the absorption of photons and reactants on the catalyst surface, the innovations on photo-reactors should include selection of light sources and distribution, shape and dimension of reactor, and design of irradiation device such as reflectors. Typically, slurry reactor is used in photo-catalysis process, yet it has limitations such as low light distribution, lower light penetration depth, excessive cost for catalyst separation and cannot maximize H_2 production. Therefore, photo-technology has been developed and recently, monolith reactor has attracted the attention of researchers among the photo-reactors such as slurry, fluidized, fixed-bed and optical fiber photo-reactor. Monolith, which contains parallel straight channels, have been exploited very efficiently due to higher illuminated surface area, high utilization of photon flux energy and tends to generate more H_2 under flow operation [34]. Previously, the monolithic support was used to enhance the photo-activity and reusability of Fe-MMT/TiO_2 heterojunction in a CO_2 reduction [28]. According to previous research, the photo-catalytic water splitting for H_2 production over MMT-Ag/pCN-TiO_2 composite based on engineering approach has never been reported.

Herein, synergistic effect between MMT-Ag/pCN-TiO_2 composite and photo-reactor systems for enhanced photocatalytic H_2 production has been investigated. The MMT-Ag/pCN-TiO_2 was synthesized by hydrothermal assisted sol-gel method and MMT-Ag/pCN-TiO_2 monolithic support was synthesized by dip-coating method. The results obtained by different photo-catalysts and parameters such as catalyst loading, sacrificial reagent, pH and stability under visible light irradiation in slurry reactor were analyzed and compared. Then, engineering approach using different type of photo-reactor system was investigated over MMT-Ag/pCN-TiO_2 under visible and UV light for H_2 production. In addition, the performance of reactor system was analyzed and compared according to the apparent quantum yields (AQY) based on the ratio of H_2 production rate with photon intensity strike on the catalyst surface. The space yield was calculated to reflect the reaction effect on the volumetric yield of reactor. Based on the experimental data, the schematic reaction mechanisms of water splitting for H_2 production over MMT-Ag/pCN-TiO_2 composite was proposed.

1.2 Problem Statement and Hypothesis of Research

Though, water splitting for H_2 production is getting increased attention in recent years, still there are certain limitations faced and the main challenges are low yield of the products and lesser selectivity. To this end, the problems and solution approaches are:

(a) Photocatalytic system required higher irradiation of light energy for H_2 production. Mostly researchers have conducted photocatalytic H_2 production under TiO_2 based photo-catalysts. However, TiO_2 is UV-active only and has higher recombination of photo-generated charge rate, resulted low yield rate. Therefore, lower TiO_2 activity can be overcome by coupling with low cost pCN. The use of pCN and Ag which is active to visible light irradiation will increase the yield rate of H_2 production under solar energy. The fabrication of MMT-Ag/pCN-TiO_2 composite can improve the efficient production rate. The plasmonic Ag metal can provide more electron from SPR effect, 2D layered MMT structure with impressive skill to capture charges and great potential for cation transfer can acts as electron trap while, pCN/TiO_2 can prevent the fast recombination through Z-scheme system. Thus, it will help to improve H_2 production efficiency through water splitting process under visible light irradiations.

(b) The performance of photo-catalytic H_2 production in liquid system has lower yield and selectivity of H_2. However, it can be improved by addition of sacrificial reagent in feed reactant to act as electron donor for reaction process to produce more H_2. Photo-catalytic water splitting has been conducted using slurry reactors and it has lower light harvesting efficiency, lower illuminated surface area and less quantum efficiency for H_2 production. The lower production rate of H_2 can be overcome using micro-channel monolith photo-reactor. The monolith can provide higher light distribution and adsorption over the catalyst surface area since the photo-catalyst is coated as thin film over the monolithic structured channels. The monolith has quantum efficiency, larger illuminated reactor volume and higher sorption process to stimulate enhanced H_2 production. Monolith photo-reactor in gas phase system can be employed to maximize the yield of H_2 as it provides large surface area contact with high illumination of light between gas reactant and catalyst

(c) The optimization of operating parameters would be helpful to improve H_2 production rate. Therefore, it is expecting that with optimizing parameters such as catalyst loading, pH and reaction time would provide higher H_2 production. Meanwhile, the comparison of different reaction systems, reaction pathways and analysis of apparent quantum yield and space yield would be helpful to get deep insight of the effectiveness towards H_2 production.

