

HIGH PHOTODEGRADATION ACTIVITY OF NIO AND CAESIUM DOPED β - NICKEL HYDROXIDE NANOPARTICLES

Submitted in partial fulfillment of the
requirements for the award of
Bachelor of Science
by

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DEPARTMENT OF CHEMISTRY

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade "A" by NAAC

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119

March - 2021



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DEPARTMENT OF CHEMISTRY

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **Mr. K.N. KAVERI LOGESH (38030007)**, **Ms. R. PRIYA DHARSHINI (38030012)** who carried out the project entitled "**HIGH PHOTODEGRADATION ACTIVITY OF NiO AND Cs doped β -Ni (OH)₂**" under my / our supervision from **01/11/2020** to **31/04/2021**.

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S. SUNITHA, M.Sc., M.Phil., Ph.D.

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Submitted for Viva voce Examination held on _____

Internal examiner

External Examiner

DECLARATION

I am **Mr. K.N. KAVERI LOGESH & Ms. R. PRIYADHARSHINI** student of Sathyabama Institute Of Science And Technology hereby declare that the Project Report entitled “**HIGH PHOTODEGRADATION ACTIVITY OF NiO & Cs DOPED β -Ni(OH)₂ NANOPARTICLES**” done by me under the guidance of **Dr. S. SUNITHA, M.Sc., M.Phil., Ph.D.** and **Dr. G. MURUGADOSS**, Scientist-E, International Research Center (IRC), Sathyabama Institute Of Science and Technology, Chennai- 600-119 is submitted in partial fulfilment of the requirements for the award of Bachelor of Science degree in Chemistry.

DATE: 1.

PLACE: 2.

SIGNATURE OF THE CANDIDATES

ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to Board of Management of **SATHYABAMA** for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to **Dr. J KARTHIKEYAN, Head of the Department, Dept. of Chemistry** for providing me necessary support and details at the right time during the progressive reviews.

I would like to express my sincere and deep sense of gratitude to my Project Guide's **Dr. S SUNITHA M.Sc., M.Phil., Ph.D. (INTERNAL GUIDE)** and **Dr. G. MURUGADOSS, SCIENTIST-E, (IRC) (External guide)** for their valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

I would like to express my sincere thanks once again to **Dr. S. SUNITHA M.Sc., M.Phil., Ph.D.**, for getting me the opportunity to do my project work in **(IRC)**.

I wish to thank **my colleagues** for their constant help and guidance throughout the project work.

I wish to express my thanks to **all Teaching and Non-teaching staff** members of the Department of Chemistry who were helpful in many ways for the completion of the project.

K.N. KAVERI LOGESH

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ABSTRACT

The advancement in nanotechnology research has helped in the development of eco-friendly methods for the synthesis of nanoparticles, which gives considerable importance to expand their applications. Nanostructured NiO and Cs doped Ni(OH)₂ have been synthesized via Co-precipitation method. The Co-precipitation method have resulted in maximum yield. A systematic change in preparation parameters like calcination temperature, pH value and calcination time has done in order to study the influence on the effect of crystallite size. XRD was used to examine the crystalline size. Fourier Transform Infrared Spectroscopy was used to examine the functional groups involved in the molecules of synthesised nanoparticles. Scanning Electron Microscope was used to investigate the particle size and morphology of the synthesised nanoparticles. The prepared samples are characterized by X-ray diffractometer, scanning electron microscopy, Fourier transform infrared spectroscopy. The optical band gap decreases from 3.21 eV [pure NiO] to 2.82 eV [Ni(OH)₂ doped with 5% Cs]. Further, the results are encouraging as the synthesized nanoparticles stand a better chance of being used as an active material for textile dyes because of their high photo degradation activity in photocatalytic applications. The synthesized NiO and Cs doped Ni(OH)₂ nanoparticles were used as efficient photocatalyst for the photocatalytic degradation of Methylene blue (MB) dye. the obtained photo degradation of MB dye was above 86.39 % in 120 min under visible light irradiation.

Keywords: Cs doped Ni(OH)₂, NiO, Methylene blue dye, Co-precipitation technique, X-Ray diffractometer, Scanning electron microscopy, Fourier transform infrared spectroscopy.

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LIST OF ABBREVIATIONS EXPANSIONS

NPs	-	Nanoparticles
NiO	-	Nickel oxide
Ni(OH) ₂	-	Nickel hydroxide
CB	-	Conduction band
VB	-	Valence band
Cs/Ni(OH) ₂	-	Cesium doped nickel hydroxide
XRD	-	X-ray diffraction
SEM	-	Scanning electron microscopy
FTIR	-	Fourier transform infrared microscopy
MB	-	Methylene blue
ROS	-	Reactive organic species

CHAPTER – 1

Chapter-1, starts with a brief introduction of the metal oxide nanostructures and their various synthetic methods. In addition to this, a short review on the targeted applications, i.e., photocatalytic applications, of this project was also discussed in this chapter. Finally, the chapter describes the objective and importance of the project.

CHAPTER-1

1. INTRODUCTION

1.1 *Nanomaterials*

Nanotechnology is a versatile field of science like chemistry, physics, material science, involve for the synthesis of nanoparticles and nanomaterials and their applications. This area of science is to understand their physical and chemical properties of nanoparticles. These properties make nanoparticles as an emerging technology and because of the novel application of nanomaterials, the nanomaterials had evolved as a frontline research area [1,2]. The fusion of nanoparticles in modern technology has a unique properties of nanoscale material possess When compared to bulk material, nanoparticles of the same material can show enhanced Catalytic, electronic, magnetic properties [3] of metal nanoparticles are sensitive to their size and shape. The material properties that make nanoparticles ideal for these applications such as band gap or emission spectrum, can be tuned by changing particle size and morphology [4]. Based on the dimension, the nanoparticles are classified 3 types as shown in Figure 1.1 Due to different synthesis of nanoparticles with different method gives a different class of nanomaterials, and it is classified into three categories based on their dimensionality: zero dimensional (0D), one dimensional (1D), two dimensional (2D) and three dimensional (3D).

In zero dimensional nanomaterials are measured within the nanoscale its dimension is not more than 100 nm. It is called as nanoparticles

In one dimensional nanomaterial is outside the nanoscale this class includenanotubes, nanorods, nanowires [5-7].

In two dimensional structures are studied extensively for the utilization of nanodevice application [8-10]. And this class include graphene, nanolayers, nanocoating

In three dimensional nanomaterials are materials that are not consider to the nanoscale in any dimension this class can contain bulk powder, bundles of

nanowires and nanotubes. 0D,1D,2D are close contact with each other and form interfaces.

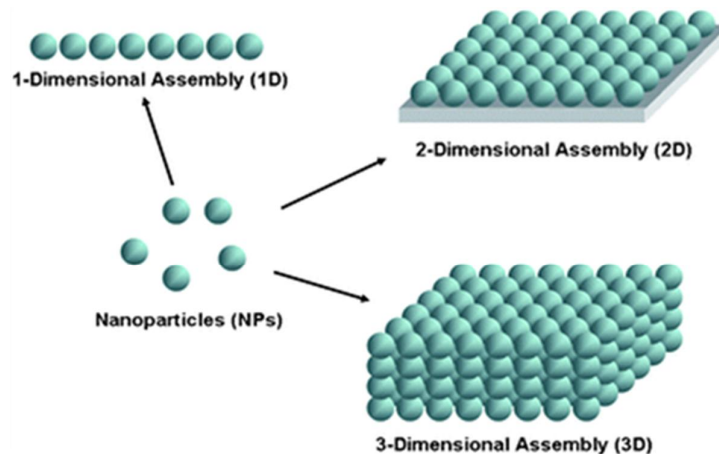


Figure 1.1 : The Classification of Dimensional Nanoparticles

The synthesis had various approaches of method like Top-down approaches and Bottom-down approaches in this regard we did a Bottom-down approaches in that of co precipitation method synthesis of nano particle synthesis and it is the most conventional method, involving the reduction of metal salts using some chemical reduction of metal salts. A stabilizing agent also added to reduce a particle agglomeration like polyvinyl pyrrolidone [PVP], polyvinyl alcohol [PVA], polyethylene glycol [PEG], the goal of the work is to synthesis of cesium doped NiO nanoparticles by the chemical co-precipitation method in the presence sodium hydroxide, it is very simple way and low in cost.

Semiconductor has a significant role in processing in the field of nanoscience and nanotechnology Usually in conductors current is considered to flow due to electrons only but here in semiconductor materials it is caused by both electron as well as hole movements. Semiconductor materials do not belong only to crystal solids but are of the 3 nature of amorphous and liquids too. A pure semiconductor is called as intrinsic semiconductor.

The electronic properties and the conductivity of semiconductor can be changed in a controlled by adding very small quantities of other materials called dopants. The field is expected to open new ideas for science and technology. Preparation and applications of the metal oxide nanostructures are belonging to Semiconducting nanomaterials and nanotechnology. Metal oxide proved to be very promising for a variety of practical applications. Metal oxides play a very important role in many areas of chemistry, physics, and materials science [11-14].

1.2 METAL OXIDES

Metal oxides are widely suitable for photocatalyst applications because of their suitable band gap and it considered as an heterogenous catalyst and some group of metals have much attraction towards their outer electronic configuration. they are applied widely in various reactions, like oxidation processes, upon light irradiation that are responsible for degradation of dyes. preparation of these metal oxides through the synthesis can be described as physical and chemical methods. In general, the synthesis of metal oxide nanoparticles is done by top-down and bottom-up approaches techniques. These approaches involved in all the three phases like solid-liquid-gas [1,2,15-20]. Due to their unique properties these metal oxides such as optical, thermal, catalytic, photochemical etc. made themselves as a high-level technological application. Hence the metal oxide nanostructure is broadly studied by researchers.

1.3 SYNTHESIS TECHNIQUES OF METAL OXIDE NANOPARTICLES

synthesis of nanoparticles with a definite structure and size is the most challenging task in the field of nanoscience and nanotechnology. So there has a variety approaches to synthesis of nanostructures such as thermal evaporation, chemical vapor deposition, hydrothermal synthesis etc. these kinds of metal oxide nanostructure synthesis provide a great interest for the researchers because of their intense properties and variety of applications. In background,

this synthesis can be broadly classified by two types such as spontaneous, and template-based category [21]. In spontaneous synthesis, the crystal structure and their surface properties grow along with a particular direction [21]. In case of template-based category synthesis, different template used to maintain the growth of nanostructures [21]. Prepare nanostructure in different approaches like top-down approaches and bottom-up approaches.

A) TOP-DOWN APPROACHES:

Cutting down a bulk material into nanoparticle. And a starting material of this approaches in only suitable for a solid state, and this process is a physical processing method. it has an advantage of chemical purification material is not required so we get a maximum quantity of nano particle but the yield we get [10-1000nm].

B) BOTTOM-DOWN APPROACHES:

Build a material from molecule to atom(cluster), and then atom to nanoparticle. The starting material for this method is suitable for gaseous and liquid state, this is made up of both physical and chemical methods. And the method is very cheaper than Top-down approaches. it has an advantage of getting an ultra-fine nanoparticle but the quantity of product is we get a smaller amount of nanoparticles.

To develop the techniques and its growth of nanostructure we did a report for different approaches in this literature, such as sol-gel technique, chemical precipitation method and so on.

1.3.1 SOL-GEL Technique

Sol-gel precipitation approach has evolved for the preparation of nanoparticles with inorganic materials [22-24]. The synthesis of sol-gel method is a more familiar approach is shown in Figure 1.2 this method is based on hydrolysis of a solution of a precursor to obtain a colloidal particle is called as sol and it is condensed to form a gel then a gel of aggregated sol particles is heated

to get a desired yield. A variety of nanoparticles are synthesized such as TiO_2 , CuO , etc. this method is used for the fabrication of metal oxides.

