

“RAPID DEGRADATION OF ROSE BENGAL DYE USING SILVER DOPED NICKEL HYDROXIDE NANOPARTICLE”

**Submitted in partial fulfillment of the requirements for the award of
Master of Science in CHEMISTRY**

by

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DEPARTMENT OF CHEMISTRY

SATHYABAMA

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

Accredited with Grade “A” by NAAC

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DEPARTMENT OF CHEMISTRY **BONAFIDE CERTIFICATE**

This is to certify that this Project Report is the bonafide work of **Ms. T. NANDHAKUMARI (39910014)** who carried out the project entitled **“RAPID DEGRADATION ROSE BANGEL DYE USING SILVER DOPED NICKEL HYDROXIDE NANOPARTICLE”** under my / our supervision.

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DECLARATION

I **Ms. T.NANDHAKUMARI** student of **SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY** hereby declare that the Project Report entitled “ **RAPID DEGRADATION OF ROSE BENGAL DYE USING SILVER DOPED NICKEL HYDROXIDE NANOPARTICLE**” done by me under the guidance of **Dr. S.SUNITHA M.Phil., PhD.,** and **Dr.G. MURUGADOSS M.Sc., Ph.D. SCIENTIST-E, IRC,** at **INTERNATIONAL RESEARCH CENTER, SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY, Chennai- 600119** is submitted in partial fulfillment of the requirements for the award of **Master of Science Degree in CHEMISTRY.**

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ABSTRACT

The aim of this study was to make nickel hydroxide nanoparticles by using different synthesis and characterization. NiO nanoparticles is very stable interesting optical – electronic properties. Nickel oxide is cubic crystal structure and p-type semiconductor. The optical properties and electronic properties, the NiO was modified with metal and metal oxide. This method is doping simple route to modification of the NiO properties. Nanomaterials and nanoparticles that are different in terms of composition, particle size, and structure and synthesis and characterization of metal/metal oxides nanoparticles in solution.

Nickel oxide nanoparticles were synthesized by a facile and cost. Effective chemical precipitation method. The influences size, morphology and structural constitution of the final product. NiO nanoparticles with average particles size of 25nm. The band gap of the bulk NiO is between 3.6-3.8 eV. The facile co-precipitation synthesis of pure silver doped nickel hydroxide nanoparticles

This thesis synthesis, characterizations, and applications of undoped and doped metal oxide nanostructures are this study like Pure NiO, and Ag doped Ni(OH)₂ were synthesis by co-precipitation method. The thesis focuses on the creation of novel synthesis techniques/procedure as that are quick, efficient, and cost-effective. The thesis also includes the use of as-synthesised nanomaterials and nanoparticles in a number of significant applications.

The thesis is divided into of the several sections that include a brief overview of nanoparticles and metal oxides nanoparticles, as well as their various synthesis method. The morphological, structural, optical, compositional, and electrical properties of the synthesized nanomaterials were investigated using a variety of techniques. Furthermore, then prepared nanoparticles were used in a variety of applications, photocatalyst. Characterization and synthesis techniques used in specific techniques., i.e. sol-gel techniques, co-precipitations method, solvothermal method and hydrothermal methods. The synthesis of various undoped and doped nanoparticles explored in thesis.

Synthesis and characterization and nanoparticles application metal oxides via facile and simple hydrothermal process using commonly used laboratory chemicals. In this work as Nickel hydroxide nanoparticles (Ni(OH)_2 , NPs) have been synthesized cost effective and convenient co-precipitation method using Rose Bengal (RB) as capping agents and NaOH as a reducing agent. A variety of spectroscopic and microscopic techniques were used to examine the as-synthesized NPs, including X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Fourier transform infrared spectroscopy (FTIR). The NiO NPs prepared at low temperature (160°C) were shown excellent adsorption and photocatalytic toward the textile dyes.

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CHAPTER – 1

INTRODUCTION

1.1 NANOPARTICLES

A nanoparticles or ultrafine particles is usually defined as a particle of matter that is between 1 and 100 nanometers (nm) in diameter [1-2]. The properties of a material in nanoparticles form are usually very different from those of the bulk material even when divided into micrometer-size particles [3]. According to physics, a particle is a small localized object which can be described by their physical properties, such as volume or mass. There are several methods for creating nanoparticles, including gas condensation, attrition, chemical precipitation, ion implantation, pyrolysis and hydrothermal synthesis. For some application, nanoparticles may be characterized in complex matrices such as water, solid, food, polymers, inks, complex mixtures of organic liquids such as in cosmetics, or blood [4-5].

Nanostructures are two main difference involving all the different studies and application of nanostructures. Nanoscience is a study of all the kind of nanostructures involving all the different properties and this process Nanotechnology is a search of different applications of the studied nanoparticles by trying to control and manipulate [6]. Semiconductor nanomaterials is of the richest class of nanomaterials. A semiconductor is a material with electrical conductivity due to electron flow intermediate insulator and conductor. Semiconductor as governed a signification role of progressing research in nanoscience and nanotechnology [7]. The diameter of the prepared particles is in the range of nanometer, then the particles is called as nanoparticles. The nanoparticles properties are two type of chemical properties and physical properties. The properties are different from those for the bulk materials [8].

These nanoparticles can be metallic prepared using different techniques such as chemical synthesis - salt reduction, solvothermal synthesis electrochemical synthesis etc. These nanoparticles can be metallic prepared using different techniques such as physical synthesis – evaporation method etc. [9] Figure 1.1. Recently it is observed that the noble or inert metal like is silver (Ag) can be used in cancer treatments [10]. Characterization and Preparation of silver nanoparticles using co-precipitation metal.

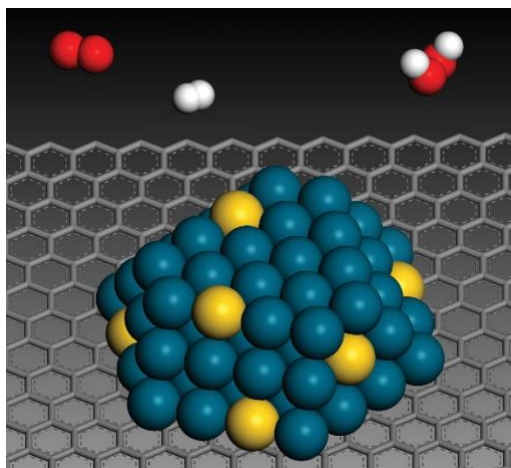


Figure 1.1 the classification of nanoparticles

1.2 METAL OXIDE NANOPARTICLE

Metal oxides play a very important role in many areas of chemistry, physics and materials science [11]. Metal oxide are crystalline solids a metal cation and an oxide anion. They typically react with water to form bases or bases or with acids to form salts. The transition metal and their compound are used in catalysts is chemical industry and in battery industry. Metals of s-block elements are used for industrial applications [12]. Metal oxide, in particles provide a fundamental stepping – stone for the development of functional nanomaterial. In an oxidative environment, such as the atmosphere of the Earth, oxides are the lowest free energy states for most metals in the periodic tables and demonstrate application ranging from semiconductor to insulator [13]. Thus this thesis is planned to

study in particular Ni(OH)_2 , doped Silver nitrate are metal oxide nanostructures. Here the synthesis of metal oxide of nanomaterials is through two routes, hydrothermal as well as thermal evaporation [14]. The only two metal oxides we study here in this work are pure and doped Ni(OH)_2 and an oxide nanomaterials.

