

**“SYNTHESIS AND CHARACTERIZATION OF
PHOTODEGRADATION ACTIVITY OF LITHIUM DOPED NICKEL
HYDROXIDE NANO PARTICLES”**

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

by

B. SINTHU (39910018)



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. B. SINTHU (39910018)** who carried out the project entitled **"SYNTHESIS AND CHARACTERIZATION OF PHOTODEGRADATION ACTIVITY OF LITHIUM DOPED NICKEL HYDROXIDE NANOPARTICLE"** under my / our supervision.

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DECLARATION

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ABSTRACT

In the presence of sodium hydroxide, the lithium doped nickel hydroxide nanoparticles were produced using the Co-precipitation method with $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ and Lithium hydroxide as Ni and Li sources of precipitator, respectively. The effect of Li doping on the structural properties and sizes of nanostructures was investigated using XRD, SEM, and FTIR techniques. A stabilizing agent also added to reduce a particle agglomeration. As part of this project, we want to look into NiO and the formation of $\text{Ni}(\text{OH})_2$ with Li ions at low temperatures. Pollutants in industrial or domestic waste water effluents must be eliminated or destroyed before being released into the atmosphere, according to the photocatalyst. The influence of photocatalytic development Undoped NiO and Li doped Nickel hydroxide nanoparticles were used to examine the FAST GREEN dye. The photocatalytic degradation efficiency of NiO and $\text{Li}/\text{Ni}(\text{OH})_2$ nanoparticles was determined by removing the FAST GREEN (FG) dye from the catalysts before exposing them to sunlight. This degradation process by the free radical formation during the light irradiation finally through the water purification process of photocatalysis.

TABLE OF CONTENTS

1	INTRODUCTION	1
1.1	NANOMATERIALS	2
1.2	METAL OXIDE	3
1.3	SYNTHESIS TECHNIQUES OF METAL OXIDE NANOPARTICLES	3
1.3.1	SOL –GEL TECHNIQUE	4
1.3.2	CO-PRECIPITATION METHOD	5
1.3.3	HYDROTHERMAL SYNTHESIS	6
1.3.4	THERMAL EVAPORATION PROCESS	7
1.3.5	ELECTROCHEMICAL DEPOSITION PROCESS	8
1.4	IMPORTANCE OF METAL OXIDE	10
1.5	TARGETED METAL OXIDE	11
1.5.1	NIO	11
1.5.2	PROPERTIES OF NIO	12
1.5.3	KEY APPLICATION OF NIO	12
1.5.4	NI(OH) ₂ NANOPARTICLES	13
1.6	TARGETED APPLICATION OF NI(OH) ₂ AND NIO NANOPARTICLES PRESENTED IN THIS PROJECT	13
1.6.1	PHOTOCATALYST APPLICATION	13
1.7	OBJECTIVE	15
2	LITERATURE SURVEY	16
3	AIM AND SCOPE OF THE PRESENT INVESTIGATION	17
4	EXPERIMENTAL AND CHARACTERIZATION TECHNIQUES	18
4.1	AIM	18
4.1.1	MATERIALS REQUIRED	18
4.1.2	EXPERIMENTAL PROCEDURE	18

4.1.3	SYNTHESIS OF NIO	18
4.1.4	SYNTHESIS OF NI(OH) ₂	18
4.2	CHARACTERIZATION OF PREPARED METALOXIDE NANOMATERIALS	19
4.2.1	X-RAY DIFFRACTION	19
4.2.2	SCANNING ELECTRON MICROSCOPE	20
4.2.3	FOURIER TRANSFORM IR SPECTROSCOPY	21
5	RESULT AND DISCUSSION	23
5.1	X-RAY DIFFRACTION	24
5.2	SCANNING ELECTRON MICROSCOPE	26
5.3	FOURIER TRANSFORM IR SPECTROSCOPY	27
5.4	PHOTOCATLYTIC ACTIVITY TEST	28
6	CONCLUSION	32
7	REFERENCES	33

LIST OF FIGURES

Figure No.	Title	Page No.
1.1	Schematic diagram of the classification of dimensional nanoparticles	2
1.2	Schematic diagram of the synthesis method of Sol-gel technique	5
1.3	The process of co-precipitation method	6
1.4	The process involves in hydrothermal autoclave reactor	7
1.5	Schematic diagram of the Thermal evaporation process	8
1.6	Schematic diagram of the Electrochemical depostion process	9
1.7	Types and applications of metal oxide nanoparticles	10
1.8	Representation of the Crystal structure of NiO	11
1.9	Decolourization of Fast Green (FG) dye under visible light irradiation	14
4.1	The working principle of XRD	20
4.2	The process of scanning electron microscope	21

4.3	The working principle of FTIR	22
5.1	photocopy of Li	23
5.2	Photocopy of Li/Ni(OH) ₂	23
5.3	XRD of NiO and Li doped Ni (OH) ₂	25
5.4	SEM image of pure NiO nanoparticles and SEM image of Li doped Ni(OH) ₂	26
5.5	FTIR of NiO and Li doped Ni(OH) ₂	27
5.6	The doping effect of Li ion at the Ni ²⁺ lattice sites of graph	29
5.7	Schematic diagram illustrating the mechanism of charge separation and photocatalytic activity of photocatalyst under the sunlight irradiation	31

LIST OF TABLE

Table No.	Title	Page No.
5.7	Photocatalytic activity result of NiO and Li/ Ni(OH) ₂ nanoparticles.	31

CHAPTER-1

1.INTRODUCTION

1.1NANOMATERIALS:

Nanoparticles are attracting several researchers due to their varied applications. The size contributes tremendously to expanding the surface area, thereby making their optical and electrical properties highly sensitive to surface morphology [1]. Nanotechnology is an effective 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 nanomaterial's had evolved as a frontline research area [2,3] the materials with the nanometre scale demonstrate the properties of a nanoparticle from the bulk materials, the nanoparticles are generally known as micro-particles that are lesser than 100 nm in diameter nanoparticles of the same material can show enhanced Catalytic, electronic, magnetic properties [4] of metal nanoparticles are sensitive to their size and shape. 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).