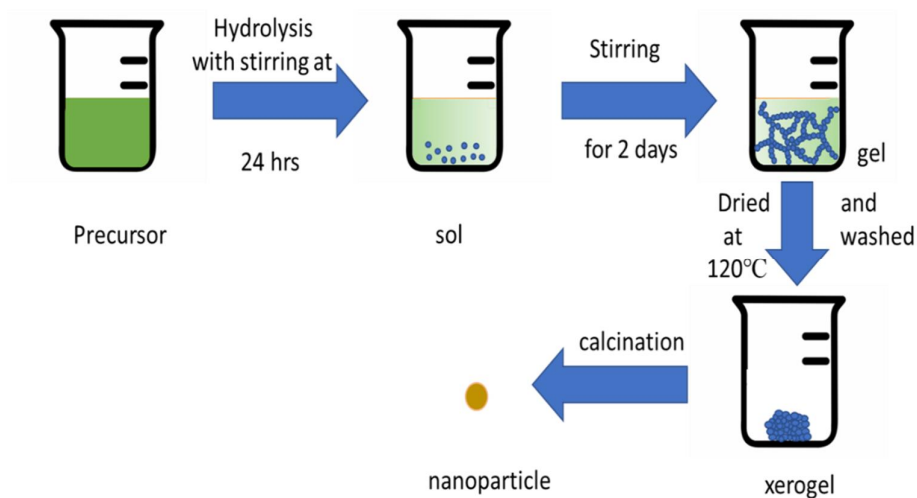


Figure 1.2 : Synthesis Method of Sol-gel Technique

1.3.2 Co-Precipitation Method

Co-precipitation method is a standard synthesis technique to prepare a pure metal oxide nanoparticle. Fig 3 shows the process of co-precipitation method. In co-precipitation, typically a salt precursor is been a nitrate, chloride and dissolved in aqueous solvents, then a base compound added to form a precipitation, add hydroxides like sodium hydroxide, ammonium hydroxide etc, [25]. and to reduce agglomeration add a stabilizing agent like PVP, PVA etc, the size, morphology of the nanoparticles is controlled [26]. The precipitate we get was washed several times with water and acetone and dried at 160°C .

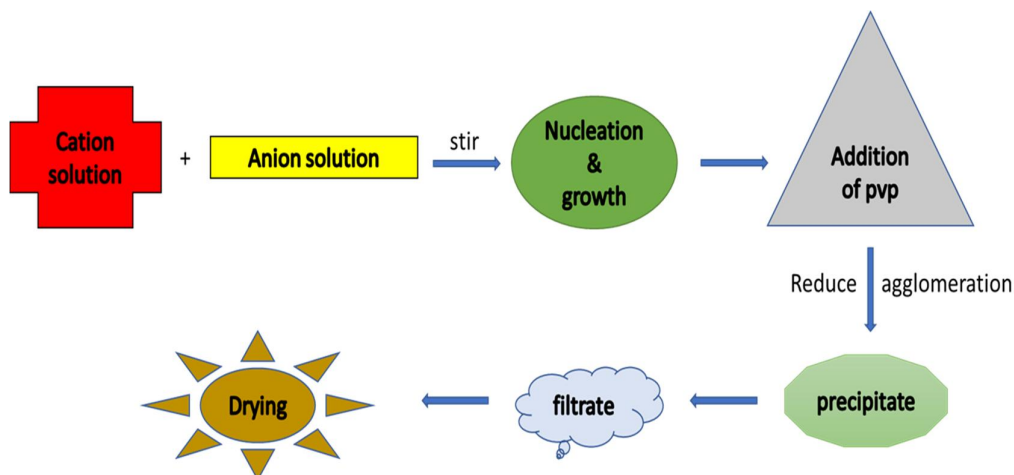


Figure 1.3: Process of Co-precipitation Method

1.3.3 Hydrothermal Synthesis

Hydrothermal method is one of the common methods for the synthesis of chemical reactions at a sealed autoclave reactor having aqueous solvent or mineraliser under high pressure (0.3 - 4 MPa) (MPa= megapascal) and high temperature (130-250°C) [27]. The process involve in autoclave reactor is shown in Fig 1.4 It is a solution-based reaction approach [28]. Advantages of the hydrothermal synthesis method include the ability to synthesize crystals of substances which are unstable near the melting point, and the ability to synthesize large crystals of high quality. Disadvantages are the high-cost equipment and the inability to monitor the crystal growth [29]. And it is well known technique for synthesis of nanostructures. The synthesized products were investigated in terms of their morphological, structural, and optical properties. Finally, the prepared nanomaterials were used for sensing, photovoltaic and photocatalytic properties.

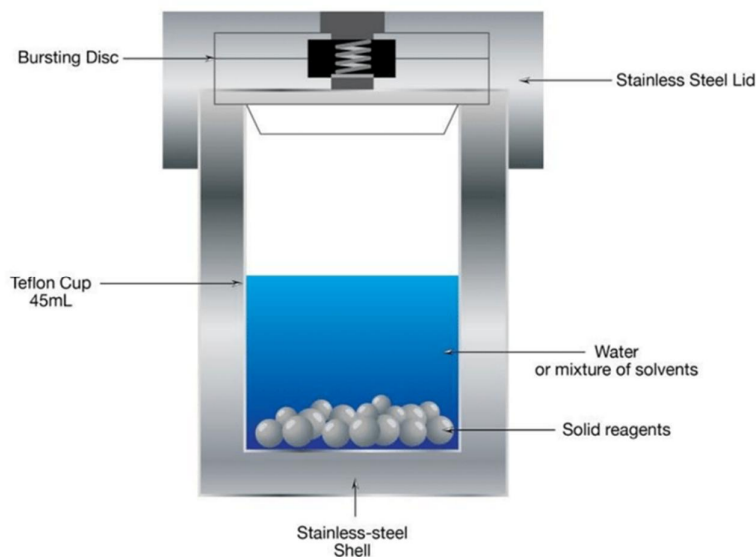


Figure 1.4 : Process Involve in Hydrothermal Autoclave Reactor

1.4 IMPORTANCE OF METAL OXIDE

Metal oxide have been attracted because of their changes in optical, electrical, catalytic and magnetic properties include with physical and chemical stability. metal Oxide nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface sites [30]. Metal oxide nanoparticles have a variety of applications such as catalyst etc. Metal oxide semiconductors are suitable photocatalysts due to their high photosensitivity, stability, low cost and suitable band gap [31]. The structure of metal oxides is greatly disturbed, because of the particle size decreases, the increasing number of surface atoms that generate stress/strain. Thermal elements are capable of forming a wide range of oxide compounds. These can adopt a vast number of structural geometries with an electronic structure that can exhibit insulator, semiconductor, or metallic characteristics. Fig 1.5 shows some commonly used types and applications of metal oxide nanoparticles [32].

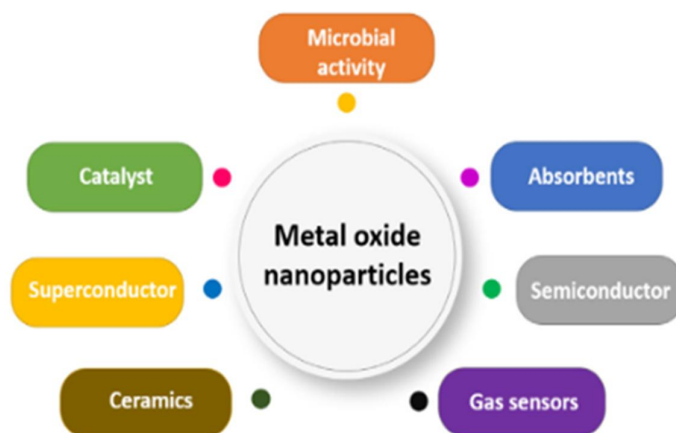


Figure 1.5 : Types and Applications of Metal Oxide Nanoparticles [32]

1.5 TARGETED METAL OXIDE:

1.5.1 NiO Nanoparticles

NiO is an important transition metal oxide with a cubic lattice structure. Fig 1.6 shows the crystal structure of NiO. The average band gap of NiO is 3.6-4 electron volt [33]. The average particle size of NiO is $\approx 30\text{nm}$ using polyvinylpyrrolidone is assisted [34]. Nickel oxide (NiO) nanoparticles have been effectively used as photocatalysts [35]. NiO is a stable p-type semiconductor metal oxide having tendency to absorb UV radiations due to its wide band gap. Nickel oxide already has a much attention due to its excellent electrical, optical and catalytic properties and its excellent durability, high photo sensitivity and high adsorptive affinity. Among various metal oxide, NiO is also used to eliminate an organic dyes and contaminants in wastewater. In a research lead by Martha Ramesh et al. [36], on the degradation of sensitive black 5 (RB-5) and methylene blue (MB) by NiO NPs, they found that the annealing of nanoparticles at a temperature is more than $400\text{ }^{\circ}\text{C}$ will increase the degradation rate. And Sabouri et al. [37], achieved about 80% of methylene blue (MB) dye removal with a concentration of 7 mg/L at the pH value of 11. The NiO nanoparticles made at a higher annealing temperature ($400\text{ }^{\circ}\text{C}$) had excellent adsorption and photocatalytic activity against textile dyes. NiO has been synthesized by several methods such as hydrothermal method, co-precipitation method, sol-gel method, thermal decomposition method etc.

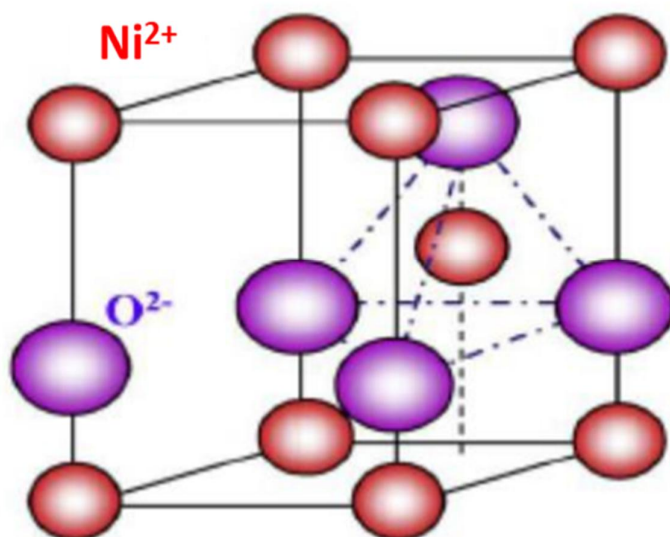


Figure 1.6 : Crystal Structure of NiO

1.5.2 Properties of NiO:

TABLE 1 : REPRESENTS THE PROPERTIES OF OXIDE NANOPARTICLES INFLUENCED THE SIZE, SURFACE EFFECTS, CRYSTAL LATTICE DEFECTS

Chemical Properties :

Chemical Data	
Chemical symbol	NiO
CAS No.	1313-99-1
Group	Nickel 4
	Oxygen 16
Electronic configuration	Nickel [Ar]3d ⁸ 4s ²
	Oxygen [He] 2s ² 2p ⁴

Chemical Composition	
Element	Content (%)
Nickel	78.55
Oxygen	21.40

Physical Properties :

The physical properties of nickel oxide nanoparticles are given in the following - table.

Properties	Metric	Imperial
Density	6.67 g/cm ³	0.240 lb/in ³
Molar mass	74.71 g/mol	-

Thermal Properties :

The thermal properties of nickel oxide nanoparticles are provided in the table below.

Properties	Metric	Imperial
Melting point	1955°C	3551°F

1.5.3 Key Applications of NiO:

- Nickel oxide particles are widely used in batteries, like lithium-ion batteries, Nickel-zinc battery etc.
- Nickel oxide is a significant material for applications in preparation of nickel cermet for the anode layer of solid oxide fuel cells (Thomas et al., 2013).
- It is also used as a catalyst and antiferromagnetic layers, and radar absorbing materials.
- In light weight structural compounds in aerospace.
- In active optical fibres, in cathode materials for alkaline batteries and materials for gas or temperature sensors such as CO sensor, H sensor etc (AzoNano,2013).