1.3 SYNTHESIS TECHNIQUES OF METAL OXIDE NANOMATERIAL

Synthesis of nanoparticles with defined structures and size is the most challenging task in the field of nanoscience and nanotechnology. The study of the nanoparticulated oxides is the synthesis of the materials [15]. The development of systematic studies for the synthesis of oxide nanoparticles is a current challenge and essentially, the corresponding preparation method may be grouped in two main streams based upon the liquid- solid and gas-solid nature [16]. Two strategies are generally prepared nanostructures: the top-down approach using physical method such as photolithography and related techniques and the bottom-up employing physical and chemical approach.

- a) Top- down: The kind of synthesis procedures involve the breaking down of large material pieces in order to generate the desired smaller resultant nanoparticles from them.
- b) Bottom-up: Implies assembling single atoms and/or molecules so as to build large nanostructures from them [17-19].

Synthesis method of the three categories: solution based, vapor state and biological methods. Such as division is based on the type of the medium in which the oxidation reaction takes place [20].

1.3.1. SOL-GEL TECHNIQUE

The process involves conversion of monomers into a colloidal solution is called (sol). Then acts as the precursor for an integrated network (or gel) of either discrete particles or network [21]. The nanoparticle is synthesis a variety of metal oxide such as nanoparticles like TiO_2 , CuO , ZnO etc. this method is used as fabrication of metal oxide [22]. The typical synthesis route of the sol- gel synthesis is the hydrolysis of metal oxide

or various inorganic precursors such as metal chlorides, nitrates, and sulfides, where the important obstacle to overcome is the control of the hydrolysis reaction occurring in solution Figure 1.2. Proper control of hydrolysis should result in no unwanted solid particles precipitating out of solution. The reaction is commonly stabilized by use of an acid (or base, depending on the reaction) to slow the reaction rate, and allow formation of the gel [23].

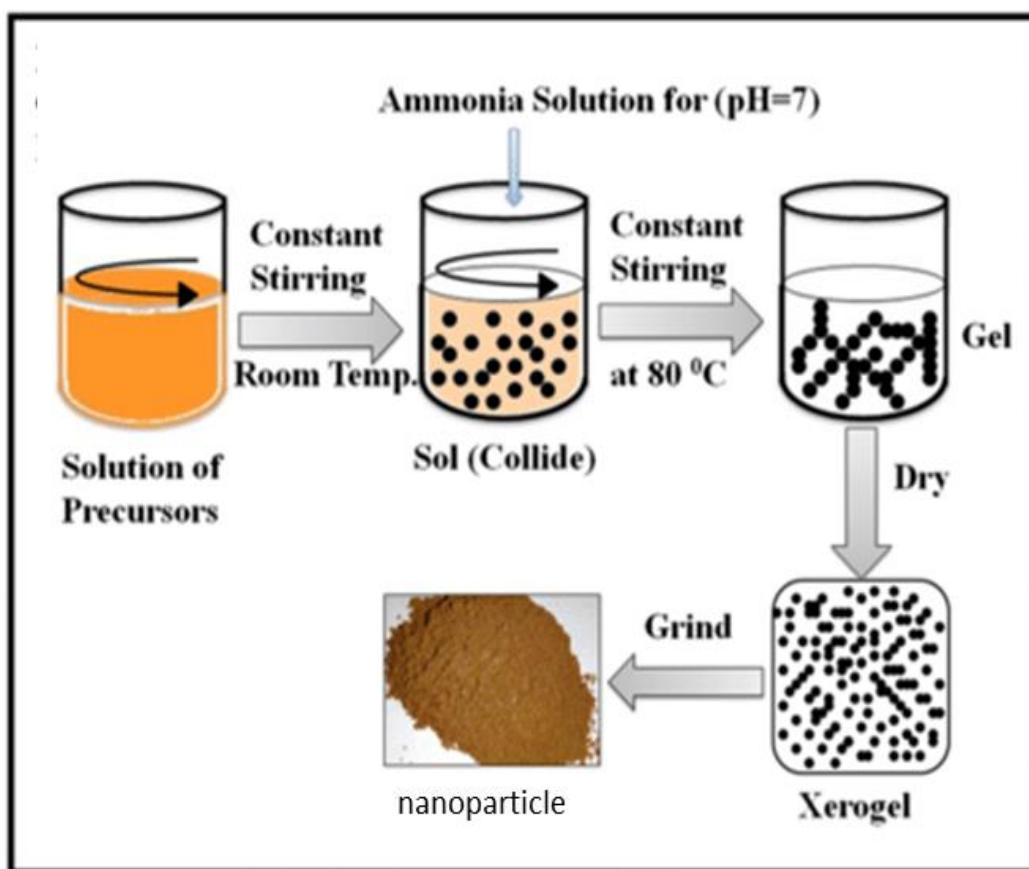


Figure 1.3.1 Synthesis method of sol-gel technique

1.3.2. Co- PRECIPITATION METHOD

Co-Precipitation method is a standard synthesis technique to prepare a pure metal oxide nanoparticle. This involves dissolving a salt precursor chloride, nitrate, etc., in water or other solvent to precipitate the oxo-hydroxide from with the help of a base. Very often, control of size and chemical homogeneity in the case of mixed – metal are difficult to achieve. Use of surfactants, chemical methods, and high-gravity reactive precipitation appear as novel and viable alternative to optimize the resulting solid morphological characteristics [24]. nanoparticles are control the precipitate we get was washed several times with water and acetone and dried at 160 C. Figure 1.3.2