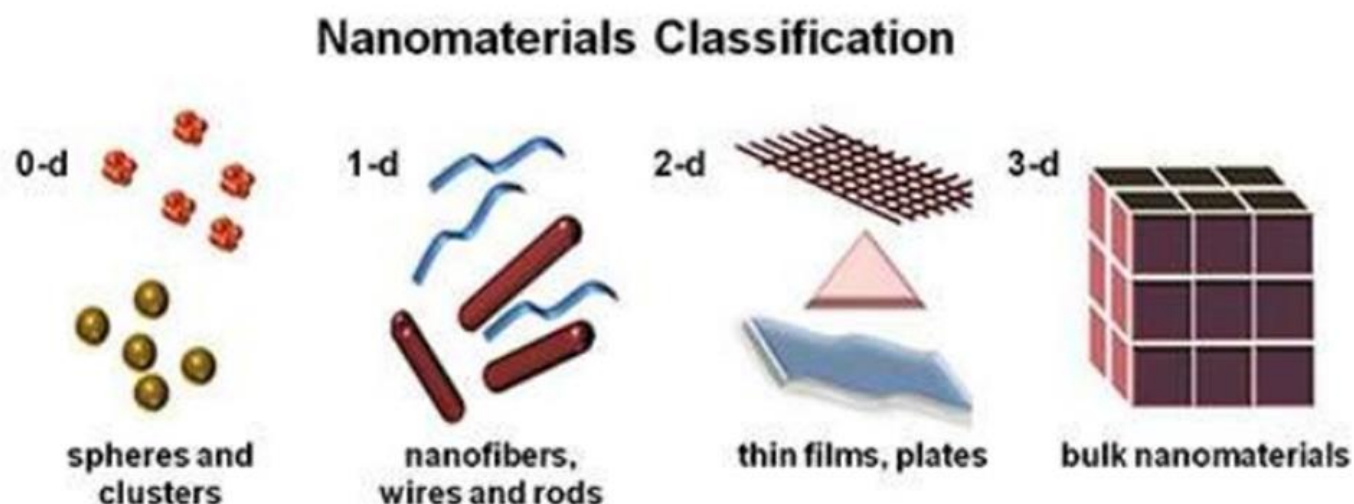
In zero dimensional nanomaterials are measured within the nanoscale its dimension is not more than 100 nm. It is called as nanoparticles.

In one dimensional nanomaterial is outside the nanoscale this class include nanotubes, nanorods [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.

Figure1.1shows the classification of dimensional nanoparticles



In this case, we used a Bottom-down approach in that we did a synthesis of nanoparticle using co precipitation method, which 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 polyvinylpyrrolidone [PVP], polyvinyl alcohol [PVA], polyethylene glycol [PEG], the goal of the work is to synthesis of lithium doped NiO nanoparticles by the chemical co-precipitation method in the presence sodium hydroxide, it is very simple way and low in cost.

Semiconductor nanoparticles is an effective class of materials that have a mechanical, optical and electronic properties. The main criteria of nanostructure based on their sources, dimensions, structural configuration and nature of core have been in that particles. The materials do not belong only to crystal solids but are of the 3 nature of amorphous and liquids too. Semiconducting core and shell materials based on their energy level. The key developments of materials in that field such as electronic, health, agricultural, food processing and so on [11]

As semiconductors are reduced to the nanometre scale, they become pure conductors. By incorporating very small amounts of other materials called dopants, the electronic properties and conductivity of semiconductors can be modified in a controlled manner. As chemical properties are reduced to the nanoscale, they change dramatically and become insulators, with no space for free electrons. Preparation and applications of the metal oxide nanostructure 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 [12-14].

1.2. METAL OXIDES:

Optical, thermal, catalytic, photochemical, and other special properties of metal oxides have made them a high-level technical application. As a result, researchers are interested in learning more about the metal oxide nanostructure. These materials exhibit the most fascinating and diverse variety of metal oxide properties. They primarily react with water to form bases or acids to form salts. Metal oxides are considered as a heterogeneous catalyst and some group of metals have much attraction towards their outer electronic configuration. This oxide involves liquid-solid or gas-solid transformations. 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 top-down and bottom-up approaches are used to synthesis a metal oxide nanoparticle [1,2,15-20].

1.3 SYNTHESIS TECHINQUES OF METAL OXIDE NANOPARTICLES:

Synthesis of metal oxide nanoparticles with the size and structure and the field of nanoscience and nanotechnology. So there has a variety approaches to synthesis of nanostructures such as thermal evaporation, chemical 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, nanoparticles having unique properties from their nanoscale dimensions with fast

ion are related also to nanoionics and nanoelectronics. Nanoscale materials can also be used for bulk application. 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.

Top-down approaches:

This approach refers to slicing or successive cutting of a bulk material to get nanoparticles 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].

Bottom-up 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 ultrafine nanoparticle but the quantity of product is we get a smaller quantity of nanoparticles.

We did a report for different approaches in this literature, such as sol-gel technique, chemical precipitation method, thermal evaporation process and electrochemical deposition process to improve the techniques and their growth of nanostructures.

1.3.1.SOL-GEL TECHNIQUE

Sol-gel process is a method of producing solid materials from small molecules. Sol-gel precipitation approach has evolved for the preparation of nanoparticles with inorganic materials [22-24]. Figure 1 and 2 shows the synthesis of the sol-gel form, which is a more familiar approach. This process is based on the hydrolysis of a precursor solution to make a colloidal particle known as a sol, which is then condensed to form a gel, which is then heated to achieve the desired yield. Nanoparticles such as TiO_2 , CuO , and others metal oxides are synthesised.