1.5.4 Ni (OH)₂ Nanoparticles

While Nickel oxide nanoparticles has certain advantages and applications, Nickel hydroxide, Ni (OH)₂ is a poor electrical conductivity p-type semiconductor has many important technological uses. Nickel hydroxide is used for battery, photocatalysis, and fuel cell applications [38,39]. Depending on the pH and other species in the solution, either nickel oxide or nickel hydroxide particles can be formed. Nickel hydroxide usually forms at 150°C –200°C. recently, nickel hydroxide has found increasing attention because of its applications in alkaline rechargeable batteries and high energy batteries owing to its high-power density and specific energy, great charge–discharge cyclic characteristics, and low toxicity [40]. Nickel hydroxide is known as an active material for cathodes in nickel-based rechargeable alkaline batteries [41-48].

*Until now a number of metal oxides nanostructures have been synthesized such as Zinc oxide (ZnO), Copper oxide (CuO), Nickel oxide (NiO) [49-51] etc. In this project work, we have planned to study in particular NiO and at low temperature formation of Ni (OH)₂ with Cs ion. Here in this project, we will

investigate the synthesis of the above-mentioned metal oxide nanostructures through co-precipitation method. The metal oxides we study here in this work is Cs doped NiO. The prepared samples size, morphology, structure was characterized under X-Ray Diffraction [XRD], Scanning Electron Microscope [SEM], UV-visible spectroscopy.

1.6 TARGETED APPLICATION OF Ni(OH)_2 AND NIO NANOPARTICLES PRESENTED IN THIS PROJECT

1.6.1 Photocatalyst Application

Catalyst is defined as speeding up the chemical reaction. Similarly, photocatalyst is nothing but speeding up the chemical reaction in the presence of UV-light. The photocatalytic process starts with an irradiation of semiconductor material by absorb a light that produces an electron hole (e^-/h^+) pair. photo-generated pairs of h^+ and e^- induces the formation of aggressive species such as hydroxyl ($\text{OH}^\cdot$) Significant features of the photocatalyst have: proper band gap, morphology of the material, more exposed surface area, stability and its reusability. This photocatalyst activity broadly divide into two types: homogeneous photocatalysis and heterogeneous photocatalysis. The photocatalyst is widely practiced in degradation process thus leads a reduction toxic metal ion into non-toxic state. The photocatalytic activity of semiconducting metal oxide can be effectively modified by ionizing radiations. the semi conducting metal oxides on irradiation with appropriate wave length leads to excitation of an electron from valence band to conducting band yielding an electron hole pair [52]. Based on photocatalyst Fig 1.7 shows catalyst nanoparticles. Upon light irradiation, these photogenerated charge carriers create radical species ($\cdot\text{OH}^\cdot$, $\cdot\text{O}_2^\cdot$) that are responsible for degradation of dyes. Photodegradation of dyes is useful for water treatment process to degrade a pollutant [53]. Commonly used dyes in photocatalytic studies are methylene blue (MB) Suitable morphology, high surface area, narrow band gap, stability, and low recombination rate of charge carriers are some of key features that a good photocatalyst should possess [54]. in this project, we did a report of the synthesis and characterization of NiO NPs, as a photocatalyst to degrade

methylene blue dye (MB) in the presence of visible light. Fig 1.8 shows the photocatalytic degradation of (MB). The structural, morphological, optical, adsorption, and photocatalytic properties of NiO and β -Ni (OH)₂ NPs with respect to temperature has been discussed.

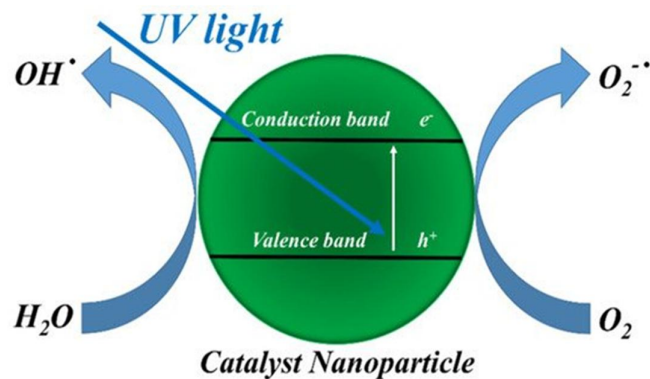


Figure 1.7 : Photocatalytic Application of Nanoparticles

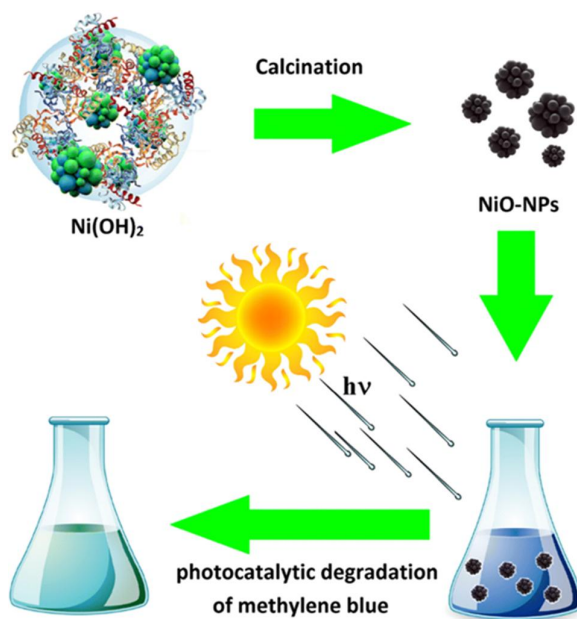


Figure 1.8 : Decolourization of Methylene Blue (MB) Dye Under Visible Light Irradiation

CHAPTER – 2

LITERATURE SURVEY

In the last two decades the nanoscience have been an exciting area of research as much of application point of view as of fundamental research.

Sankar, S., Sharma, S. K., An, N., Lee, H., Kim, D. Y., Im, Y. B., ... Purohit, L. P. (2016). *Photocatalytic properties of Mn-doped NiO spherical nanoparticles synthesized from sol-gel method. Optik, 127(22), 10727–10734.*

Akbari, A., Sabouri, Z., Ali Hosseini, H., Hashemzadeh, A., Khatami, M., & Darroudi, M. (2020). *Effect of nickel oxide nanoparticles as a photocatalyst in dyes degradation and evaluation of effective parameters in their removal from aqueous environments. Inorganic Chemistry Communications, 107867.*

Chen, W., Liu, F.-Z., Feng, X.-Y., Djurišić, A. B., Chan, W. K., & He, Z.-B. (2017). *Cesium Doped NiO x as an Efficient Hole Extraction Layer for Inverted Planar Perovskite Solar Cells. Advanced Energy Materials, 7(19), 1700722.*

El-Kemary, M., Nagy, N., & El-Mehasseb, I. (2013). *Nickel oxide nanoparticles: Synthesis and spectral studies of interactions with glucose. Materials Science in Semiconductor Processing, 16(6), 1747–1752.*

Dubal, D. P., Lee, S. H., & Kim, W. B. (2012). *Sponge-like β -Ni(OH)₂ nanoparticles: synthesis, characterization and electrochemical properties. Journal of Materials Science, 47(8), 3817–3821.*

Primc, D., Belec, B., & Makovec, D. (2016). *Synthesis of composite nanoparticles using co-precipitation of a magnetic iron-oxide shell onto core nanoparticles. Journal of Nanoparticle Research, 18(3).*

Zorkipli, Nurul Nadia Mohd, Noor Haida Mohd Kaus, and Ahmad Azmin Mohamad used the sol-gel process to synthesis nickel oxide (NiO) nanoparticles. The pH of the solution was kept at 11, and the calcination temperature was at 450°. composition, morphology, and particle size of NiO was analysed. The

cubic structure of NiO was formed without impurities, according to structural analysis. The ratio of NiO, Ni, and O was observed through morphological and elemental studies.

Zorkipli, Nurul Nadia Mohd, Noor Haida Mohd Kaus, and Ahmad Azmin Mohamad. "Synthesis of NiO nanoparticles through sol-gel method." Procedia chemistry 19 (2016): 626-631.

Khairnar, Subhash Dharmraj, and Vinod Shankar Shrivastava have synthesised NiO nanoparticles through chemical synthesis method. The NiO NPs that were synthesised were found to be effective for removing dyes from their aqueous suspensions. The result reveals that the NiO NPs synthesised have better photocatalytic activity for removing MB dye from aqueous suspension than the Rh B dye.

Khairnar, Subhash Dharmraj, and Vinod Shankar Shrivastava. "Facile synthesis of nickel oxide nanoparticles for the degradation of Methylene blue and Rhodamine B dye: a comparative study." Journal of Taibah University for Science 13, no. 1 (2019): 1108-1118.

Khandagale, Pooja, and Dipali Shinde have reported that nickel oxide nanoparticles were successfully synthesised using the co-precipitation process in this analysis (using nickel chloride & ammonia). characterization was done by using XRD, SEM, TEM, FTIR, and EDAX techniques. The results of the SEM study showed that the synthesised nanoparticles were rod-shaped. The particle size of Nanoparticle was found out using TEM data. The synthesised NiO nanoparticles can also be used for the degradation of toxic dyes.

Khandagale, Pooja, and Dipali Shinde. "Journal Homepage:-www.journalijar.com."

Tientong, Jeerapan, Stephanie Garcia, Casey R. Thurber, and Teresa D. Golden have Chemically synthesised, nickel powders with nanosized particles by the reduction of nickel salt with hydrazine hydrate in a simple aqueous solution at 55–65°C, using sonication to monitor the reduction. Titrating nickel chloride

solution with hydrazine-sodium hydroxide formed the nickel hydroxide nanopowder. The simplicity of operation, high yield, and small particle sizes are the most notable features of this process. Pure nanocrystalline powders with particle sizes varying from 7 to 14 nm were synthesised using this process.

Tientong, Jeerapan, Stephanie Garcia, Casey R. Thurber, and Teresa D. Golden. "Synthesis of nickel and nickel hydroxide nanopowders by simplified chemical reduction." Journal of Nanotechnology 2014 (2014).

Zuo, Ranfang, Gaoxiang Du, Weiwei Zhang, Lianhua Liu, Yanming Liu, Lefu Mei, and Zhaohui Li have confirmed that nano-TiO₂ has strong catalytic activity, but it is easy to agglomerate, low levels of photocatalytic activity or even total loss. Diatomite impregnated with TiO₂ provided an assisted anatase catalyst for MB photodegradation. MB can be totally degraded in 20 minutes at a concentration of 15 ppm. The results indicate that TiO₂/diatomite photocatalysis is a promising method for removing colour from wastewater.

Zuo, Ranfang, Gaoxiang Du, Weiwei Zhang, Lianhua Liu, Yanming Liu, Lefu Mei, and Zhaohui Li. "Photocatalytic degradation of methylene blue using TiO₂ impregnated diatomite." *Advances in Materials Science and Engineering* 2014 (2014).

Samuel, Joshua J., and F. K. Yam have confirmed that the use of dye sensitizers on TiO₂ was studied for use of a photocatalytic dye degradation process, which was tested using methylene blue in this case. When degraded with extracted chlorophyll, the concentration of methylene blue decreased to 40% of its initial concentration, according to the analysis. When completely optimised, the potential usage of dye sensitization as a cost-effective photocatalysis is the key for the effective waste vegetation and can also be useful to an ecofriendly approach of organic pollution reduction.

Samuel, Joshua J., and F. K. Yam. "Photocatalytic degradation of methylene blue under visible light by dye sensitized titania." *Materials Research Express* 7, no. 1 (2020): 015051.

CHAPTER – 3

AIM AND SCOPE

- Nickel based metal oxides are attracted much attention due to its applications in catalysis and dye degradation.
- Polyvinylpyrrolidone will be used as surfactant to reduce particles size
- Influences of Cesium salt on the size, morphology and structural constitution will be studied.
- Low temperature based hexagonal structure of Cs (5%) ion doped $\text{Ni}(\text{OH})_2$ nanoparticles will synthesized using the chemical precipitation method.
- Investigation on photocatalytic performances of Cs doped $\text{Ni}(\text{OH})_2$ nanoparticles will be synthesized.
- Effect of the photocatalytic activity Methylene Blue dye will be performed using undoped and Cs doped NiO nanoparticles will be investigated.
- Studies on size, morphology and structure will be characterize using X-Ray Diffraction [XRD], Scanning Electron Microscope [SEM], Fourier Transform Infrared Spectroscopy [FTIR].