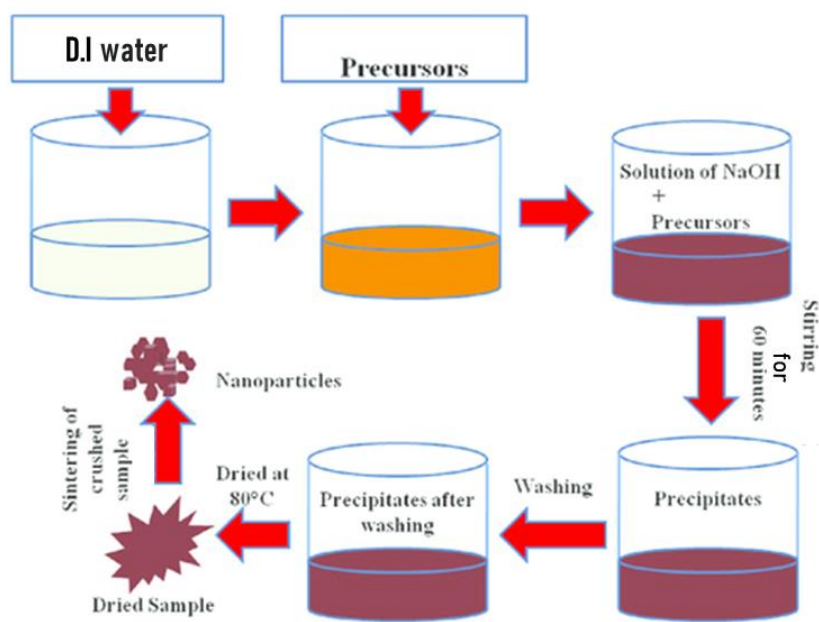


Figure 1.3.2 process of co-precipitation method

1.3.3. HYDROTHERMAL SYNTHESIS

Hydrothermal synthesis can be defined as method of synthesis of single crystals that depends on the solubility of minerals in hot water under high pressure. The crystal growth is performed in an apparatus consisting of a steel pressure vessel is called autoclave [25]. And it is well 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. Advantage of the hydrothermal synthesis method include the ability to synthesis crystal of substance which are unstable near the melting point, and the ability to synthesize large crystals of high quality. The solubility of many oxide in hydrothermal solution of salts is much higher than in pure water, such as salts are called mineralizers. Disadvantages are high cost of equipment and the inability to monitor crystals in the process of their growth [26]. Figure 1.3.3

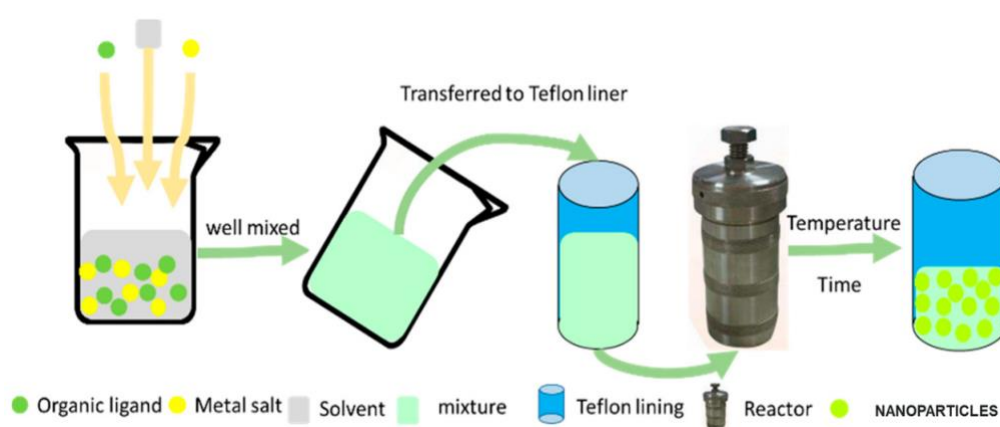


Figure 1.3.3. Hydrothermal method

1.3.4. SOLVOTHERMAL SYNTHESIS

The solvothermal synthesis route is very similar to the hydrothermal synthesis route in that it involves a Teflon lined stained steel autoclave that is sealed and heated to elevated temperature. This gives the synthesis the advantages of the sol-gel synthesis method as different precursors, such as metal oxide, can be used that would normally not be miscible or would normally decompose in an aqueous solution. Hydrothermal synthesis is used for the preparation of hydroxides and oxides due to the chemical composition of water and various aqueous solution as the solvent. Solvothermal synthesis, on the other hand, allow for the development of non-oxides materials such as nitrides and chalcogenides although can certainly be used for synthesis of metal oxides as well [27]. Figure 1.3.4. The temperatures are typically above the boiling temperature of the solvents used; thus, elevated temperatures are achieved inside the autoclave.

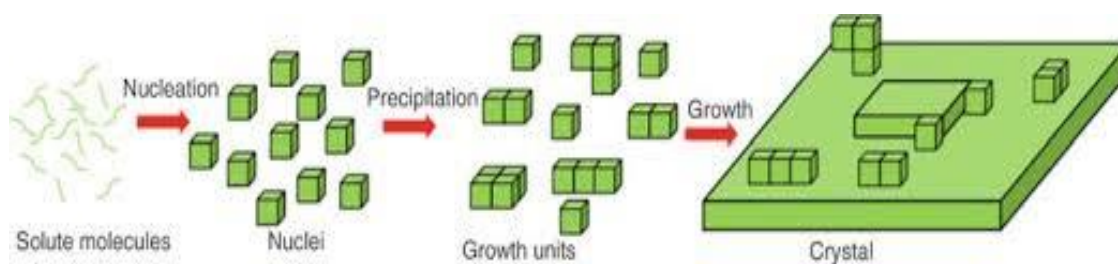


Figure 1.3.4. solvothermal method

1.4. APPLICATION OF METAL OXIDE

Metal oxide nanoparticles have a variety of application such as catalyst etc. metal oxide semiconductors are suitable photocatalysts due to their high photosensitivity, stability, low cost and suitable band gap [28]. 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 [29]. The metal oxide of greatly disturbed, because of the particles size decreases, the increasing number of surface atom that generate stress/strain. This method can be 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. Some commonly used types and applications of metal oxide nanoparticles [30].

1.5 TARGETED OF METAL OXIDE

1.5.1. NiO

Nickel oxide NiO is an important transition metal oxide with cubic lattice structures [31]. Nickel oxide nanoparticles can be manufactured by thermal decomposition of freshly prepared nickel hydroxide by chemical precipitation route at 450°C. The nickel oxide nanoparticles created using this method can be characterized using X-ray diffractometer and vibrating sample magnetometer. Nickel oxide is an important role of transition metal oxide with a cubic lattice structure. The crystal structure of Nickel oxide. The average band gap of NiO is 3.6-4 electron volt [32]. 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 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 [33]. There are many different methods reported for the synthesis of NiO nanoparticles, such as ultrasonic radiation, hydrothermal synthesis, carbonyl method, laser chemical method, pyrolysis by microwave, sol-gel method, Co-precipitation method decomposition method [34].

1.5.2. PROPERTIES OF NiO

The properties of oxide nanoparticles influenced the size, surface effects, crystal lattice defects.

Table 1.5.2 Chemical and physical and thermal properties

Chemical Properties:

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

Physical properties:

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

Properties	Metric	Imperial
Density	6.67 g/mol	0.240lb/in3
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. APPLICATION OF NiO:

The key applications of nickel oxide nanoparticles are as follows:

- In electrochromic coatings, plastics and textile.
- Nickel oxide particles are widely used in batteries, like lithium-ion batteries, Nickel-Zinc battery etc.,
- Nickel oxide is a significant material for, application in preparation of nickel cermet for the anode layer of solid oxide fuel cells (Thomas et al., 2013).
- In active optical filters.
- In ceramic structures.
- In cathode materials for alkaline batteries.
- P-type transparent conductive films.
- Materials for gas or temperature sensors, such as CO sensor, H₂ sensor, and formaldehyde sensors.
- As a catalyst and as antiferromagnetic layers.