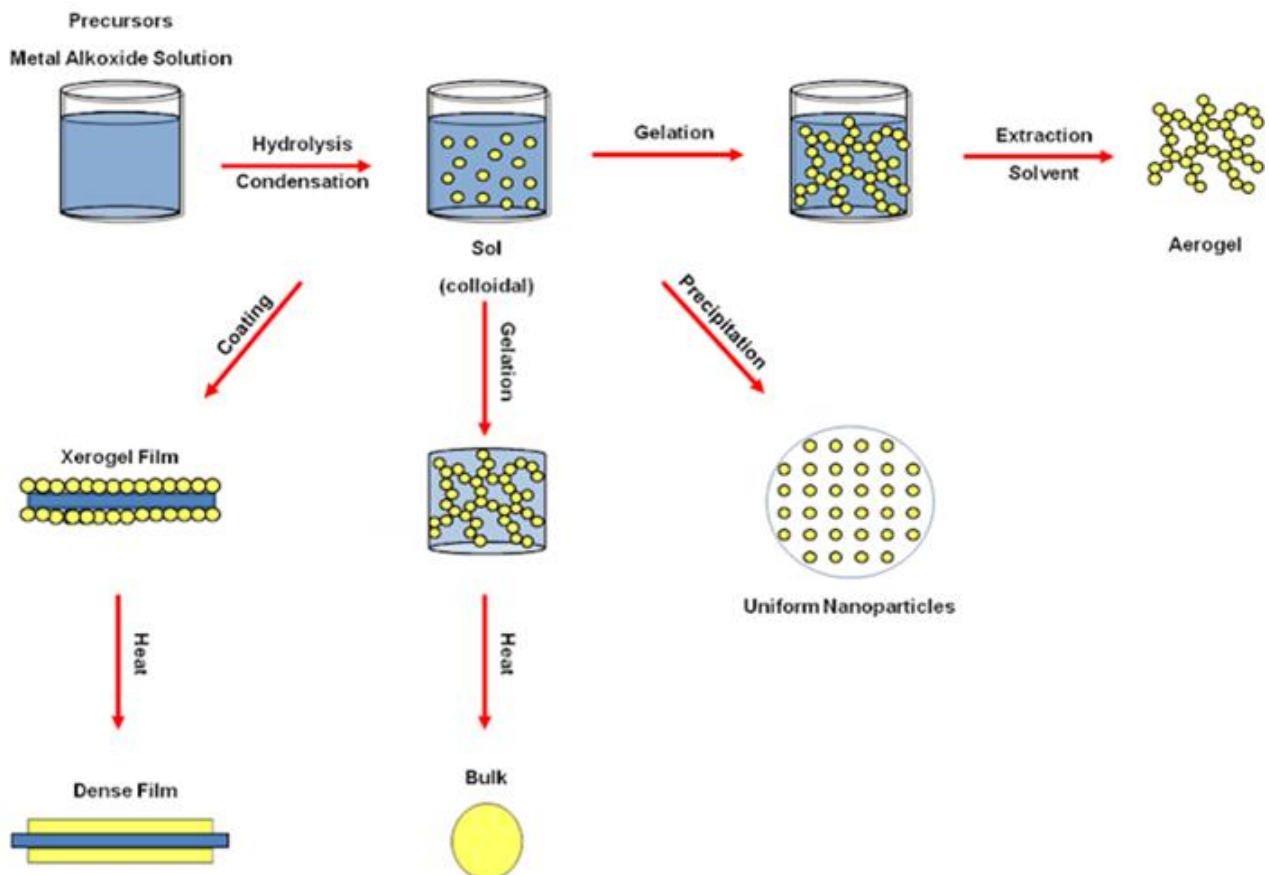
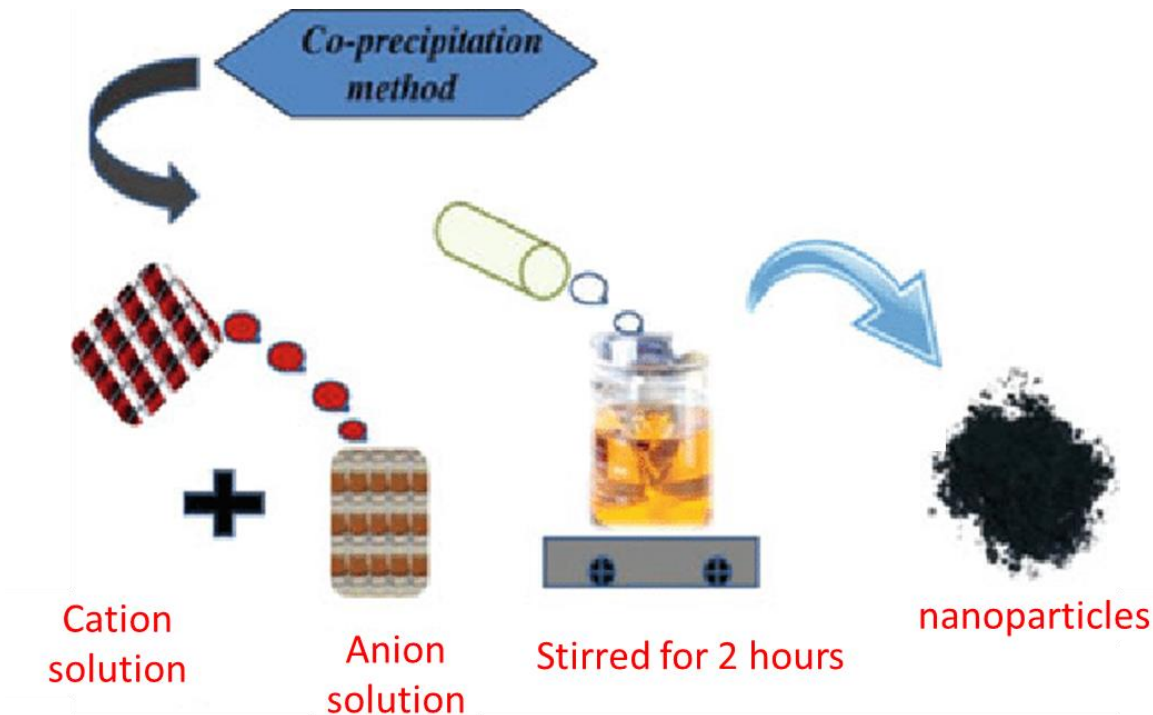


Fig 1.2. Synthesis method of Sol-gel technique

1.3.2.CO-PRECIPITATION METHOD:

The simultaneous occurrence of nucleation, formation, and agglomeration processes is known as a co-precipitation reaction. In generally, insoluble species formed under condition of high supersaturation. Nucleation is a key step to form a large number of small particles are obtained. Co-precipitation method is a standard synthesis technique to prepare a pure metal oxide nanoparticle. Fig 1.3 shows the process of co-precipitation method. In co-precipitation, a salt precursor is usually a nitrate, chloride, and dissolved in aqueous solvents, followed by a base compound and hydroxides such as sodium hydroxide, ammonium hydroxide, and others [25]. Add a stabilising agent like PVP, PVA, etc. to minimise agglomeration, and control the size and morphology of the nanoparticles [26]. The precipitation that we get has been washed several times with water and acetone and dried at 160 °C.