CHAPTER- 4

EXPERIMENTAL DETAILS

Chapter 4 deals with the details of the synthesis and characterization techniques used for the prepared metal oxide nanomaterials targeted in this project. Techniques used in this project, i.e., co-precipitation method have been used for the synthesis of various undoped and doped nanomaterials explored in this project. The synthesized nanomaterials were examined by variety of techniques in terms of the morphological, structural, optical, compositional and electrical properties. Moreover, the prepared nanomaterials were used for various applications such as photo dye degradation and photocatalytic applications. In a word, this chapter provides all the detailed procedures for the synthesis, characterizations and applications of targeted nanomaterials in this project.

CHAPTER - 4

4.1 INTRODUCTION

The metal oxide nanomaterials in this project are NiO, Cs-doped Ni (OH)₂. The metal oxide nanomaterials were prepared by specific technique, i.e., simple and facile low-temperature co-precipitation process. The synthesized NiO and at low temperature β -Ni(OH)₂ nanostructures were extensively characterized in terms of their morphological, structural, optical, compositional and electrical properties. To examine the structural properties, the prepared nanomaterials were characterized by XRD pattern. The elemental and chemical compositions of prepared nanomaterials were investigated by FTIR spectroscopy. The optical properties were examined by UV-visible spectroscopy at room-temperature. Finally, the synthesized nanomaterials were used for photocatalytic applications.

4.2 MATERIALS

All chemicals used in this project were analytical grade and used without any further purification. Nickel (II) chloride Hexahydrate (NiCl₂.6H₂O) (MERCK), Molecular weight of NiCl₂.6H₂O is 237.69 g/mol, caesium chloride (Cscl), (Sigma Aldrich) molecular weight of Cscl is 168.36 g/mol, Sodium Hydroxide (NaOH), (SRL) Molecular weight of NaOH is 40 g/mol, Polyvinyl pyrrolidone (PVP), (SRL) Molecular weight of PVP is 2.50 g/mol, Ethanol, Acetone (SRL), and All samples were prepared by using De-ionised water.

4.3 EXPERIMENTAL PROCEDURE

Pure NiO, and Cs doped Ni (OH)₂ were synthesized by co-precipitation method.

4.3.1 *Synthesis of NiO*

NiO nanoparticles were synthesized by co-precipitation of Ni (OH)₂ by modification of the method. Nickel (II) chloride hexahydrate (0.5M) in D.I water were used as precursors or solvents, and to reduce agglomeration add 1g of polyvinyl pyrrolidone (PVP). The pH was adjusted by sodium hydroxide by

adding drop by drop. The reaction was stirred for 2 hours at room temperature. The resultant product was washed thoroughly with deionized water for removal of reaction residues followed by washing with acetone. Finally, deep green nanoparticles were formed, and dried in vacuum. The Nickel hydroxide nanoparticles were converted to NiO via calcination of the prepared sample at 450°C.

4.3.2 Synthesis of Ni (OH)₂

Cs doped Ni (OH)₂ nanoparticles were synthesized by co-precipitation method. Nickel (II) chloride hexahydrate (0.5M) in D.I water were used as precursors or solvents, add 5% of caesium chloride and to reduce agglomeration add 1g of polyvinyl pyrrolidone (PVP). The pH was adjusted by sodium hydroxide by adding drop by drop. The reaction was stirred for 2 hours at room temperature. The resultant product was washed thoroughly with Deionized water for removal of reaction residues followed by washing with acetone. Finally, deep green nanoparticles were formed, and dried at 120°C.

4.4 CHARACTERIZATION OF PREPARED METAL OXIDE NANOMATERIALS

The prepared metal oxide nanomaterials presented in this project were characterized in detail in terms of their morphological, structural, compositional and optical properties. The structural properties were examined by XRD in the range of 10-80°. Images with sufficient quality of nanoparticle size was characterized by scanning electron microscope (SEM). The elemental and chemical compositions were examined by using FTIR spectroscopy. The optical properties were examined by using UV-visible spectroscopy at room-temperature.

4.4.1 X-Ray Diffraction

X-Ray diffraction is a non-destructive technique used to determine the crystal structure of the material. A crystal lattice is a regular three-dimensional distribution of atoms in space are arranged to form a parallel plane separated by

one another by a distance (d), The XRD pattern obtained from the sample depends on the shape and size of basic unit cell of the crystal [55-59]. To determine the structural nature of a material after its synthesis is very essential. XRD are the major technique to determine the crystal structure of a material because the properties of a material are very much dependent on its structure. The standard database (JCPDS database) for XRD pattern is used for phase identification for a large variety of crystalline phases in the solid specimens. X-Ray Diffraction (XRD) has an advantage of simultaneous characterize both the precursor and end products with a detailed nanoparticle behaviour [60]. XRD is used for assessment of phase purity and crystallinity of inorganic materials.[61]. X-ray diffraction is based on Bragg's law

$$(n\lambda = 2d\sin\theta)$$

where d is the perpendicular distance between pairs of adjacent planes, θ is the angle of incidence or Bragg angle, λ is the wavelength of the beam, and n denotes an integer number, known as the order of the reflection and is the path difference, in terms of wavelength, between waves scattered by adjacent planes of atoms [62]. A monochromatic beam of X-rays is allowed to incident on a sample, and the reflected X-rays are detected by a detector [63]. Fig 4.1 shows the working principle of XRD. The XRD patterns of the synthesized materials were evaluated with Debye-Scherrer equation

$$D = K \lambda / \beta \cos(\theta)$$

Where D is the average crystalline size, λ is the wave length of the incident light (Cu-K α) (1.5405Å), β is the Full Width Half Maximum (FWHM) of the peak, K is the particle shape coefficient (0.9 for spheres), θ is the diffraction angle.

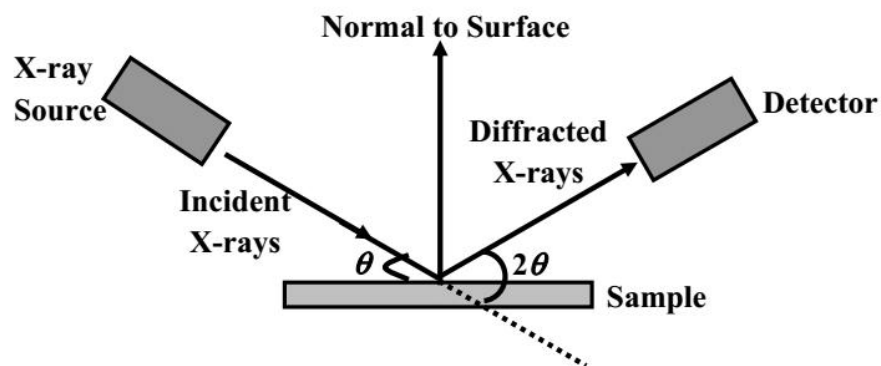


Figure 4.1 : Working Principle of XRD

4.4.2 Scanning Electron Microscope

Scanning electron microscope is a type of electron microscope, which produce images of a samples by scanning the surface of a focused beam of a high-energy electron to generate a variety of signals at the surface of solid specimens. Scanning electron microscope (SEM) is one of the most widely used instrumental methods for the examination and analysis of micro and nanoparticle imaging characterization of solid objects. Fig 4.2 shows the process of SEM. One of the reasons that SEM is preferred for particle size analysis is due to its resolution of 10 nm, that is, 100 Å. This instrument may also be used in conjunction with other related techniques of energy-dispersive X-ray microanalysis (EDX, EDS, EDAX), for the determination of the composition or orientation of individual crystals or features [64]. EDX (Energy dispersive X- Ray analysis) may also include specific information on elemental composition.

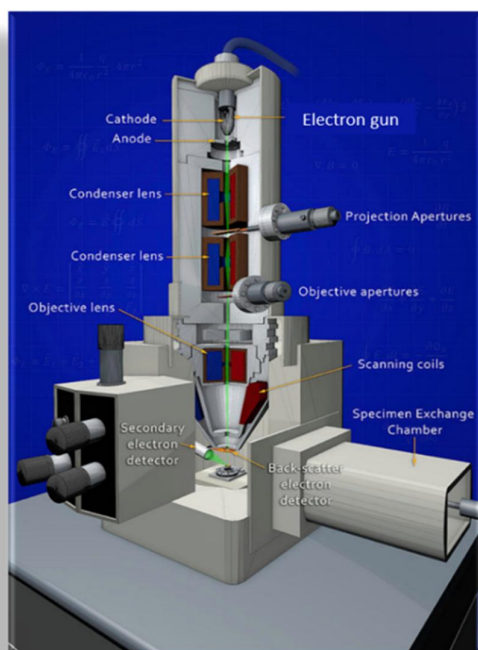


Figure 4.2 : The Process of Scanning Electron Microscope

4.4.3 Fourier Transform Infrared Spectroscopy

FTIR the term refers as Fourier transform infrared spectroscopy; it is a vibrational spectroscopic technique. Based upon the wavenumber, infrared light can be categorized as far infrared ($4 \sim 400 \text{ cm}^{-1}$), mid infrared ($400 \sim 4,000 \text{ cm}^{-1}$) and near infrared ($4,000 \sim 14,000 \text{ cm}^{-1}$) are shown in Fig.4.3. The structure and the components of a prepared samples are verified by spectroscopic techniques. if IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, and giving a picture of the molecular energy levels, is shown in Fig 4.4. It is used to identify unknown materials, and to determine the quality or consistency of a sample and the amount of components in a mixture. FTIR analysis provides information about the chemical bonding or molecular structure of materials, whether organic or inorganic. FTIR will measure samples with a diameter of up to 11mm and weigh the bulk or the top 1 mm of the sample [65]. The goal of any absorption spectroscopy (FTIR, UV-Visible spectroscopy, etc.) is to measure how much a sample absorbs light at each wavelength. In order to

obtain infrared spectra, a Fourier Transform Infrared (FTIR) spectrometer first generates an interferogram of a sample signal with an interferometer, which simultaneously analyses all infrared frequencies. The incoming infrared beam is split into two optical beams by a beam splitter in an interferometer. A flat mirror, which is fixed in place, reflects one beam. A flat mirror reflects another beam just moves a short distance (typically a few milli meters) away from the beam splitter. the two beams reflect from their respective mirrors. When they are meet together at the beam splitter, they are recombined. The same frequencies of energy are adsorbed by the sample when the interferogram signal is transmitted through the sample surface due to the excited vibration of function groups in molecules. The infrared signal generated by interacting with the sample is unique to the sample. When the beam reaches the detector, it is measured by the detector. It is difficult to interpret the observed interferogram directly. It must be "decoded" using a mathematical method known as Fourier Transformation. The computer will evaluate the Fourier transformation and display an infrared spectrum that plots adsorbance (or transmittance) against wavenumber.