1.5.4. Ni(OH)₂ NANOPARTICLES

Nanoparticles are white spherical high surface area metal particles. Nanoscale Nickel Hydroxide particles are typically 10-30 nanometers (nm) with specific surface area range (SSA) in the 130-150 m²/g. Nickel Hydroxide is known as an active material for cathodes in nickel based rechargeable alkaline batteries [35]. Nickel oxide nanoparticles has certain advantage and application 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 application [36-37]. Metal oxide nanostructures have been synthesized such as Zinc oxide (ZnO), Copper oxide (CuO), Nickel oxide (NiO) [38-39]. In this project work, we have planned to study in particles NiO and at low temperature formation of Ni(OH)₂ with Ag ion. Here in this project, we will investigate the synthesis of the above-mentioned metal oxide nanostructures through co-precipitation method. The metal oxide we study here in this work is Ag doped NiO. The prepared samples size, morphology, structure was characterized under X-Ray Diffraction [XRD], Scanning Electron Microscope [SEM]., UV-visible spectroscopy. Nickel hydroxide powders were prepared using chemical precipitation, in which sodium hydroxide (NaOH) was added into nickel chloride hexahydrate Ni(OH)₂ [40].

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 (OH[•]) 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 oxide on irradiation with appropriate wave length leads to excitation of an electron from valence band to conducting band yielding an electron hole pair [41]. The structural, morphological, optical, adsorption, and photocatalytic properties of NiO and Ni(OH)₂ NPs with respect to temperature has been discussed.

1.7 OBJECTIVE

- Investigation on photocatalytic performances of Ag doped Ni(OH)₂ nanoparticles synthesis by a facile co-precipitation method.
- Influences of Silver ion on the size, morphology, and structural constitution will be studied.¹
- Effect of the photocatalytic activity Rose Bangle dye was performed using undoped and Ag doped NiO nanoparticles were investigated.
- Synthesis of low temperature- based Ag doped Ni(OH)₂ nanoparticles.
- Studies on size, morphology and structure using X-Ray Diffraction [XRD], Scanning Electron Microscope [SEM], UV-spectroscopy and Fourier transform infrared spectroscopy [FTIR].
- Herein, the effects of the photocatalytic activity Rose Bangle dye were performed using undoped and Ag doped NiO nanoparticles.

CHAPTER 2

LITERATURE SURVEY

Lippmann, Morton, Mark Frampton, Joel Schwartz, Douglas Dockery, Richard Schlesinger. "The US Environmental Protection Agency Particulate Matter Health Effects Research Centers Program: a midcourse report of status, progress, and plans." *Environmental Health Perspectives*. Based on his research paper entitled as preparation and characterization of nanoparticles. M.Mnzur, Electrochemistry Communications application.

V. Mohanraj, Y. Chen, Tropical Journal of Pharmaceutical Research and research paper is used, Yan-Na, Saiqa. "Role of Nanomaterials and their Application as Photo-catalyst and this research paper is XRD method are used in preparation.

Dar GN. Metal oxide nanostructures and their application this metal oxide is application and characterization of nanomaterials.

Nanoparticle through a Modified Sol-Gel Method with Application in Energy Storage. Anu Mary Ealias and M.P Saravanakumar A review on the classification, characterisation, synthesis of nanoparticles and their application Materials Science and Engineering and used in the sol- gel method of the application and it the catalyst.

CHAPTER 3

AIM AND SCOPE OF THE PRESENT INVESTIGATION

The present work has been planned as an effort to develop a metal oxides and nanoparticles and nanostructure. NiO nanoparticles is a very stable and interesting optical electronic properties. The band gap of the bulk NiO is between 3.6-3.8 eV.

NiO is p-type semiconductor and cubic crystal structure. To enhance the optical properties and electronic properties, the NiO was modified with metal and metal oxides. The method of doping is samples route to modification of the NiO properties. The effects of the photocatalytic activity rose bengal dye was performed using undoped and Ag doped NiO particles.

To the crystal structure surface morphology, organic functional groups on the surface of the nanoparticles. Nanoparticles and optical properties synthesis and characterization of using silver doped nickel hydroxides using X-Ray Diffraction [XRD], Scanning electron microscopy [SEM], and Fourier transform infrared [FTIR] spectroscopy, and photocatalyst. Silver influences the size, morphology and structural constitution of the final product.

CHAPTER 4

EXPERIMENTAL AND CHARACTERIZATION TECHNIQUES

4.1 INTRODUCTION

The metal oxides nanomaterials in this project are 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 a low temperatures 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 synthesis nanomaterials were used for photocatalytic applications.

4.2 MATERIALS

All chemical used in this project were analytical grade and used without any further purification. Nickel (II) chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) (MERCK). Molecular weight of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ is 237.69 g/mol, Silver Nitrate (AgNO_3), (SIGMA-ALDRICH), Molecular weight of AgNO_3 is 169.87 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-ionized water.

4.3 EXPERIMENTAL PROCEDURE

Pure NiO, and Ag doped Ni(OH)₂ were synthesis 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 Figure 2.3.1. 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 calcinations of the prepared samples at 450°C.

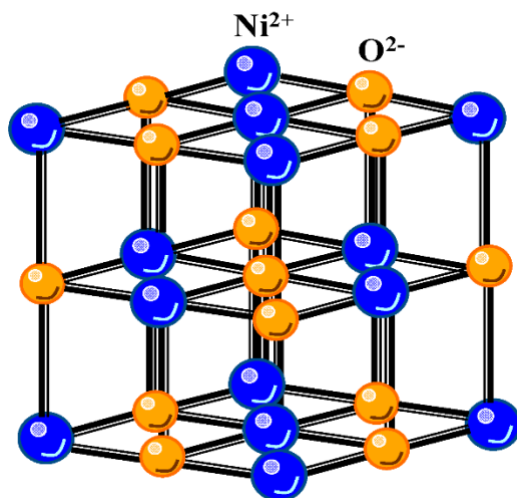


Figure.4.3.1. NiO

4.3.2. SYNTHESIS OF Ni(OH)₂

Ag 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 Silver nitrate and to reduce agglomeration add 1g of polyvinyl pyrrolidone (PVP). The pH as 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°. Image with 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 (XRD)

X-Ray Diffraction is an incredibly useful technique for determining the crystalline structure of the materials [42]. A crystal lattice is a regular three- dimensional distribution of atom in space. X-Ray Diffraction technique have played crucial role in the identification and evaluation of crystalline perfection if crystals. It is one of the oldest and non-destructive methods of analysis of samples and is still valid for all branches of solid-state analysis [43]. Diffraction pattern is obtained only when the X-rays are interacted with the crystal. Information like symmetry of the unit cell, stress, strain, size of crystallite and growth orientation etc. are obtained from the diffraction pattern [44]. XRD is used for assessment of phase purity and crystallinity of inorganic materials. When a material is irradiated with a monochromatic light of wavelength around the inter atomic distance of

the material, light gets dispersed by the atoms and interfere to give rise to diffracted light with a higher intensity Figure 2.4.1. The arrangement of atoms in the material is related to the intensity of the scattered light. If wavelength of light is λ and the incident angle is θ and the constructive interference is obtained at an angle 2θ from the incident beam of light if Bragg's equation is satisfied.