Fig.1.3 process of co-precipitation method



1.3.3. HYDROTHERMAL PROCESS:

The method of making single crystals that relies on mineral solubility in hot water under high pressure. Especially very well to the formation of large, high-quality crystals while retaining composition control. Figure 1.4 illustrates the mechanism involved in an autoclave reactor. It's a solution-based approach [27]. The ability to synthesize crystal substances that are unstable near the melting point, as well as the ability to synthesize large crystals of high quality, are two advantages of the hydrothermal synthesis process. The high cost of the equipment and the inability to monitor the crystal growth are also drawbacks [28]. And it is a 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 photocatalytic properties.

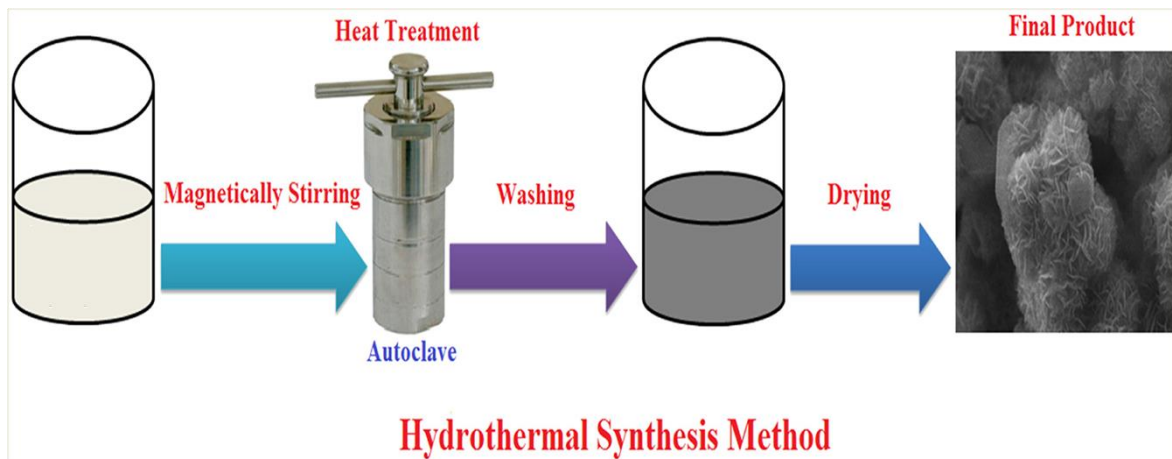


Fig.1.4 process involve in hydrothermal autoclave reactor

1.3.4. THERMAL EVAPORATION PROCESS:

The nanostructures are grown up by the many materials of this process is known as thermal evaporation method. This technique needs of high temperature thermal furnace used for vaporising the source of materials and facilities. The deposition of nanostructures at relatively low temperature. The variety of semiconductor materials wide range of elemental nanowires, grown up the numerous nanoparticles. Fig 1.5 shows this process generated first by physical or chemical method under a condition followed by temperature, pressure on silicon substrate. The thermal evaporation process contains horizontal quartz tube, furnace, with rotatory pump and gas supply system [29].

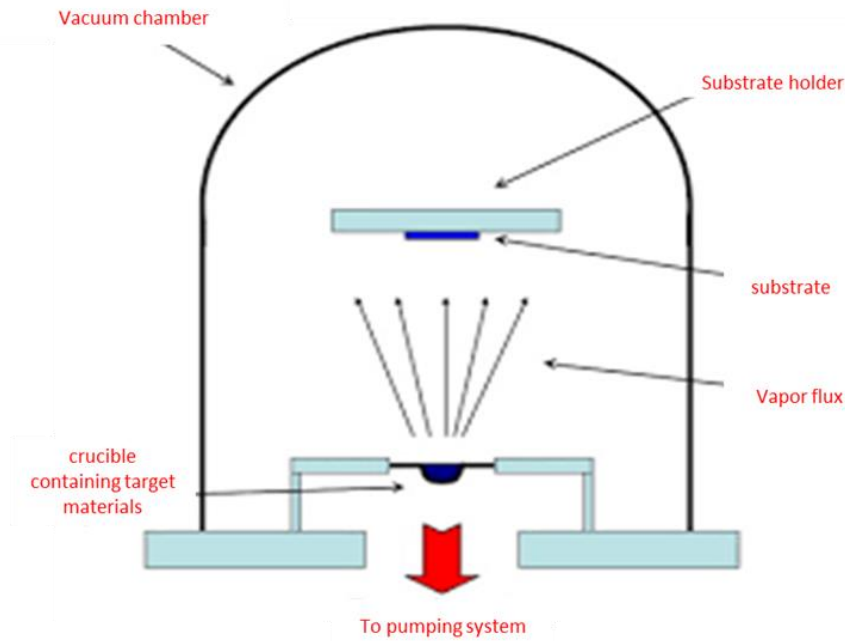


Figure1.5 Thermal evaporation process

1.3.5. ELECTROCHEMICAL DEPOSITION PROCESS

This chemical method used for the fabrication of metallic nanowires in the porous structures. In the low cost and a scalable technique, in the largescale semiconductor thin film product. Recently this method is attracted due to its short reaction times and low cost. Fig 1.6 shows this anode and cathode of this electrochemical method diagram of this process refers to a film grow formation of a metallic coating on a base material due to the electrochemical reduction metal ions form an electrode [30].