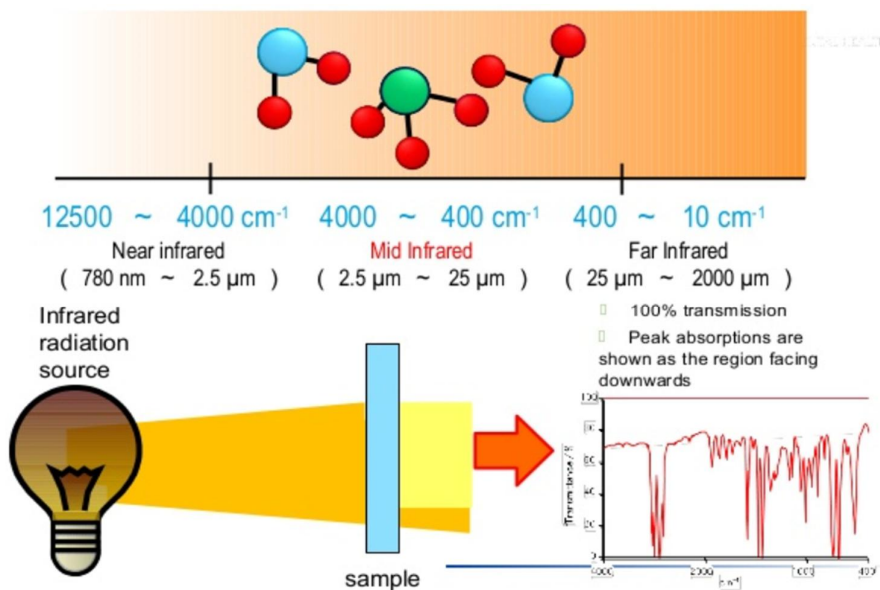


Figure 4.3 : Wave Number Classification of FT-IR Spectroscopy

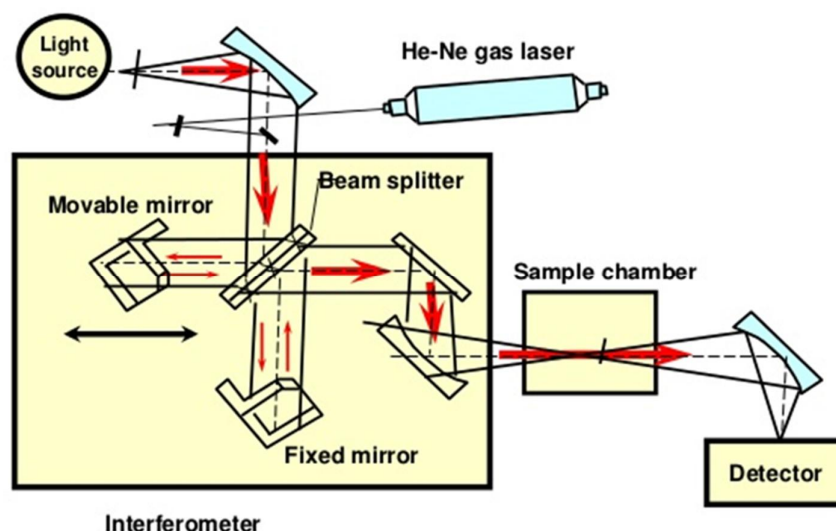


Figure 4.4 : The Working Principle of FTIR

4.4.4 UV-Visible Spectroscopy

This technique is based on the light absorption in the UV-visible spectral regions, in which molecules and chemical compounds undergo electronic transitions, and hence to determine the electronic bonding in a molecule [66]. It is known as absorption spectroscopy. UV-Visible spectroscopy cover a wavelength of ranging up to 200-800nm. UV-visible spectroscopy is produced when incident radiation interacts with the electron cloud in a chromophore, resulting in an electronic transition involving the promotion of one or more outer shell or bonding electrons from a ground state to a higher energy state. In a standard UV-Vis spectrometer, the beam of light is split into two parts by a partial mirror; one half of the beam (the sample beam) is directed through a transparent cell containing a solution of the compound being analysed, and one half (the reference beam) is directed through an identical cell that does not contain the compound but contains the same solvent wherein the compound is dissolved. The solvents are must to be taken care of to be transparent for the interested light. The difference of light intensities scanned over a range of wavelength are taken and compared. The intensity profile over a range of wavelengths is observed. In complex molecules the energy levels are more closely spaced and

photons of near ultraviolet and visible light can affect the transition. These substances, therefore, will absorb light in some areas of the near ultraviolet and visible regions [67]. UV-Vis. spectroscopy was used to examine the optical properties of prepared metal oxide nanomaterials. Fig 4.5 shows the working principles of UV-Visible spectroscopy. metal nanoparticle light absorption is produced by the coherent oscillation of their conduction band electrons, induced by the interaction of the electromagnetic of the incident light.

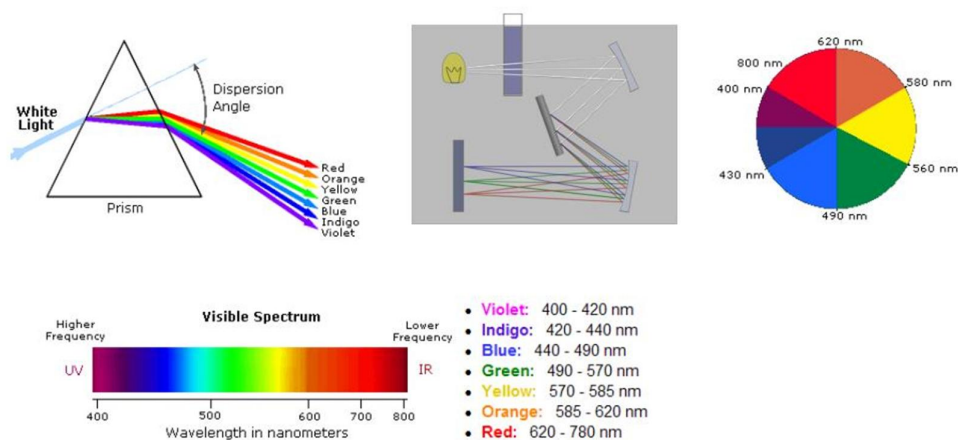


Figure 4.5: Working Principle of UV-Visible Spectroscopy

CHAPTER - 5

RESULTS AND DISCUSSION

Chapter 5 describes the main results and discussion of the project. This chapter is divided into several sections and each section describes the synthesis, detailed characterizations and particular application of metal oxide nanomaterial.

Section 5.1 describes X-Ray diffraction of the NiO and Cs doped Ni(OH)₂ crystalline structure, characterization and applications of nanoparticle grown via facile and simple co-precipitation method using commonly used laboratory chemicals.

Section 5.2 demonstrates the SEM images and their characterization based on Cs doped Ni(OH)₂ and NiO.

Section 5.3. demonstrates the characterization of (FTIR) in this chapter.

Finally, Section 5.4 exemplifies the photodegradation activity of NiO and Cs doped Ni(OH)₂.

CHAPTER-5

RESULTS AND DISCUSSION

In this project, we done a characterization analysis of NiO and Cs doped Ni(OH)₂ nanoparticles using co-precipitation method and observing a structure, morphology, crystal size of our prepared samples using X-Ray diffraction, Scanning Electron Microscope [SEM], Fourier Transform Infrared spectroscopy [FTIR], UV-Visible spectroscopy. And we plot the points and calculate the crystal and particle size of our sample. Fig 5.1 clearly seen as green in colour then we identify, it is known as formation of Ni(OH)₂ and Fig 5.2 clearly seen as black in colour, then we identify, it is the formation of NiO



Figure 5.1 : Photocopy of Prepared Cs Doped Ni(OH)₂



Figure 5.2 : Photocopy of Prepared NiO

5.1 X-RAY DIFFRACTION

The structural properties of nanoparticles can be revealed using XRD, it is a unique technique. It gives more details on the phase, chemical composition, unit cell symmetry, crystallinity, lattice constant, and nanoparticle properties. This technique can be used to identify both single and multiphase nanoparticles [68-69].

Only when the X-rays interact with the crystal will you get a diffraction pattern. The diffraction patterns include information such as the unit cell's symmetry, stress, strain, crystallite size, and growth orientation etc. This standard data will be identified by (JCPDS database) XRD will provide you more detail about the crystallite size. The Debye Scherrer equation can be used to measure the average crystallite size based on the peak broadening of the diffraction peaks:

$$D = K\lambda/\beta \cos \theta$$

crystalline parameters of the prepared NiO nanoparticle sample through a Cu K α source wavelength of $\lambda = 1.5405\text{\AA}$, $K = 0.9$, = full width half maximum. Diffraction pattern was noted at a number of differing angles of diffraction (2θ) between 10° and 90° .

Figure 5.3 NiO shows the Co-Precipitation method, XRD patterns of NiO hollow spheres, with five major diffraction peaks, 111, 200, 220, 311, and 222, corresponding to the standard cubic NiO structure (JCPDS card 04-0835). There was no impurity-phase peak detected, showing that the products are pure NiO in its pure phase.

Figure 2 shows the XRD patterns of NiO hollow spheres by hydrothermal method, five main diffraction peaks, namely (111), (200), (220), (311) and (222), matched with the standard cubic NiO structure (JCPDS card 04-0835). No impurity-phase peak was found, demonstrating the products are pure phase NiO. Because there are two different shapes, we put them in

the same XRD picture together Figure 5.2 shows the XRD patterns of NiO hollow spheres by hydrothermal method, five main diffraction peaks, namely (111), (200), (220), (311) and (222), matched with the standard cubic NiO structure (JCPDS card 04-0835). No impurity-phase peak was found, demonstrating the products are pure phase NiO. Because there are two different shapes, we put them in the same XRD picture together Figure 5.2 shows the XRD patterns of NiO hollow spheres by hydrothermal method, five main diffraction peaks, namely (111), (200), (220), (311) and (222), matched with the standard cubic NiO structure (JCPDS card 04-0835). No impurity-phase peak was found, demonstrating the products are pure phase NiO. Because there are two different shapes, we put them in the same XRD picture together.

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Figure 5.3 Cs-doped Ni (OH)₂ spheres which exhibits several well-defined diffraction reflections at 19.12°, 31.78°, 38.61°, 45.461°, 51.87°, 59.25° and 75.18°. The XRD patterns of the sample treated under various experimental conditions gives high purity and crystallinity. According to (JCPDS card No 14-0117), all of the peaks in Figure 5.3 shows as Cs doped β -Ni(OH)₂ can be indexed as hexagonal phase. The XRD of bare Ni substrate, which showed two peaks at 45° and 75°. The diffraction peaks for the plane are (001), (100), (101), (102), and (110).

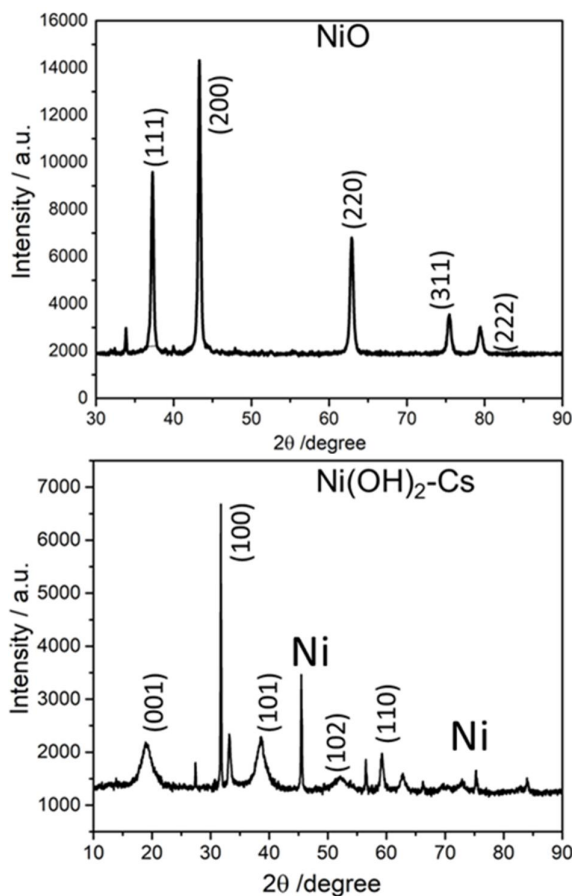


Figure 5.3 : XRD of NiO and Cs Doped Ni (OH)₂

The average crystallinity size of the NiO nanoparticle is 14.3 nm was calculated using XRD

5.2 SCANNING ELECTRON MICROSCOPY

The high-resolution scanning electron microscope (SEM) is ideal for determining nanoparticle size and shape. Because the necessary sample preparation and image acquisition are relatively fast and simple, Despite the fact that the SEM image is a two-dimensional (2D) representation of the three-dimensional (3D) objects from a precise viewing angle, The SEM image does contain some 3D information that can be used to recreate the shape of a basic structure with sub-nm precision using model-based measurement [70]. The morphological variations in the sample that were synthesized are observed and

analysed. Figure 5.4(a) displays a SEM analysis of nickel oxide. The images of Cs doped nickel hydroxide nanoparticles thus synthesized at low temperatures were analysed. In Figure 5.4(b)