Bragg's equation is given by, $n\lambda = 2d \sin \theta$

where, n is the diffraction order, d is the distance between the diffracting planes and θ is the Bragg's angle. X-Ray Diffraction is based on Bragg's law [45].

$$D = k\lambda / \cos \theta$$

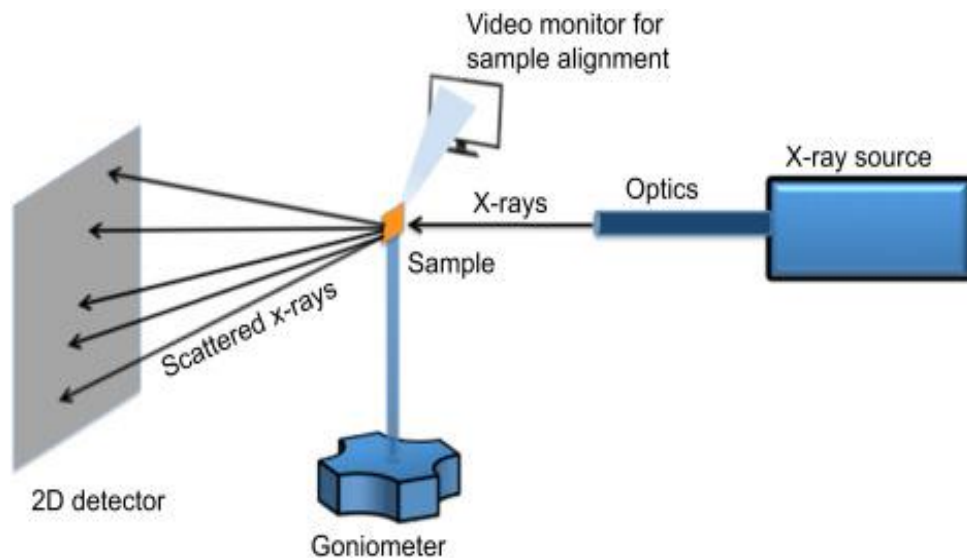


Figure 4.4.1 Working of X-Ray Diffraction

4.4.2 SCANNING ELECTRON MICROSCOPE (SEM)

Scanning electron microscope (SEM) is one of the most important tools in order to study the overall appearance of the grown nanostructures. It helps us in the analyzing

different parameter like quality, shape, density, diameter, thickness, length and orientation of the as grown nanostructures [46]. A scanning electron microscope (SEM) is a type of electron microscopes that image a sample by scanning it's with a high- energy beam of electron in a raster scan pattern. The electron interacts with the atom that make up the sample producing signals that contain information about the samples surface topography, composition, and other properties. Figure 2.4.2. Scanning electron microscope (SEM) produces the image scanning the sample with high energy beam of the electron. As the electron interact with the electron produce the secondary electron characteristic X- ray. The light microscope uses visible light for imaging. In a similar way electron microscope uses electron for imaging.

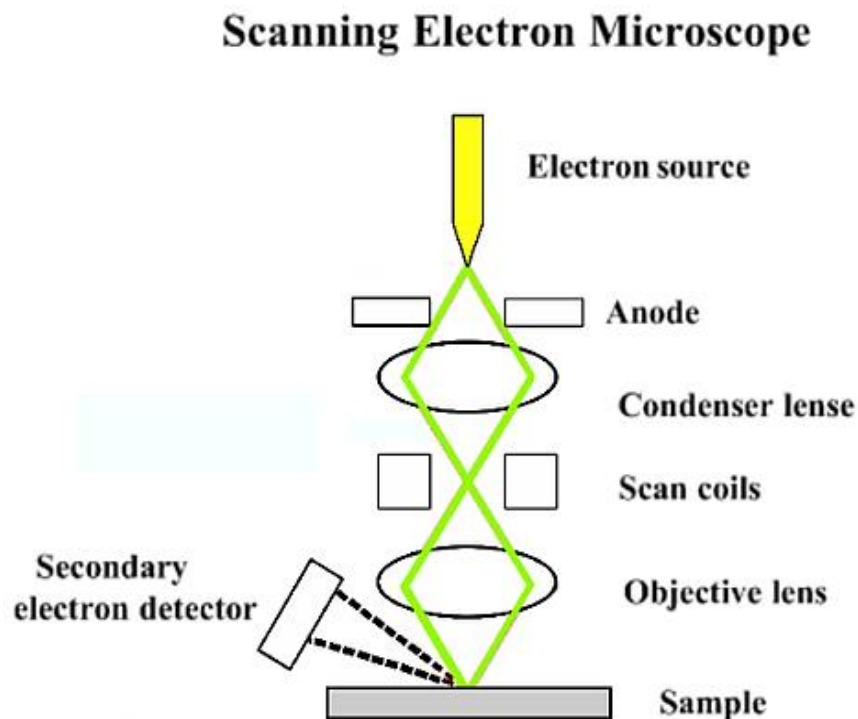


Figure 4.4.2 Scanning electron microscopy

4.4.2.1 WORKING OF SCANNING ELECTRON MICROSCOPE

In scanning electron microscopy source is used to produce electrons. It produces at the top of the column. When the thermal energy of electrons overcomes the work function of source material, the electron is being emitted. Then the electrons are accelerated and attracted by positively charged anode. The electron column was under vacuum and it was placed in a special chamber to protect it against contamination, vibrations or noise. So that images with high resolutions were obtained. If it is contaminated, it causes the electron beam to deflect and reduce the image quality. High vacuum condition increases the collection efficiency of electron by the detectors that are in the column. Condenser lens are used to control the path of electrons. A scanning coil is used to raster the beam on to the samples. Two types of electron were used to scanning electron microscopy. They are Backscattered electron (BSE) and the secondary electrons. For the detection of BSE, solid state detectors are used and for the detection of secondary electron. Everhart-Thomley detector is mainly used. It has a scintillator which is used to accelerate electrons. Secondary electrons are used to form three-dimensional image of samples.

4.4.3. FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

The instrument that determines the absorption spectrum for a compound is called a spectrophotometer. Fourier transform spectrophotometer provides the IR spectrum much more rapidly compared to the traditional spectrophotometer [47]. FTIR spectroscopy technique and provides specific important structural and functional information obtained from the analysis. The vibrational excitation of molecular bonds by absorption of infrared light energy is the basis of FTIR spectroscopy (only the middle infrared section). The vibrational excitation of molecular bonds by absorption of infrared light energy is the basis of FTIR spectroscopy (only the middle infrared section). FTIR Analysis is used to analyze organic materials. Bulk and small particle materials can be analyzed [48-49]. FTIR microscopy allows for the identification of particle as small as 10 μm Figure 2.4.3



Figure 4.4.3 Fourier transform infrared spectroscopy

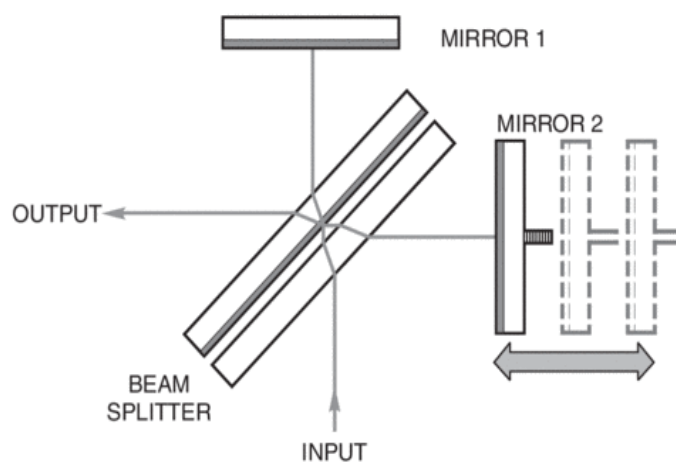


Figure 4.4.3 working of Fourier transform infrared spectroscopy.