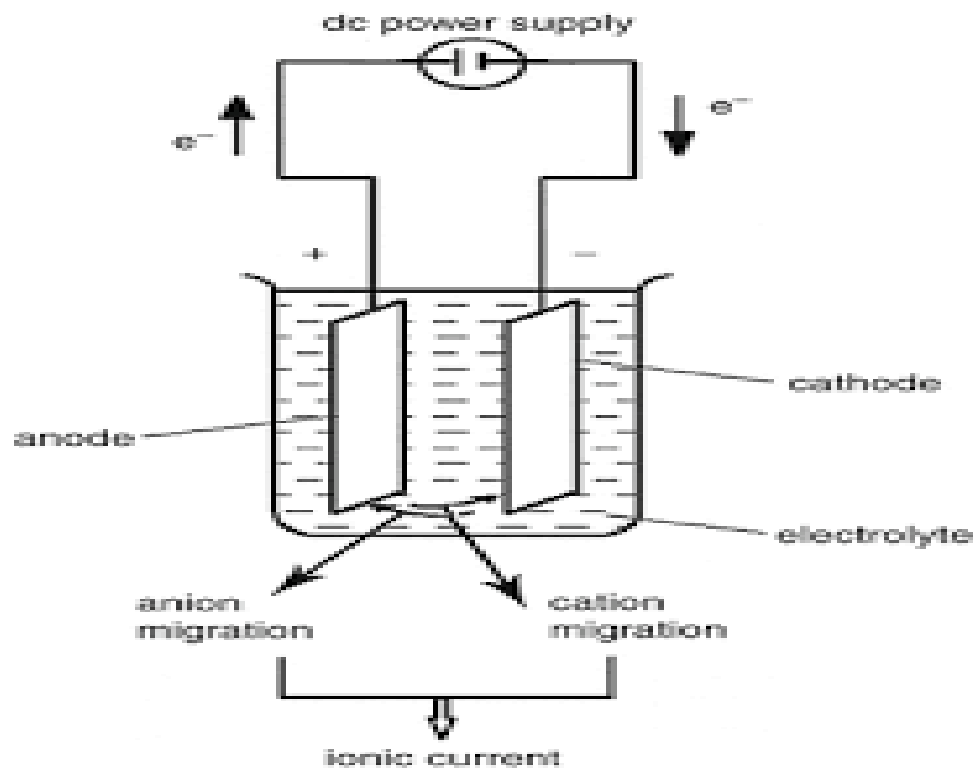


Figure 1:6*Electrochemical deposition process*

1.4 IMPORTANCE OF METAL OXIDE:

This is oxide for effective interaction with target materials reducing the grain size to nanoscale increases the active surface such as bandgap wide at room temperature. The optical, electrical, catalytic, and magnetic properties of metal metal ions, and also their physical and chemical stability, have attracted researchers. Due to various its small size and high density of corner or edge surface sites, metal Oxide nanoparticles may have unusual chemical and physical properties. [31]. Metal oxide nanoparticles have a variety of applications such as catalyst etc. [32]. 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 can create a wide variety of oxide compounds. These can have a wide range of structural geometries, as well as an electronic structure that can have insulator, semiconductor, or metallic properties.



Fig 1.7 Types and applications of metal oxide nanoparticles [33]

1.5 TARGETED METAL OXIDE:

1.5.1 NiO:

This particle focused on that production of nanostructured metal oxide materials such as NiO particles. NiO particles have been develop many methods including low pressure spray pyrolysis surfactant mediated method simple liquid phase process and other techniques this various method for controlled synthesis of that nanostructure. The average diameter of 30nm using a polyvinylpyrrolidone of that nanomaterials. NiO is an important transition metal oxide with a cubic lattice structure. Fig 1.6shows the crystal structure of NiO. Because of its wide band gap, this stable p-type semiconductor metal oxide has a tendency to absorb UV radiation. NiO has a band gap of 3.4-4 eV on average [34]. NiO nanoparticle is more effective photocatalyst. NiO is a transition metal oxide with a cubic lattice structure that is widely used. Nickel oxide has attracted considerable attention because of its excellent electrical, optical, and catalytic properties, as well as its high photo sensitivity

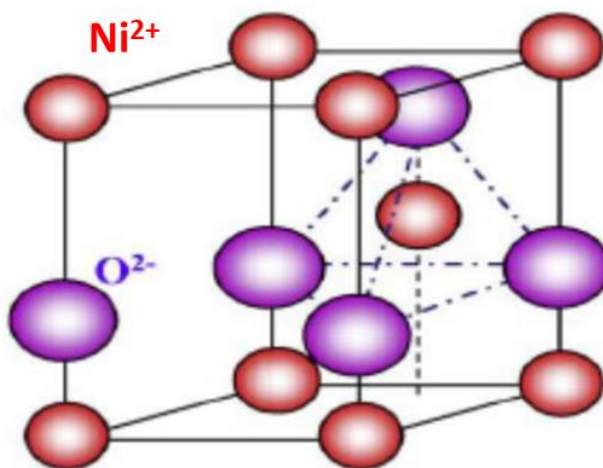


Fig 1.8 Crystal structure of NiO

1.5.2 PROPERTIES OF NiO:

Chemical Symbol	NiO
Molecular weight	74.71 g/mol
Melting point	1955 °C
Density	6.67 g/cm ³
Electronic configuration	Nickel [Ar]3d ⁸ 4s ² Oxygen [He] 2s ² 2p ⁴

1.5.3KEY APPLICATION OF NiO:

- [1] In electrochromic coatings, plastics and textiles
- [2] In nanowires, nanofibers and specific alloy and catalyst applications
- [3] Adhesive and colouring agents for enamels
- [4] In active optical filters
- [5] In ceramic structures
- [6] In automotive rear-view mirrors with adjustable reflectance
- [7] In cathode materials for alkaline batteries
- [8] Electro chromic materials
- [9] Energy efficient smart windows
- [10]P-type transparent conductive films

1.5.4. Ni (OH)₂ NANOPARTICLES:

Nano material using the high developing field of research production due to small size and their unique properties of micro constituents. Nanosized structures with different morphologies demand in such important spheres. Human activities as medicine energy microelectronics etc. controlling the dispersion and particle shape has not been dissolved. Nickel hydroxide forms around 200°C, whereas Nickel hydroxide has some advantage as well as applications. In Nickel hydroxide a non-aggregated β- phase hexagonal brucite structure was observed. In exhibit long term cyclic stability of 93% after 1000 cycles. Nickel hydroxide can be formed upon their pH [35]. We intend to investigate NiO and the formation of Ni (OH)₂ with Li ion at low temperatures as part of this project. We will use the co-precipitation method to explore the synthesis of the above-mentioned metal oxide nanostructures in this project. The metal oxides we're looking at in this paper are Li doped Nickel hydroxide. X-Ray Diffraction [XRD], Scanning Electron Microscope [SEM], Fourier transform infrared spectroscopy (FTIR) were used to characterise the size, morphology, and structure of the prepared samples.

