# Contents

Abstract i  
List of abbreviations ii  

## Chapter 1  Introduction  

1.1 Fundamental principles of Semiconducting photocatalysis 2  
1.2 Overview of Photocatalytic materials 3  
1.3 Challenge and Opportunities 4  
1.4 Nanostructured photocatalysts 6  
1.5 Utilization of Solar energy 8  
1.6 Future Fuel (energy) - Hydrogen 10  
  1.6.1 Principle of Water splitting (Photoelectrochemical and Photocatalytic methods) 13  
    1.6.1.1 Brief survey of photocatalytic hydrogen generation via water splitting 15  
  1.6.2 Principle of Hydrogen Sulfide (H$_2$S) splitting 16  
    1.6.2.1 Why H$_2$S splitting for solar light driven hydrogen production? 17  
    1.6.2.2 Brief survey of photocatalytic hydrogen generation via H$_2$S splitting 20  
1.7 Selective significant nanomaterials with its properties 20  
  1.7.1 Zinc Indium Sulphide (ZnIn$_2$S$_4$) 20  
  1.7.2 Nitrogen doped TiO$_2$ (N-doped TiO$_2$) 22  
  1.7.3 Fe$_2$O$_3$ 24  
1.8 Motivation of the present work 25  

References 29
Chapter 2  Fundamental and Experimentation

2.1  Introduction 35

2.2  Synthesis methods 35
   2.2.1 Hydrothermal/solvothermal method 35
   2.2.2 Thermolysis method 38

2.3  Design and fabrication of experimental setup for H₂S splitting 39

2.4  Characterization techniques 41
   2.4.1 X-ray diffraction (XRD) 41
   2.4.2 Scanning electron microscope (SEM) 41
   2.4.3 Transmission Electron Microscope (TEM) 42
   2.4.4 X-ray photoelectron spectroscopy (XPS) 44
   2.4.5 Ultra Violet-Visible Defuse Reflectance Spectroscopy (UV-vis DRS) 45
   2.4.6 Photoluminescence (PL) 45
   2.4.7 Fourier Transform Infrared Spectroscopy (FTIR) 46
   2.4.8 Raman Spectroscopy 48
   2.4.9 Mössbauer Spectroscopy 50
   2.4.10 BET Surface Area Measurement 52
   2.4.11 Thermogravimetric Analysis (TGA/DTA) 53
   2.4.12 Vibrating Sample Magnetometer (VSM) 54
   2.4.13 Gas Chromatography (GC) 55
   2.4.14 Gas Chromatography-Mass Spectroscopy (GC-MS) 55

References 57
Chapter 3  Hierarchical nanostructured ZnIn$_2$S$_4$ for an efficient hydrogen production

3.1  Introduction  58
3.2  Experimental  59
   3.2.1 Optimization of controlled synthesis of ZnIn$_2$S$_4$ nanostructures  59
   3.2.2 Phase formation  62
3.3  Results and discussion  65
   3.3.1 Morphological Study  65
   3.3.2 TEM study  71
   3.3.3 Growth mechanism  73
   3.3.4 Optical study  76
   3.3.5 X-Ray photoelectron Spectroscopy (XPS) analysis  79
   3.3.6 Photocatalytic Activity  80
3.4  Conclusions  83

References  85

Chapter 4 Visible light driven nano-architectured N doped TiO$_2$
for hydrogen production

4.1  Introduction  87
4.2  Experimental  88
   Optimization for controlled synthesis (architecture) of N doped TiO$_2$  88
4.3  Results and discussion  92
   4.3.1 XRD study  92
   4.3.2 FESEM and TEM studies  93
   4.3.3 Optical Study  96
   4.3.4 XPS analysis  97
Chapter 5 Synthesis of maghemite (hematite) core (shell)-Fe$_2$O$_3$ nanorods for photocatalytic hydrogen generation

5.1 Introduction

5.2 Experimental details
5.2.1 Preparation of ligand
5.2.2 Preparation of Iron complexes
5.2.2 Preparation of Iron Oxide Nanorods

5.3 Results and discussion
5.3.1 Structural study
5.3.2 Optical Study
5.3.3 FTIR-Study of 2'HC ligand and its Fe-complex
5.3.4 Mössbauer spectroscopy study
5.3.5 Raman spectroscopy study
5.3.6 Morphological study
5.3.7 TEM study
5.3.8 Thermogravimetric analysis
5.3.9 Magnetic Properties
5.3.10 Possible mechanism
5.3.11 Photocatalytic activity