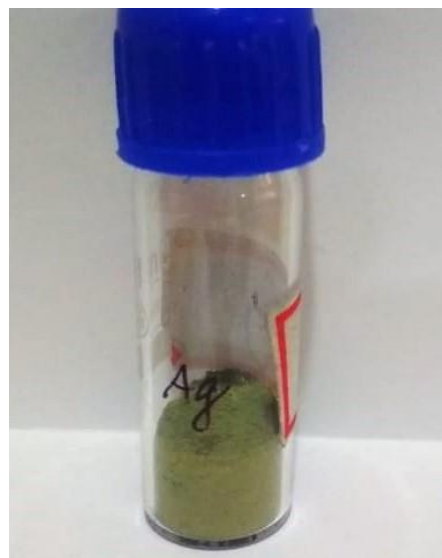
CHAPTER 5

5. RESULTS AND DISCUSSION

The following instruments were used to characterize the synthesised nanoparticles crystal structure, morphology, compositional information, organic functional groups on the surface, and photocatalytic application. X-ray Diffraction (XRD), scanning electron microscopy (SEM), Fourier Transform Infrared spectroscopy (FTIR), and UV-vis spectroscopy are some of the techniques used. These approaches that have been highlighted exits Figure 5.



Figure 5. (A) NiO ,



(B) Ag/Ni(OH)₂

5.1 X-RAY DIFFRACTION (XRD)

The XRD approach is used to determine whether a material is crystalline or amorphous. When monochromatic light with a wavelength around the material's inter atomic distance is irradiated, light is scattered by the atoms and interferes, resulting in diffracted light with a greater intensity. The intensity of dispersed light is proportional to the arrangement of atoms in the material. If wavelength of light is λ and the incident angle is θ and the constructive interference is obtained at an angle 2θ from the incident beam of light if Bragg's equation is satisfied.

Bragg's equation is given by,

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

where, n is the diffraction order, d is the distance between the diffracting planes and θ is the Bragg's angle.

Generally, in XRD Cu- $K\alpha$ is the incident light for which the $\lambda = 1.5405$ nm. Distance between the diffracting planes is calculated according to Bragg's equation [50]. For phase identification of a wide variety of crystalline phases in concrete specimens, the standard database (JCPDS database) for XRD pattern is used [51]. The use X-Ray light is important since it has a short enough wavelength to interact with and diffract off of crystalline lattices of similar size. The electromagnetic force interacts with an atom's electron cloud as X-Ray light passes through it. The structure of prepared silver nanoparticles has been investigated by XRD study.

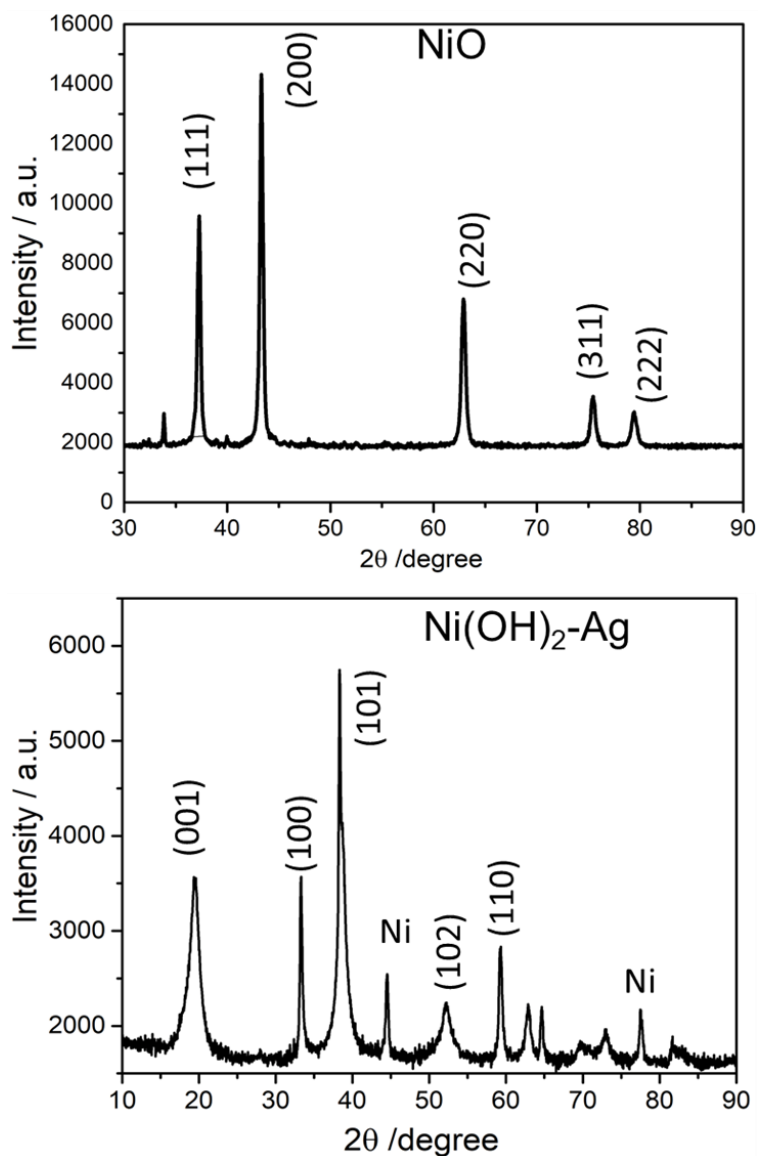


Figure 5.1 XRD of NiO , XRD of Ag doped Ni(OH)₂

NiO spheres exhibits well defined diffraction reflection at 19.03°, 34.45°, 39.85°, 53.67°, and 59.58°. The bare Ni substrate show peak at 44° and 77°. The diffraction peaks for NiO are shown in planes of (111), (200), (220), (311) and (222) are shown in Fig.3.1 XRD of NiO. All diffraction peaks show a cubic crystalline structure of pure NiO. The standard spectral data of (JCPDS, No. 04-0835). The reflection peaks effect of instrumental

broadening is calibrated which were computed using measured values for the spacing of the (111) plane.

Ag doped Ni(OH)₂ peak at (001), (100), (101), (102), and (110) planes. The crystallite structure size of the Silver doped Nickel oxide nanoparticles found as sing Debye-Scherrer's formula,

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

Where k is the empirical constant which is equal to 0.9, λ is the wavelength of X-ray radiation (1.5405 Å for Cu-K α), β is the full width at half maximum of the diffraction peak and θ is the peak position [52].