1.6 TARGETED APPLICATION OF Ni(OH)₂ and NiO NANOPARTICLES PRESENT IN THIS PROCESS

1.6.1 PHOTOCATALYST APPLICATION:

Organic chemicals found as pollutants in waste water effluents from industrial or domestic sources must be removed or destroyed before being released into the environment. The aqueous system for natural purification. The breakdown of organic molecules into simpler molecules occurs in lagoons, ponds, streams, rivers, and lakes as a result of sun. The photocatalyst absorbs photons with energies that are equal to or greater than its band gap of threshold energy. Photocatalyst are also self-regenerate and can be reused or recycle. Each photon of the required energy that hits electrons in the occupied valence band of the semiconductor atom [36]. Photocatalyst is defined as speeding up the chemical reaction in the presence of UV-light. The photocatalytic process begins with the absorption of light by a semiconductor material, which creates an electron hole (e⁻/h⁺) pair. The formation of aggressive species such as hydroxyl is induced by photo-generated pairs of h⁺

and e^- (OH^-). Some of the significant features of the photocatalyst have proper band gap, morphology of the material, stability and its reusability. The photocatalyst is widely practiced in degradation process thus leads a reduction toxic metal ion into non-toxic state. Based on photocatalysts nanoparticles. These photogenerated charge carriers create radical species that cause dye degradation when exposed to light. With respect to temperature, the structural, morphological, optical, adsorption, and photocatalytic properties of NiO and Li -Ni (OH)₂ NPs have been investigated using FAST GREEN dye.

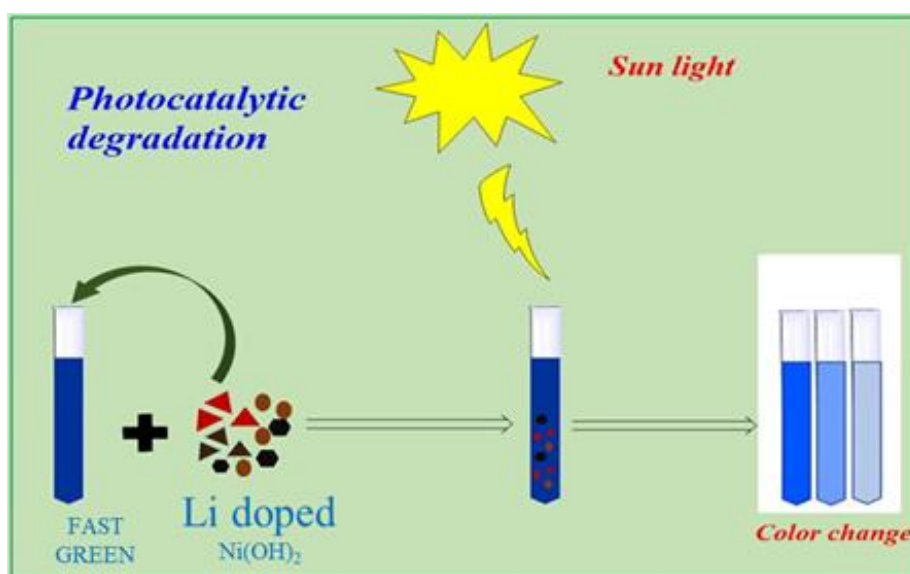


Fig 1.9 Decolourization of Fast Green (FG) dye under visible light irradiation.

1.7OBJECTIVE:

- Investigation on photocatalytic performances of Li doped Ni(OH)₂ nanoparticles synthesized by a facile co-precipitation method.
- Influences of Lithium ion on the size, morphology and structural constitution will be studied.
- Effect of the photocatalytic activity FAST GREEN dye was performed using undoped and Li doped Nickel hydroxide nanoparticles were investigated.
- Synthesis of low temperature-based Li doped Ni (OH)₂ nanoparticle.
- Studies on size, morphology and structure using X-Ray Diffraction [XRD], Scanning Electron Microscope [SEM], Fourier transform infrared spectroscopy [FTIR].

CHAPTER- 2

2. LITERATURE SURVEY

In this application photo- catalyst are due to light and catalyst, nanomaterials most efficient photo catalyst are semiconductor photocatalytic activity, of this is oxide for effective interaction with target materials reducing the grain size to nanoscale increases the active surface such as bandgap wide at room temperature. The optical, electrical, catalytic, and magnetic properties of metal metal ions, and also their physical and chemical stability, have attracted researchers. The photocatalyst NiO nanoparticle is more efficient. NiO is a commonly used transition metal oxide with a cubic lattice structure. As part of this research, we want to look into NiO and the formation of Ni (OH)₂ with Li ions at low temperatures. This project by using NiO and Li-doped Ni (OH)₂ as nanomaterials. Metal oxide nanomaterials were prepared by specific technique, simple and low-temperature co-precipitation process. The prepared nanomaterials were characterised using an XRD pattern to analyse their structural properties. FTIR spectroscopy was used to determine the elemental and chemical compositions of prepared nanomaterials whereas the scanning electrode microscope was used to determine the size of the nanoparticles, Finally, photocatalytic applications were developed using the synthesised nanomaterials. The effect of doped samples and Li doped Nickel hydroxide nanoparticles on the photocatalytic behaviour of the FAST GREEN dye was investigated.

CHAPTER -3

AIM AND SCOPE OF THE PRESENT INVESTIGATION

The present work has been planned as an effort of these particles, synthesised the particles of nanomaterials of a key factor of nanostructure were focused on their origins, dimensions, structural configuration, and type of centre. The products are not only crystal solids, but also amorphous and liquids of three different natures. The photocatalytic application of the process as rebuild themselves and can be reused or recycled. Each photon with the required energy that strikes electrons in the semiconductor atoms occupied valence band. The characterization of that X-Ray Diffraction [XRD], Scanning Electron Microscope [SEM], Fourier transform infrared spectroscopy (FTIR) were used to characterise the size, morphology, and structure of the prepared samples. Aim of that project photodegradation activity of Nanoparticles prepared at the low temperature of lithium doped nickel hydroxide it is the simple and low cost at the characterized particles of photodegradation using the dye FAST GREEN.