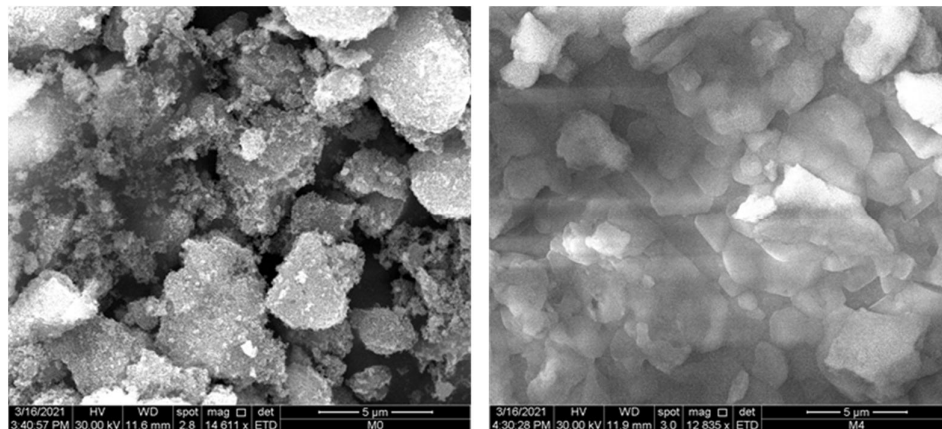


Figure 5.4 : (a) Scanning Electron Microscopy (SEM) Image of Pure NiO Nanoparticles and (b) SEM Image of Cs Doped Ni (OH)₂ Nanoparticles. The SEM Images were Measured for Powder Samples for the Both Samples (NiO and Cs doped Ni (OH)₂). The Powder Samples were Pasted on the Carbon Tape for Measuring the SEM Images

Since nickel oxide is agglomerated, it has a higher surface energy than caesium doped nickel hydroxide [71]. caesium doped nickel hydroxide has finite structures as compared to pure nickel oxide. Results shows that the synthesised NiO nanoparticles are smooth, spherical in shape, and some are agglomerated due to their high surface area to volume ratio and lower cohesive energy than pure NiO nanoparticles compared to bulk materials. Caesium doped nickel hydroxide SEM images are spherical. Because of its high dispersion, caesium doped nickel hydroxide has a limited size relative to pure nickel oxide [72], Nickel oxide can be de-agglomerated by calcination at a higher temperature of around 160°C [73]. The size of caesium doped nickel hydroxide, according to SEM photos, may be less than 10nm.

5.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY

The absorbance of infrared light at different wavelengths in a sample is measured using FTIR study to analyse the material's molecular composition and structure.

The O-H bond is represented by a peak on the FT-IR spectrum about 3422 cm^{-1} . The hydroxyl groups are obtained, due to the absorption at 1630 cm^{-1} . The presence of carbonates is confirmed by the absorption bonds at 1440 cm^{-1} and 1126 cm^{-1} , while the bond at 2900 cm^{-1} corresponds to the CH_2 stretching mode [74].

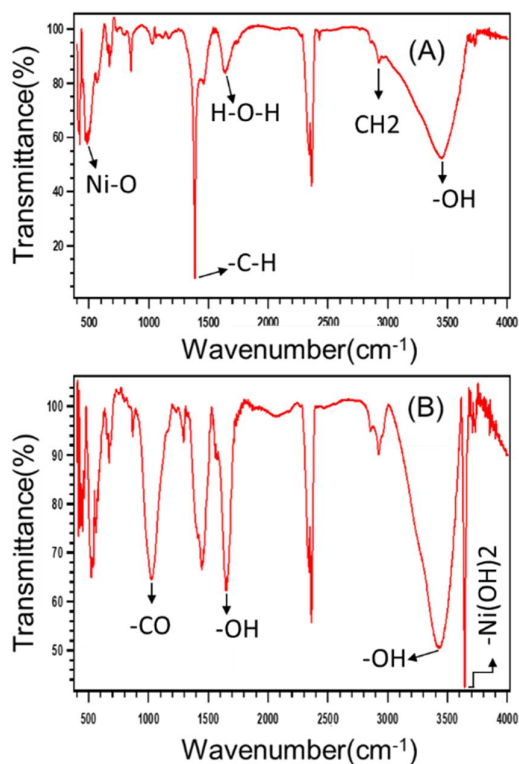


Figure 5.5 : (A) NiO , (B) Ni(OH)_2

The absorption bonds at 460 and 580 cm^{-1} are associated to the Ni-O vibration bond, as shown in fig.5.5 (A)

The FT-IR spectra of Cs doped Ni(OH)_2 are shown in Figure 3.3 (B) reveals chemical information and major functional groups in the Ni(OH)_2 . The

sharp peak at 3642 cm^{-1} is assigned to the stretching vibrational mode (OH) of nonhydrogen bonded hydroxyl groups in the Nickel hydroxide, while the broad band at 3445 cm^{-1} to the stretching mode of hydrogen bonded hydroxyl groups in the same layered structure. The peak at 516 cm^{-1} is attributed to the in-plane deformation vibration of water (OH) [75].

5.4 PHOTOCATALYTIC ACTIVITY TEST

Methylene blue (MB) was chosen as the model organic pollutants to test the photocatalytic dye degradation analysis for the catalyst. Typically, a catalyst (20 mg) was added into the 50 ml of aqueous dye solution (20 mg L^{-1}) then, the dye solution was kept in dark room to attain the adsorption and desorption equilibrium of dyes on surface of catalyst. Then, the dye solution was irradiated on sunlight for overall time period of 120 minutes. 2 ml of solution was taken at appropriate interval of time. The collected solution was centrifuged and filtered using a $0.2\text{ }\mu\text{m}$ syringe filter. The degradation efficiency is determined by the degradation of organic dyes on UV-Visible spectrophotometer. The degradation efficiency is calculated by the formula,

$$\text{Degradation efficiency (\%)} = \frac{C - C_0}{C} \times 100 \quad (1)$$

Where C and C_0 are initial and final concentration of dye molecules. After 1st completed the photocatalytic reaction, the catalyst was collected by centrifuge and washed with water and dried at 70°C . The regenerated catalyst was used to evaluate the stability and reusability of catalyst by degradation the dye solution.

The first order kinetic reaction value was used to determine the correct degradation rate of MB as follows;

$$-\ln(C/C_0) = kt \quad (2)$$

Where, k = rate of reaction constant and t is time. The Photocatalytic dye degradation activity were performed during the well sun shining between 11:00 AM to 2:00 PM in clear sky. The average solar light intensity was measured about 1.00×10^5 lux. Likewise, the effect of some other important operational parameters such as pH of dye solution and catalyst dosage study were

performed. In the pH test, dye solution pH was adjusted by the addition of 0.1 M hydrochloric acid solution and 0.1M of sodium hydroxide solution.

Photocatalytic activity :

The photocatalytic degradation efficiency of as prepared catalysts of NiO, and Cs/ Ni(OH)₂ nanoparticles were resolved by the degradation of MB dye under irradiation of Sun light. When irradiating the MB dye solution, the concentration of the dye was noticeably reduced and it was confirmed by the significant colour variation. For NiO catalyst, the catalyst influences the organic molecule to achieve the efficiency of about 46.32% within 120 mins. The photocatalyst experiment was further extended to doped samples. The degradation reaction of Cs/ Ni(OH)₂ samples were performed by the same process. The rate of electron-hole pair recombination was delayed by doping the metal ions into the NiO structure results increased the degradation efficiency. Compared to NiO catalyst, Cs/Ni(OH)₂ catalysts reached high efficiency of degradation of 86.39%, respectively [76].

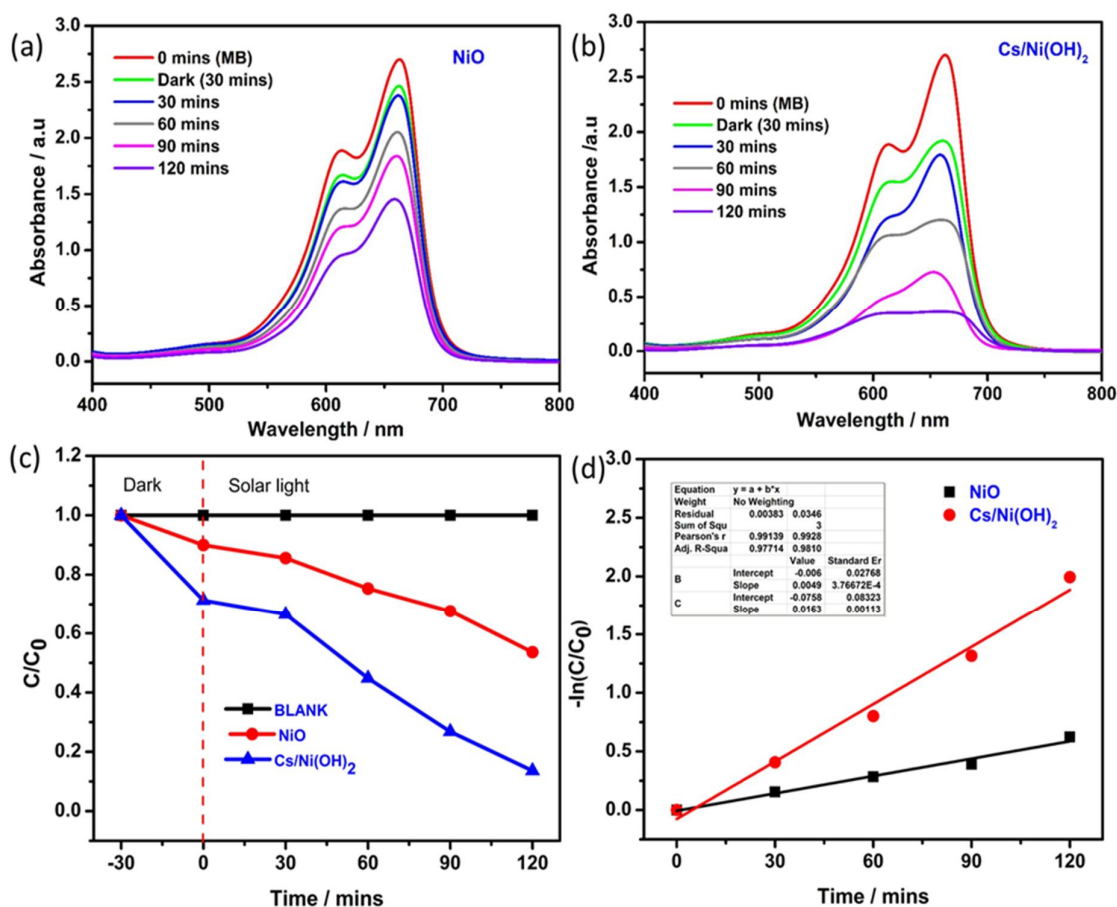


Figure 5.6.1 : (a-b) Represents the UV-visible Spectra for the MB with Occurrence of Catalysts NiO, and Cs/ Ni(OH)₂ were Irradiated by Sun Light for 120 minutes. (c) C/C₀ Versus Time Interval Plot of MB dye (d) -ln (C/C₀) Versus Time Interval Plot of MB dye

The significant improvement of the degradation efficiency is due to the doping effect of Cs ion at the Ni²⁺ lattice sites, it reduce recombination of excited charge carriers (electron-hole pair). Figure 5.6.1 (c) depicts the changes in initial and final concentration of dye in the presence and absence of photocatalyst against the light irradiation with respect to time (t) for the MB dye solution. The Cs/Ni(OH)₂ catalyst shows highest degradation ability due the fact that sub energy-level act as traps for recombination. Based on the sub energy level of the conduction band helps to produce the large number of reactive

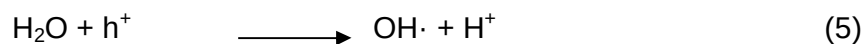
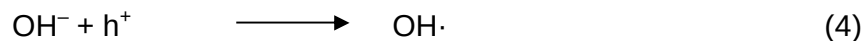
oxygen species (ROS). Addition investigation of pseudo-first order kinetics studies shown in Figure.5.6.1 (d) the degradation rates were 0.0112 min^{-1} and 0.0375 min^{-1} for NiO, and Cs/ Ni(OH)₂ nanoparticles against MB dye, respectively. From the studies rate constant value and R² values are listed in the Table.2 The Figure.5.6.2 showed schematic diagram illustrating the mechanism of charge separation, generation of reactive oxygen species (ROS) and photocatalytic activity of photocatalyst under the sunlight irradiation [77].