1.3 Objectives of Study

The aim of this research is to investigate the performance of new develop MMT-Ag/pCN-TiO_2 composite heterojunction in slurry and monolith photo-reactor for H_2 production. In order to achieve this objective, the following sub-objectives are identified:

(a) To develop MMT-Ag/pCN-TiO_2 modified nanocatalysts for photo-catalytic H_2 production;

(b) To investigate performance of newly developed catalysts and study effect of operating parameters for photocatalytic H_2 production under visible light irradiation;

(c) To evaluate performance of photo-reactors systems for H_2 production using slurry, fixed-bed and monolith photo-reactors;

(d) To propose reaction mechanism and determine quantum analysis in different reactions systems for maximum H_2 production.

1.4 Scope of Study

This study was focused on developing new MMT-Ag/pCN-TiO_2 catalysts for photo-catalytic H_2 production and following are the scope of this research:

(a) The pristine TiO_2 was prepared from Titanium (IV) isopropoxide and hydrolysed by acetic acid by sol-gel method while, the pCN was prepared from melamine under pyrolysis process and protonated by HNO_3. MMT-Ag/pCN-TiO_2 composite was synthesized using sol-gel method and MMT-Ag/pCN-TiO_2 supported monolithic was coated by sol-gel dip-coating method. The TiO_2 and pCN based catalysts were characterized using X-ray Diffraction (XRD), Raman, XPS, Field Emission Scanning Electron Microscopy (FESEM), EDX Mapping, Transmission Electron Microscopy (TEM), Brunauer-Emmerr-Teller (BET) Surface Area, UV-Visible Spectrophotometer and PL spectra.

(b) The Ag and pCN loading on TiO_2 were investigated for optimizing of deposited for H_2 yield while the TiO_2-based, pCN-based and newly developed catalyst were tested the performance of H_2 production. The operating parameters such as catalyst loading, type of sacrificial reagents and operating pH were investigated using slurry photo-reactor under visible light irradiation. The catalyst loading was investigated using 0.05, 0.1, 0.15 and 0.2 g catalysts dispersed in the alcohol-water mixture. The effect of different sacrificial reagents was tested in different type of feed alcohol-water mixture included water and 5% of methanol, ethanol, ethyl glycol and glycerol. The effect of pH solution on catalyst performance was experimented in neutral medium of DI water, acidic and basic medium. The DI water was added with HCl and NaOH for adjustment to acidic and basic condition, respectively. The stability of the catalyst was tested for three cycles with 4 h/cycle and the reactor was purged with N_2 gas without light for 1 h to remove H_2 and other gasses at the end of each run.

(c) The effect of sacrificial reagent in gas phase was tested using gas feed of water and 5% of alcohol-water mixture. The alcohols include methanol, ethanol, ethyl glycol and glycerol. The effectiveness of photo-reactors system was investigated using slurry, fixed-bed and monolith reactor to observe the production of H_2. In fixed-bed catalyst was distributed inside the reactor. Meanwhile, the monolith made from ceramic with channels per square inch (CPSI) = 100 was cut into cylinder with length 1 cm and diameter 6 cm. The monoliths were coated with catalyst using sol-gel dip coating method. Then, the coated monoliths were placed in the reactor chamber with light source at the top of reactor. In addition, the photo-reactor system was analysed and compared using apparent quantum yield and space yield.

(d) After proper analysis and study of results obtained, reaction mechanism of type II heterojuction which electrons transfer between semiconductor from CB of higher negative to CB of lower negative while Z-scheme system transfer electron from CB of lower negative to VB of lower positive semiconductor through solid mediator were proposed. In different system, the quantum analysis was determined using apparent quantum yield (AQY) and space yield. The AQY was evaluated based on the ratio of H_2 production rate with photon intensity strike on the catalyst surface. The space yield was calculated to reflect the reaction effect on the volumetric yield of reactor.

1.5 Significant of Study

This study has immersed contribution to researchers in photo-catalysis, the scientific community and the public for the following reasons. Firstly, the research on MMT-Ag/pCN-TiO_2 composite and the monolith reactor provides more insight as it workable under lower light intensity as well as direction on the mechanism of composite during water splitting. In addition, the effect of parameter in water splitting can be better understood from this research. A photo-catalyst which is stable, high charge separation and environmental is introduced.

1.6 Thesis Outline

This thesis comprised of five chapters. Chapter 1 is the introduction; it consists of research background of photo-catalytic water splitting for H_2 production, problem statement and hypothesis, objectives, scopes, and the significant of the research. Chapter 2 is the literature review which consists of fundamentals and thermodynamics of photo-catalysis. The advancements in photo-catalysts for water splitting are reviewed from previous studies. The heterojunction construction, factors that influence photo-catalyst activity and advancements in photo-reactor are explained. In Chapter 3, the catalysts synthesis, and characterization procedure are explained. The reactor setup, parameter study and apparent quantum yield and space yield calculation are also discussed. The results and discussion are presented in Chapter 4 while, conclusions and recommendations are stated in Chapter 5.

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