CHAPTER-4

EXPERIMENTAL AND CHARACTERIZATION TECHNIQUES

4.1 AIM

To synthesize the photodegradation activity of lithium doped nickel hydroxide nanoparticles.

4.1.1 MATERIALS REQUIRED:

All chemicals used in this project were analytical grade and used without any further purification. Nickel chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) (MERCK), Lithium bis (trifluoromethanesulfonimide) (Li) (SIGMA ALDRICH), Sodium Hydroxide (NaOH), (SRL), Polyvinyl pyrrolidone (PVP), (SRL), Ethanol, Acetone (SRL), and All samples were prepared by using De-ionised water.

4.1.2 EXPERIMENTAL PROCEDURE:

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

4.1.3 SYNTHESIS OF NiO:

The technique of synthesis is modified to produce NiO nanoparticles by co-precipitation of Ni (OH)₂. nickel (II) chloride hexahydrate (0.5M) in D.I water, with 1g of polyvinyl pyrrolidone added to reduce agglomeration (PVP). Sodium hydroxide was added drop by drop to alter the pH. At room temperature, the reaction was stirred for 2 hours. The final product was thoroughly washed with deionized water to remove reaction residues before being washed with acetone. Finally, vacuum drying was used to create deep green nanoparticles. The prepared sample was calcined at 450°C to convert the nickel hydroxide nanoparticles to NiO.

4.1.4 SYNTHESIS OF Ni (OH)₂:

Co-precipitation has been used to synthesis Li doped Ni (OH)₂ nanoparticles. In D.I water dissolve (0.5M) of nickel (II) chloride hexahydrate, add 5% lithium, and 1g polyvinyl alter the pH. At room temperature, the reaction was stirred for 2 hours. The

end product was thoroughly washed with Deionized water to remove reaction residues after that washed with acetone. Finally, 120°C was used to dry the prepared nanoparticles.

4.2 CHARACTERATION OF PREPARED METAL OXIDE NANOPARTICLES

The nanomaterials of the metal oxide are presented in this project when prepared in terms of XRD was used to characterise the morphology, structural properties, and optical properties of these nanoparticles. The scanning electron microscope was used to determine the size of the nanoparticles, as well as the chemical composition of the nanoparticles using FTIR spectroscopy.

4.2.1 X-RAY DIFFRACTION:

When X-Rays interact with matter, they are reflected, scattered ineffectually, absorbed, refracted, and transmitted. Amorphous glass does not long-range atomic order and therefore produces only broad scattering features. When analysing nanoscale materials, XRD data is frequently encountered. The nanoparticles of various sizes, shapes, and dimensionalities [37]. X-Ray diffraction issued to determine the crystal structure of the material. After a material has been synthesised, it is critical to ascertain its structural nature. Because a material's properties are highly dependent on its structure. For phase identification of a wide range of crystalline phases in solids, the standard database (JCPDS database) for XRD patterns is used as a solid specimen. XRD is used for assessment of phase purity and crystallinity of inorganic materials [38]. X-ray diffraction is based on Bragg's law:

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

where d denotes the perpendicular distance between pairs of adjacent planes, θ denotes 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 between waves dispersed by adjacent planes of atoms in terms of

wavelength [39]. 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 K represents the particle shape coefficient (0.9 for spheres), θ is the diffraction angle β is the peak's Full Width Half Maximum (FWHM), D represents the average crystalline size, λ is the incident light wave length (Cu-K) (1.5405).

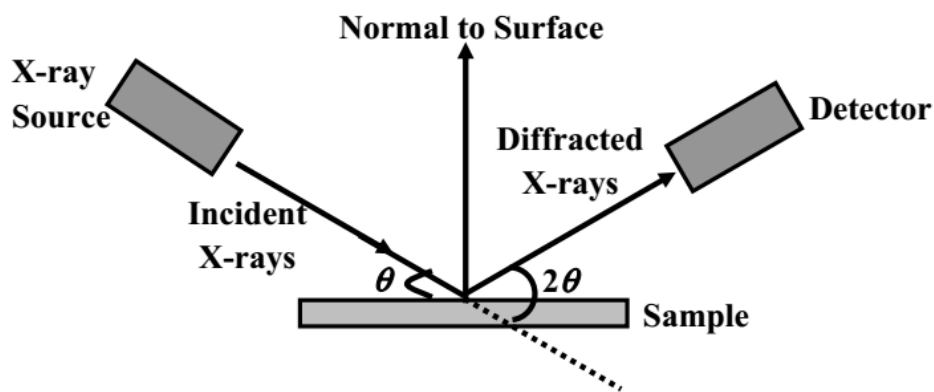


Fig 4.1 working principle of XRD

4.2.2 SCANNING ELECTRON MICROSCOPE:

One of the most popular methods for imaging the microstructure and morphology of materials is to use a scanning electron microscope (SEM). In a scanning electron microscope (SEM), a low-energy electron beam is radiated to the specimen and scans the sample's surface. The electron gun is a device that emits electrons and is located on top of the column. The electrons are then accelerated to energy levels ranging from 0.1 to 30 keV, resulting in a large diameter electron beam that can be used to create high-resolution images. Control the shape of electron beam to form a small focused electron spot on the sample using magnetic lenses and beams. A high-vacuum environment that allows electrons moving without being scattered or absorbed by the air[40]. 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[41]fig4.2 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. EDX (Energy dispersive X-Ray analysis) may also include specific information on elemental composition.