The mechanism of MB dye degradation for the Cs/Ni(OH)₂ catalyst is described as follows,

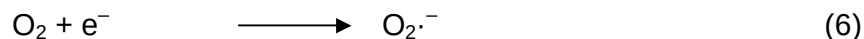
(i) Formation of electron hole pair



(ii) Formation of hydroxyl radicals



(iii) Formation of superoxide radicals



(iv) Photo degradation of crystal violet and rhodamine B dye



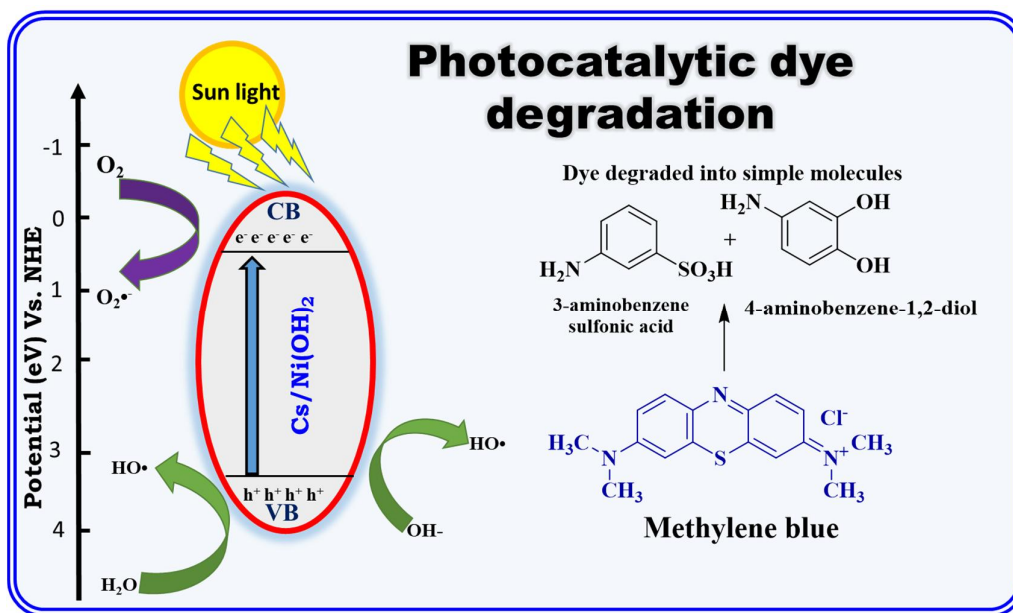


Figure 5.6.2 : Schematic Diagrams Illustrating the Mechanism of Charge Separation and Photocatalytic Activity of Photocatalyst Under the Sunlight Irradiation

TABLE 2 : PHOTOCATALYTIC ACTIVITY RESULT OF NIO AND CS/ Ni(OH)₂ NANOPARTICLES

S.No	Catalyst	Band gap (eV)	Degradation efficiency (%) (120 minutes)	K (min ⁻¹)	R ²
Methylene blue (20 ppm)					
1	NiO	3.21	46.32	0.0112	0.9772
2	Cs/Ni(OH) ₂	2.82	86.39	0.0375	0.9810

CHAPTER - 6

CONCLUSION

In recent years, numerous intensive research efforts in the field of nanotechnology have shown great potentials. There have been significant improvements for the synthesis of desired inorganic nanomaterials and their several potential applications are presented by the scientists and reported in the literature. The development of nanomaterials helps us to move towards a bright and comfortable future. These materials offer more precise methods of addressing the several unsolved problems and needs of the mankind. There are endless possibilities for improved devices, structures and materials if understanding can be developed for assembly at small structures.

Nanotechnology has reached to the level of leading science with fundamental and research prospects in all basic cognitive sciences. An important feature of the nanotechnology is to bridge the gap between the atomic and molecular scale of fundamental sciences

The overall conclusions of our work as presented in the previous chapter is presented here systematically as,

A brief introduction of metal oxide nanostructures, their various synthetic methods and the properties of targeted metal oxides, i.e. undoped and doped $\text{Ni}(\text{OH})_2$ have been presented in chapter 1. In addition to this, the utilization of prepared metal oxide nanomaterials for photocatalytic applications are also demonstrated in this chapter. Finally, the chapter 1 describes the objective and importance of this project.

The prepared metal oxide nanomaterials were mainly synthesized by co-precipitation processes and characterized in detail in terms of their morphological, structural, compositional, optical and electrical properties. Further, the prepared metal oxide nanomaterials were used for the various highly efficient photo dye degradation and photocatalytic applications. These all details are mentioned in chapter 2.

The main results and discussion are demonstrated and discussed in chapter 3.

CHAPTER - 6

6.1 SUMMARY AND CONCLUSION

- ❖ Nickel based metal oxides are attracted much attention due to its applications in catalysis, dye degradation, battery cathodes, antiferromagnetic layers, p-type transparent conducting films, and electrochromic films.
- ❖ Polyvinylpyrrolidone was used as surfactant to reduce particles size
- ❖ Herein, high temperature based cubic phase of NiO nanoparticles were synthesized. At first, the Ni(OH)_2 was synthesized chemical precipitation method and then the wet sample was calcinated at 500°C for 4 hours at air atmosphere.
- ❖ Low temperature based hexagonal structure of Cs (5%) ion doped Ni(OH)_2 nanoparticles were synthesized using the chemical precipitation method.
- ❖ Crystallite structure and size were confirmed by XRD. The size of the crystallite was calculated using Debye Scherrer formula, the size was found that 14.3 nm and 8.1 nm for NiO and Cs doped Ni(OH)_2 nanoparticles, respectively.
- ❖ The SEM microscopy demonstrated the synthesized nanoparticles were in nanoscale region with spherical shape.
- ❖ Organic functional groups presence on the surface of the nanoparticles were identified using FT-IR spectroscopy.

- ❖ The investigated photocatalytic result demonstrated that Cs doped Ni(OH)_2 showed as excellent dye degradation of Methylene blue (MB) than the pure NiO nanoparticles due to its effective charge carriers creation and followed by the free radical formation during the light irradiation.
- ❖ Thus, the prepared cost effective and low temperature based Cs doped Ni(OH)_2 could be the suitable candidate for water purification, through the photocatalysis process.

REFERENCES

- [1] C. N. R. Rao, A. Muller, A. K. Cheetham, *The Chemistry of Nanomaterials: Synthesis, Properties and Applications* Willey, Weinheim (2004).135
- [2] A. K. Bandyopadhyay, *Nano Materials: in Architecture, Interior Architecture and Design*, New Age International, New Delhi (2008).
- [3] W.J. Stark, P.R. Stoessel, W. Wohlleben, A. Hafner, Industrial applications of nanoparticles, *Chem. Soc. Rev.* 44 (2015) 5793–5805.
- [4] K. Pushpanathan, S. Sathya, M.J. Chithra, S. Gowthami, & R. Santhi . Influence of reaction temperature on crystal structure and band gap of ZnO nanoparticles. *Materials and Manufacturing Processes*, 27(12) (2012), 1334-1342.
- [5] Meindl, D. James, Qiang Chen, and Jeffrey A. Davis. "Limits on silicon nanoelectronics for terascale integration." *Science* 293.5537 (2001): 2044-2049.
- [6] C. M. Leiber, *Sci. Am.* 58 (2001).
- [7] Yang, Peidong. "The chemistry and physics of semiconductor nanowires." *MRS bulletin* 30, no. 2 (2005): 85-91.
- [8] Coffey, C. David, and David S. Ginger, "Time-resolved electrostatic force microscopy of polymer solar cells." *Nature materials* 5, no. 9 (2006): 735-740.
- [9] Muccini, Michele. "A bright future for organic field-effect transistors." *Nature materials* 5, no. 8 (2006): 605-613.
- [10] Nomura, Kenji, Hiromichi Ohta, Akihiro Takagi, Toshio Kamiya, Masahiro Hirano, and Hideo Hosono. "Room-temperature fabrication of transparent

flexible thin-film transistors using amorphous oxide semiconductors." *nature* 432, no. 7016 (2004): 488-492.

- [11] J. L. G. Fierro, *Metal Oxides: Chemistry and Applications*, CRC Press, Florida (2005).
- [12] V.E., Henrich, and P. A. Cox. "The surface chemistry of metal oxides." *Cambridge University Press: Cambridge, UK* (1994).
- [13] C. Noguera, *Physics and Chemistry at Oxide Surfaces*, Cambridge University Press, Cambridge, UK (1996).
- [14] Rodríguez, A. José, and Marcos Fernández-García, eds. *Synthesis, properties, and applications of oxide nanomaterials*. John Wiley & Sons, 2007.
- [15] Wu, Yiyang, Haoquan Yan, Michael Huang, Benjamin Messer, Jae Hee Song, and Peidong Yang. "Inorganic semiconductor nanowires: rational growth, assembly, and novel properties." *Chemistry—A European Journal* 8, no. 6 (2002): 1260-1268.
- [16] C.N.R., Rao, F. Leonard Deepak, Gautam Gundiah, and Achutharao Govindaraj. "Inorganic nanowires." *Progress in Solid State Chemistry* 31, no. 1-2 (2003): 5-147.
- [17] Dai, Zu Rong, Z. Wei Pan, and Z. L. Wang. "Novel nanostructures of functional oxides synthesized by thermal evaporation." *Advanced Functional Materials* 13, no. 1 (2003): 9-24.
- [18] Bavykin, V. Dmitry, Jens M. Friedrich, and Frank C. Walsh. "Protonated titanates and TiO₂ nanostructured materials: synthesis, properties, and applications." *Advanced materials* 18, no. 21 (2006): 2807-2824.
- [19] C. Nützenadel, A. Züttel, D. Chartouni, G. Schmid, and L. Schlapbach. "Critical size and surface effect of the hydrogen interaction of palladium clusters." *The European Physical Journal D* 8, no. 2 (2000): 245-250.

- [20] G. C. Bond, C. Louis, D. T. Thompson, *Catalysis by Gold*, Imperial College Press, London (2006).
- [21] G. Cao, "Nanostructures & Nanomaterials: Synthesis, Properties & Applications", Imperial College Press, London (2004).
- [22] C.J. Brinker, and G. W. Scherer. "Structural changes during heating: amorphous systems." *Sol-Gel Science: The Physics and Chemistry of Sol-Gel Processing* (1990): 555.
- [23] Hench, L. Larry, and Jon K. West. "The sol-gel process." *Chemical reviews* 90, no. 1 (1990): 33-72.
- [24] O'regan, Brian, Jacques Moser, Marc Anderson, and Michael Graetzel. "Vectorial electron injection into transparent semiconductor membranes and electric field effects on the dynamics of light-induced charge separation." *Journal of Physical Chemistry* 94, no. 24 (1990): 8720-8726.
- [25] Soytaş, Serap Hayat, Oğuzhan Oğuz, and Yusuf Ziya Menceloğlu. "Polymer nanocomposites with decorated metal oxides." In *Polymer Composites with Functionalized Nanoparticles*, 2019, pp. 287-323.
- [26] Huang, Guoming, Chun-Hua Lu, and Huang-Hao Yang. "Magnetic nanomaterials for magnetic bioanalysis." In *Novel Nanomaterials for Biomedical, Environmental and Energy Applications*, 2019, pp. 89-109.
- [27] da Silva, Amandha Kaiser, Thiago Gomes Ricci, Ana Lúcia de Toffoli, Edvaldo Vasconcelos Soares Maciel, Carlos Eduardo Domingues Nazario, and Fernando Mauro Lanças. "The role of magnetic nanomaterials in miniaturized sample preparation techniques." In *Handbook on Miniaturization in Analytical Chemistry*, 2020, pp. 77-98.
- [28] Gan, Yong X., Ahalapitiya H. Jayatissa, Zhen Yu, Xi Chen, and Mingheng Li. "Hydrothermal synthesis of nanomaterials." (2020).
- [29] O'Donoghue, Michael. *A guide to Man-made Gemstones*. Van Nostrand Reinhold, 1983.