5.2 SCANNING ELECTRON MICROSCOPE (SEM)

The high-resolution scanning electron microscope (SEM) is excellent for nanoparticle size and shape characterization, because the necessary sample preparation and image acquisition are relatively quick and simple. Despite the fact that the SEM image is a two-dimensional (2D) representation of the three-dimensional (3D) objects from a specific viewing angle, it does contain some 3D information [53]. The scanning electron microscope works similarly to an optical microscope, except it detects electrons dispersed from the sample rather than photons. Because electrons can be accelerated by an electric potential, their wavelength can be made shorter than that of photons. As a result, images can be magnified up to 200.000 times by the SEM [54]. Scanning electron microscope (SEM) is a type of electron microscope used to investigate the surfaces of solid objects directly. It uses a beam of centred electrons of relatively low energy as an electron probe that is scanned over the specimen in a normal pattern [55].

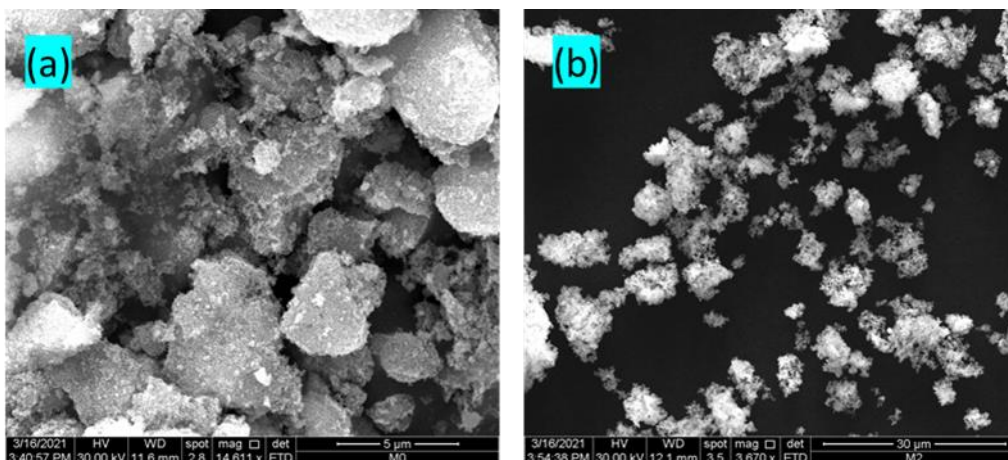


Figure 5.2(a) Scanning electron microscopy (SEM) image of pure NiO nanoparticles and (b) SEM image of Ag doped Ni(OH)₂ nanoparticles. The SEM images were measured for powder samples for the both samples (NiO and Ag doped Ni(OH)₂). The powder samples were pasted on the carbon tape for measuring the SEM images.

Nickel oxide shows high surface energy than the silver doped nickel hydroxide because it is agglomerated.

Compared to the pure nickel oxide silver doped nickel hydroxide shows finite structures. Results shows that the synthesized NiO nanoparticles are smooth, spherical in shape and some are agglomerated as they have high surface area to volume ratio and has less cohesive energy compared to the bulk materials. SEM photographs of silver doped nickel hydroxide are spherical. Silver doped nickel hydroxide shows small size compared to the pure nickel oxide due to its high dispersion [56].

When calcinated to higher temperature of about 160°C nickel oxide can be de-agglomerated. The size according to the SEM images of silver doped nickel hydroxide maybe below 10nm.

5.3 FOURIER TRANSFORM INFRA-RED SPECTROSCOPY

Fourier transform infrared spectroscopy Nickel oxide and silver doped Nickel hydroxide. The formation of nanoparticles can be observed from colour change. The vibration of ions is the focus of FTIR. Each functional group has its own discrete vibrational energy that can be used to determine a molecule when all of the functional groups are combined. This makes IR spectroscopy perfect for identifying samples, characterization of multilayer films, and particle analysis. More information on the fundamentals of FTIR Spectroscopy can be found here. The formation of nanoparticles can be observed from this colour changed.

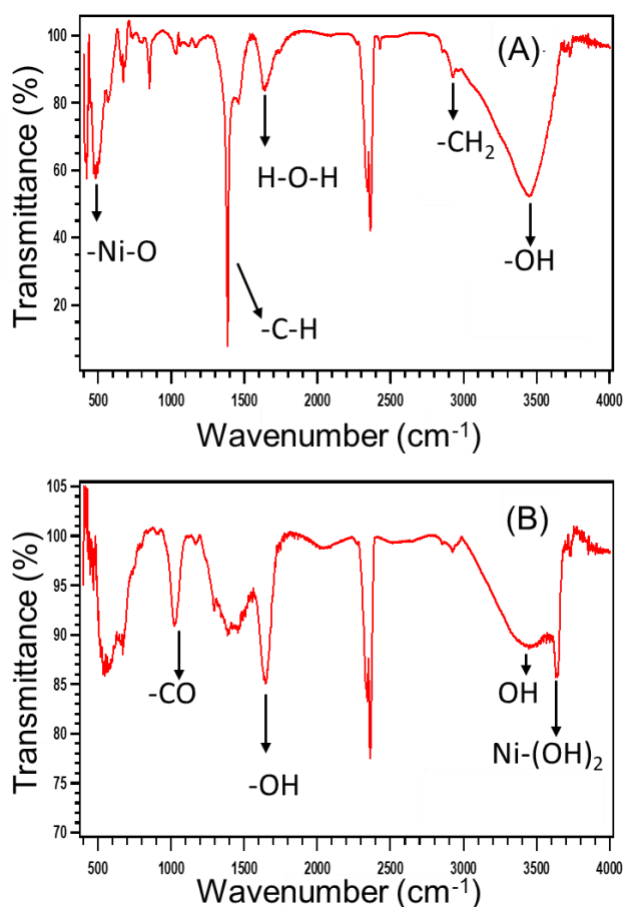


Figure 5.3 (A) FT-IR peak of NiO, (B) FT-IR peak of Ag/Ni(OH)₂

The vibrational peak at 3475 cm⁻¹ is due to the O-H stretch and the peak around 588 cm⁻¹ is described as the Ni-O stretching vibration.

The broad absorption band in the region 500-600 cm⁻¹ is the Ni-O stretching vibration. The size of the samples used in this study is less than the bulk forms of NiO. The broad spectrum at 3430 cm⁻¹ is due to the O-H stretching vibrations and a weak band at 1685 cm⁻¹ is the H-O-H bending vibrations.

The broad band at 3458 cm⁻¹ is the stretching vibration of water molecules present in the nickel hydroxide nanoparticle. In the Figure 5.3 (B) shows the peaks of FT-IR of silver doped nickel hydroxide.

5.4. PHOTOCATALYTIC ACTIVITY TEST

Rose bengal (RB) 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 µm syringe filter. The degradation efficiency is determined by the degradation of organic dyes on UV-Vis spectrophotometer. The degradation efficiency is calculated by the formula,

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

Where C and C₀ 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 were used to determine the correct degradation rate of RB 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.

5.4.1. PHOTOCATALYTIC ACTIVITY

The photocatalytic degradation efficiency of as prepared catalysts of NiO, and Ag/Ni(OH)₂ nanoparticles were resolute by the deprivation of RB dye under irradiation of Sun light. When irradiating the RB dye solution, the concentration of the dye was noticeably reduced and it was confirmed by the significant color variation. For NiO catalyst, the catalyst influences the organic molecule to achieve the efficiency of about 51.89 % within 120 mins. The photocatalyst expriemnt was further extended to doped samples. The degradation reaction of Ag/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 strucuture results increased the degradation efficiency. Compared to NiO catalyst, Ag/Ni(OH)₂ catalysts reached high efficiency of degradation of 88.32%, respectively [57].

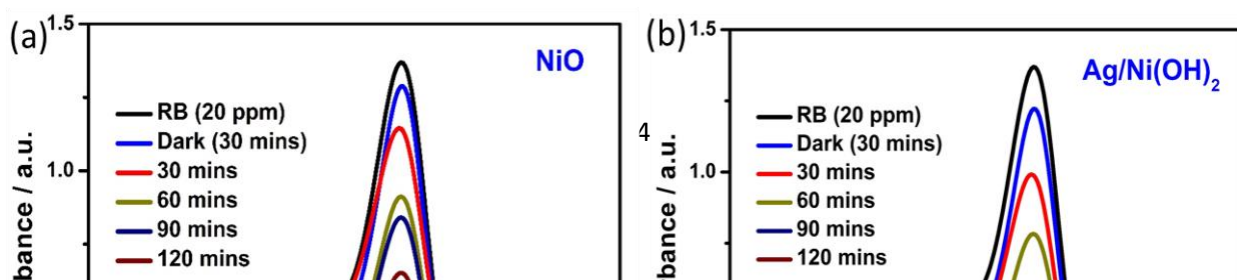


Figure.5.4.1(a-b) represents the UV-visible spectra for the RB with occurrence of catalysts NiO, and Ag/ Ni(OH)₂ were irradiated by sun light for 120 minutes. (c) C/C₀ versus time interval plot of rose bengal dye (d) $-\ln (C/C_0)$ versus time interval plot of RB dye

The significant improvement of the degradation efficiency is due to the doping effect of Ag ion at the Ni²⁺ lattice sites, it reduce recombination of excited charge carriers (electron-hole pair).

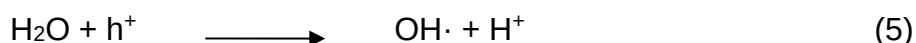
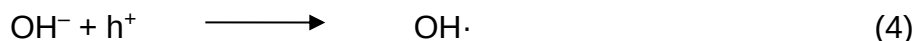
Figure.5.4.1c depicts the changes in initial and final concentration of dye in the presence and absence of photocatalyst against the light irradiation with respect to time (t) for the RB dye solution. The Ag/Ni(OH)₂ catalyst shows highest degradation ability due the fact that sub energy-level act as 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.5.4.1d the degradation rates were 0.013 min⁻¹ and 0.041 min⁻¹ for NiO, and Ag/ Ni(OH)₂ nanoparticles against RB dye, respectively. From the studies rate constant value and R² values are listed in the Table.1. The Figure 5.4.1.1 showed schematic diagram illustrating the mechanism of charge separation, generation of reactive oxygen species (ROS) and photocatalytic activity of photocatalyst under the sunlight irradiation (58).

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

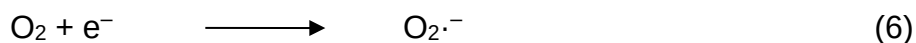
(i) Formation of electron hole pair



(ii) Formation of hydroxyl radicals



(iii) Formation of superoxide radicals



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



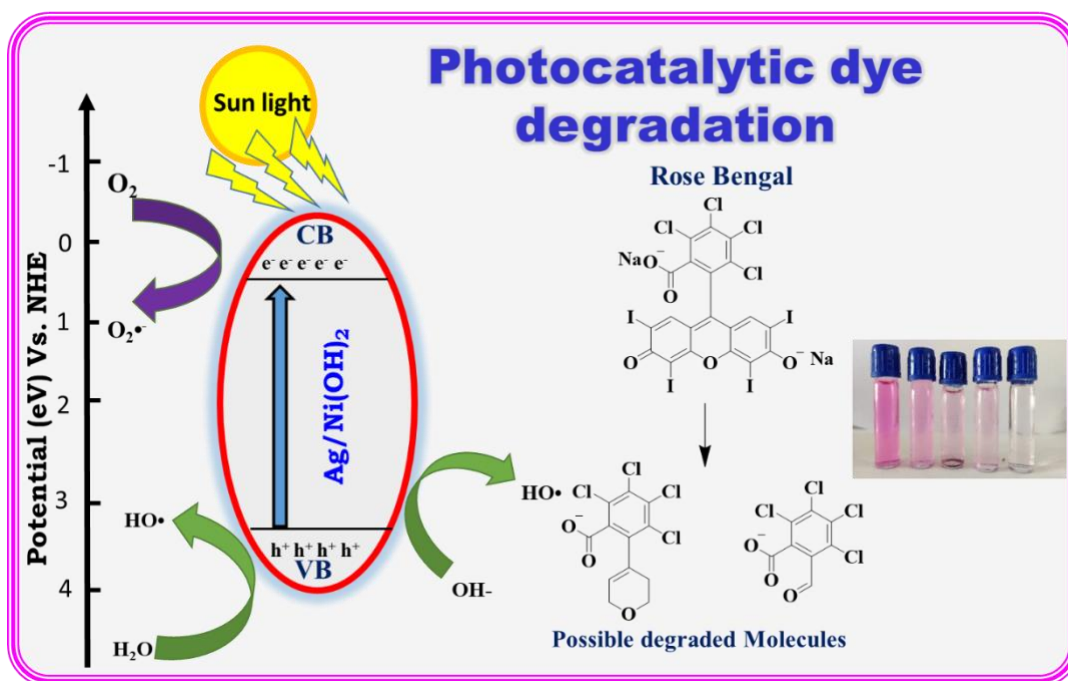


Figure. 5.4.1.1 Schematic diagram illustrating the mechanism of charge separation and photocatalytic activity of photocatalyst under the sunlight irradiation

Table 5.4.1.1 photocatalytic activity result of NiO, and Ag/ Ni(OH)₂ nanoparticles.

S.No	Catalyst	Band gap (eV)	Degradation efficiency (%) (120 minutes)	K (min ⁻¹)	R ²
Rose bengal (20 ppm)					
1	NiO	3.21	51.89	0.013	0.9809
2	Ag/Ni(OH) ₂	2.82	88.32	0.041	0.9248

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 Ag (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 7.6 nm for NiO and Ag 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 Ag doped Ni(OH)_2 showed as excellent dye degradation of Rose Bengal 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 Ag doped Ni(OH)_2 could be the suitable candidate for water purification, through the photocatalysis process.

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