- [30] Fernández-García, Marcos, and José A. Rodríguez. "Metal oxide nanoparticles." *Encyclopedia of inorganic and bioinorganic chemistry* (2011).
- [31] M.M. Khan, S.F. Adil, A. Al-Mayouf, "metal oxide as photocatalyst", *Journal of Saudi Chemical Society*, 19 (2015) 462-464.
- [32] Fig 4 reference Taiba Naseem, Tayyiba Durrani, "The role of some important metal oxide nanoparticles for wastewater and antibacterial applications: A review"
- [33] Wang, Dingsheng, Run Xu, Xun Wang, and Yadong Li. "NiO nanorings and their unexpected catalytic property for CO oxidation." *Nanotechnology* 17, no. 4 (2006): 979.
- [34] Rahdar, Abbas, M. Aliahmad, and Y. Azizi. "NiO nanoparticles: synthesis and characterization." (2015): 145-151.
- [35] Malik, Ritu, Pawan S. Rana, Vijay K. Tomer, Vandna Chaudhary, S. P. Nehra, and Surender Duhan. "Nano gold supported on ordered mesoporous WO₃/SBA-15 hybrid nanocomposite for oxidative decolorization of azo dye." *Microporous and Mesoporous Materials* 225 (2016): 245-254.
- [36] Ramesh, Martha, Martha Purna Chander Rao, Sambandam Anandan, and Hosakoppa Nagaraja. "Adsorption and photocatalytic properties of NiO nanoparticles synthesized via a thermal decomposition process." *Journal of Materials Research* 33, no. 5 (2018): 601-610.
- [37] Z. Sabouri et al., "Facile green synthesis of NiO nanoparticles and investigation of dye degradation and cytotoxicity effects." *J. Mol. Struct.* 1173, 931–936 (2018)
- [38] Freitas, M. B. J. G. "Nickel hydroxide powder for NiO·OH/Ni(OH)₂ electrodes of the alkaline batteries." *Journal of Power Sources* 93, no. 1-2 (2001): 163-173.

- [39] Hall, S. David, David J. Lockwood, Shawn Poirier, Christina Bock, and Barry R. MacDougall. "Raman and infrared spectroscopy of α and β phases of thin nickel hydroxide films electrochemically formed on nickel." *The Journal of Physical Chemistry A* 116, no. 25 (2012): 6771-6784.
- [40] Bagheri, Fathollah, and Saba Mosivand. "Ball-like nickel hydroxide nanoparticles: Electro-synthesis, characterization and application." *Materials Today Communications* (2020): 101714.
- [41] Cai, Feng-Shi, Guo-Ying Zhang, Jun Chen, Xing-Long Gou, Hua-Kun Liu, and Shi-Xue Dou. " β -Ni(OH)₂ tubes with mesoscale dimensions as positive-electrode materials of alkaline rechargeable batteries." *Angewandte Chemie* 116, no. 32 (2004): 4308-4312.
- [42] Liu, Xiaohong, and Lan Yu. "Synthesis of nanosized nickel hydroxide by solid-state reaction at room temperature." *Materials letters* 58, no. 7-8 (2004): 1327-1330.
- [43] X.J. Han, P. Xu, C. Q. Xu, L. Zhao, Z. B. Mo, and T. Liu. "Study of the effects of nanometer β -Ni(OH)₂ in nickel hydroxide electrodes." *Electrochimica Acta* 50, no. 14 (2005): 2763-2769.
- [44] Gao, Yuan, Huitao Liu, and Mianjun Ma. "Preparation and shape control of β -Ni(OH)₂ nano-particle." *Open Chemistry* 6, no. 1 (2008): 23-25.
- [45] Ma, Ming-Guo, Jie-Fang Zhu, Jian-Xin Jiang, and Run-Cang Sun. "Hydrothermal–polyol route to synthesis of β -Ni(OH)₂ and NiO in mixed solvents of 1, 4-butanediol and water." *Materials Letters* 63, no. 21 (2009): 1791-1793.
- [46] González-Perea, Mario, Martín M. Dávila-Jiménez, Aditaín García, María de la P. Elizalde-González, and Patricia Ruiz. "Electrosynthesis of β -Ni(OH)₂ and electrochemical response of β -Ni(OH)₂/CB-PVC composite electrode." *ECS Transactions* 36, no. 1 (2011): 299.

- [47] Tientong, Jeerapan, Stephanie Garcia, R. Casey, Thurber, and Teresa D. Golden. "Synthesis of nickel and nickel hydroxide nanopowders by simplified chemical reduction." *Journal of Nanotechnology* 2014 (2014).
- [48] Liang, Zhen-Hua, Ying-Jie Zhu, and Xian-Luo Hu. " β -Nickel hydroxide nanosheets and their thermal decomposition to nickel oxide nanosheets." *The Journal of Physical Chemistry B* 108, no. 11 (2004): 3488-3491.
- [49] Boschloo, Gerrit, and Anders Hagfeldt. "Spectroelectrochemistry of nanostructured NiO." *The Journal of Physical Chemistry B* 105, no. 15 (2001): 3039-3044.
- [50] Liu, X. M., and X. G. Zhang. "NiO-based composite electrode with RuO₂ for electrochemical capacitors." *Electrochimica acta* 49, no. 2 (2004): 229-232.
- [51] Nuli, Yan-Na, Sheng-Li Zhao, and Qi-Zong Qin. "Nanocrystalline tin oxides and nickel oxide film anodes for Li-ion batteries." *Journal of Power Sources* 114, no. 1 (2003): 113-120.
- [52] Ikram, Saiqa. "Role of Nanomaterials and their Applications as Photocatalyst and Senors: A Review." *Nano Res* 2, no. 10 (2016).
- [53] Chan, Samuel Hong Shen, Ta Yeong Wu, Joon Ching Juan, and Chee Yang Teh. "Recent developments of metal oxide semiconductors as photocatalysts in advanced oxidation processes (AOPs) for treatment of dye waste-water." *Journal of Chemical Technology & Biotechnology* 86, no. 9 (2011): 1130-1158.
- [54] Djurišić, B. Aleksandra, Yanling He, and Alan MC Ng. "Visible-light photocatalysts: Prospects and challenges." *APL Materials* 8, no. 3 (2020): 030903.
- [55] Kanjilal, D., S. Chopra, M. M. Narayanan, Indira S. Iyer, Vandana Jha, R. Joshi, and S. K. Datta. "Testing and operation of the 15UD Pelletron at

NSC." *Nuclear Instruments and Methods in Physics Research. Section A* 328, no. 1/2 (1993): 97-100.

- [56] J.F. Ziegler, J. P. Biersack, and K. Littmark. "Stopping and ranges of ions in Matter Pergamon Press." *New York* (1985).
- [57] Lal, Krishan. "Real structure of real crystals." *PROCEEDINGS-INDIAN NATIONAL SCIENCE ACADEMY PART A* 64 (1998): 609-636.
- [58] Pinsker, Zinovii Grigorievich. "Dynamical Scattering of X-rays in Crystals." (1978).
- [59] Batterman, W. Boris, and Henderson Cole. "Dynamical diffraction of X rays by perfect crystals." *Reviews of modern physics* 36, no. 3 (1964): 681.
- [60] Rajeswari, A., E. Jackcina Stobel Christy, Sreerag Gopi, K. Jayaraj, and Anitha Pius. "Characterization studies of polymer-based composites related to functionalized filler-matrix interface." *Interfaces in Particle and Fibre Reinforced Composites* (2020): 219-250.
- [61] Ijaz, Kashif, Hamad Khalid, and Aqif Anwar Chaudhry. "Zinc-substituted hydroxyapatite." In *Handbook of Ionic Substituted Hydroxyapatites*, pp. 217-236, 2020.
- [62] Patel, Jignesh P., and Parsotam H. Parsania. "Characterization, testing, and reinforcing materials of biodegradable composites." *Biodegradable and Biocompatible Polymer Composites; Elsevier: Amsterdam, The Netherlands* (2018): 55-79.
- [63] Narain, Ravin, ed. *Polymer Science and Nanotechnology: Fundamentals and Applications*.
- [64] Raval, Nidhi, Rahul Maheshwari, Dnyaneshwar Kalyane, Susanne R. Youngren-Ortiz, Mahavir B. Chougule, and Rakesh K. Tekade. "Importance of Physicochemical Characterization of Nanoparticles in

Pharmaceutical Product Development." In *Basic Fundamentals Of Drug Delivery*, pp. 369-400. Academic Press, 2019.

- [65] Devaraj, Preetha, Prachi Kumari, Chirom Aarti, and Arun Renganathan. "Synthesis and characterization of silver nanoparticles using cannonball leaves and their cytotoxic activity against MCF-7 cell line." *Journal of nanotechnology* 2013 (2013).
- [66] Pérez Mirabet, Leonardo. *Synthesis, characterization and functionalization of metal and metal oxide nanoparticles TEM microscopy study*. Universitat Autònoma de Barcelona, 2014.
- [67] Dar, Ghulam Nabi. "Metal oxide nanostructures and their applications." PhD diss., 2015.
- [68] Ealia, S. Anu Mary, and M. P. Saravanakumar. "A review on the classification, characterisation, synthesis of nanoparticles and their application." In *IOP Conference Series: Materials Science and Engineering*, vol. 263, no. 3, p. 032019. IOP Publishing, 2017.
- [69] Ana Luísa Pécurto Cartaxo; Nanoparticles types and properties – understanding these promising devices in the biomedical area 2015.
- [70] Vladár, András E., and Vasile-Dan Hodoroaba. "Characterization of nanoparticles by scanning electron microscopy." In *Characterization of Nanoparticles*, pp. 7-27.
- [71] Srihasam, Saiganesh, Krishnan Thyagarajan, Mallikarjuna Korivi, Veeranjanya Reddy Lebaka, and Siva Pratap Reddy Mallem. "Phytogenic generation of NiO nanoparticles using Stevia leaf extract and evaluation of their in-vitro antioxidant and antimicrobial properties." *Biomolecules* 10, no. 1 (2020): 89.
- [72] Yang, Chun-Chen. "Synthesis and characterization of active materials of Ni (OH) 2 powders." *International Journal of Hydrogen Energy* 27, no. 10 (2002): 1071-1081.

- [73] Kovalenko, Vadym, and Valerii Kotok. "The popcorn effect": obtaining of the highly active ultrafine nickel hydroxide by microwave treatment of wet precipitate." (2018): 12-20.
- [74] A.Sharma, Pallavi., S.Kumar., S.Dahiya , N.Budhiraja, Advances in Applied Science Research. 4(2013)124-130.
- [75] Saghatforoush, Lotf Ali, Mohammad Hasanzadeh, Soheila Sanati, and Robabeh Mehdizadeh. "Ni (OH) ₂ and NiO nanostructures: synthesis, characterization and electrochemical performance." *Bulletin of the Korean Chemical Society* 33, no. 8 (2012): 2613-2618.
- [76] Sun S, Zhang Y-Chao, Shen G, Wang Y, Liu X, Duan Z, Pan L, Zhang X, Zou J-Jun, Photoinduced Composite of Pt decorated Ni(OH)₂ as Strongly Synergetic Cocatalyst to Boost H₂O Activation for Photocatalytic Overall Water Splitting, *Applied Catalysis B: Environmental* 243 (2019) 253-261.
- [77] Kumar DR, Ranjith KS, Nivedita LR, Asokan K, Rajendra Kumar RT Swift heavy ion induced effects on structural, optical and photo-catalytic properties of Ag irradiated vertically aligned ZnO nanorod arrays. *Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atoms* 450 (2019) 95–99.