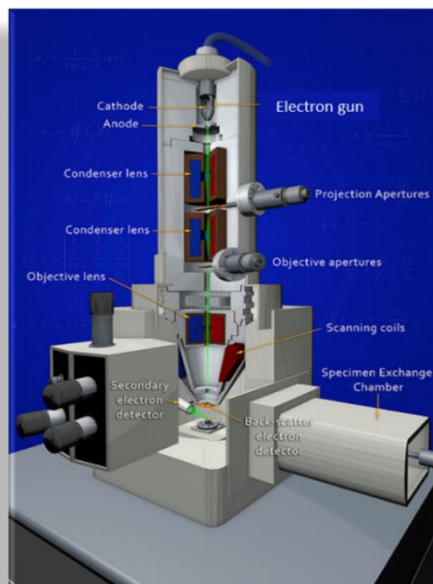


Fig 4.2 The process of scanning electron microscope

4.2.3 FOURIER TRANSFORM IR SPECTROSCOPY:

Fourier transform infrared (FTIR) spectroscopy is an old yet emerging instrumentation technique that uses molecular interaction with samples through infrared electromagnetic radiation to predict the structure of small molecules and natural products, functional groups present in molecules, analysis of nanoparticle surfaces, and nanoparticle functionalization, and also recognition of various diseases and imaging FTIR the term refers as Fourier transform infrared spectroscopy; Molecules have a vibrational excitation energy of 10^{13} – 10^{14} Hz, which corresponds to infrared radiation. 5 As a result, IR spectroscopy can be used to observe the vibrational transitions of self-assembled functional groups coordinated to nanoparticle surfaces, and quantitative. Fourier transform infrared (FTIR) is a spectroscopy method that can identify variations in the total composition of microorganisms through the determination of changes in functional groups in biomolecules. The sample absorbs some of the infrared radiation and passes some of it through (transmitted). The resulting spectrum represents molecular absorption

and transmission while also depicting molecular energy levels, is shown in Fig 4.3. 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 [42]. Fourier Transform Infrared (FTIR) spectrometer first generates an interference of a sample signal with an interferometer, which simultaneously analyses all infrared frequencies. One beam is reflected by a flat, surface mounted mirror. The reflection is reflected in a flat mirror. Another beam simply moves away from the beam splitter a short distance (usually a few millimetres). From their respective mirrors, the two beams refract. When they are meet together at the beam splitter, they are recombined. By measuring the vibrational frequencies of the chemical bonds involved, FT-IR spectroscopy provides useful insight into the various functional groups that are present in a system. The infrared signal generated by interacting with the sample is unique to the sample. FTIR is a technique which is used to obtain infrared spectrum of absorption, emission, and photoconductivity of solid, liquid, and gas. it can be used for microscopy and imaging of rubber and particle interfacial modifications [43]. When the beam reaches the detector, it is measured by the detector. It must be "decoded" using a mathematical method known as Fourier Transformation. The machine can perform a Fourier analysis and display an infrared spectrum plotting absorbance against wavenumber.

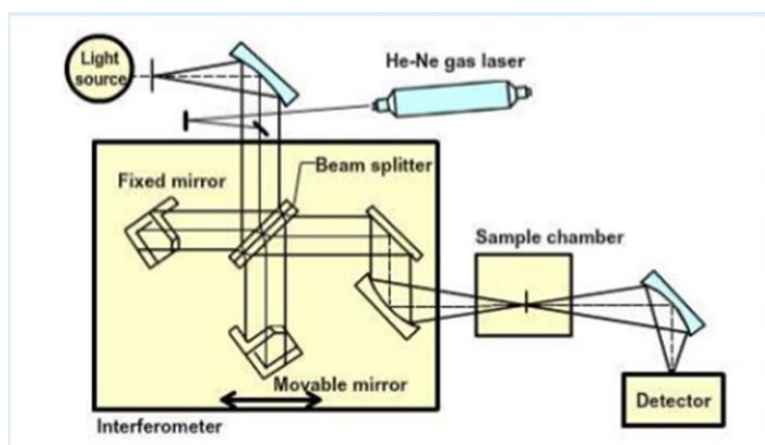


Fig 4.3 the working principle of FTIR

CHAPTER-5

RESULTS AND DISCUSSION:

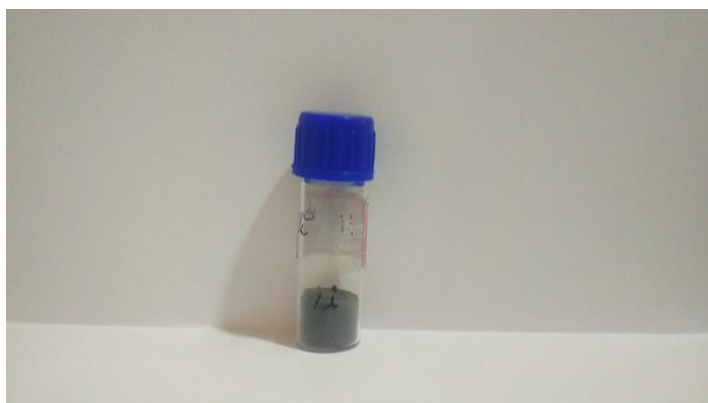


Fig.5.1. photocopy of Li



Fig.5.2. Photocopy of Li/Ni(OH)₂

5.1. X-RAY DIFFRACTION:

XRD is a unique technique for revealing the structural properties of nanoparticles. It goes over phase, crystallinity, and nanoparticle properties. This method can distinguish between single and multiphase nanoparticles [44-45] The crystalline size was calculated using Debye Scherrer equation:

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

CuK α source wavelength of $\lambda = 1.5405 \text{ \AA}$, $K = 0.9$, β = full width half maximum crystalline parameters of the prepared NiO nanoparticle diffraction patterns were observed at a variety of angles of diffraction (2θ), ranging from 10° to 90° .

Figure 5.3 XRD patterns of NiO hollow spheres, with five main diffraction peaks corresponding to the standard cubic NiO structure, 111, 200, 220, 311, and 222. (JCPDS card 04-0835). The absence of an impurity-phase peak indicates that the products are pure. NiO in its purest form.

Figure 5.3: Diffraction reflections at 19.17° , 31.64° , 39.60° , 45.81° , 51.70° , 59.69° and 75.16° for Li doped β - Ni (OH) $_2$ spheres. The sample's XRD patterns under different experimental conditions. The XRD patterns of nanoparticles samples obtained under different experimental conditions. According to (JCPDS card No 14-0117), all of the peaks are belongs to the hexagonal phase of β -Ni(OH) $_2$. Other precursor compounds of bare Ni substrate show distinctive peaks at 44° and 75° shows that as the reaction time increases, the diffraction peaks become more intense, indicating that a longer reaction time improves the crystallinity of the products.

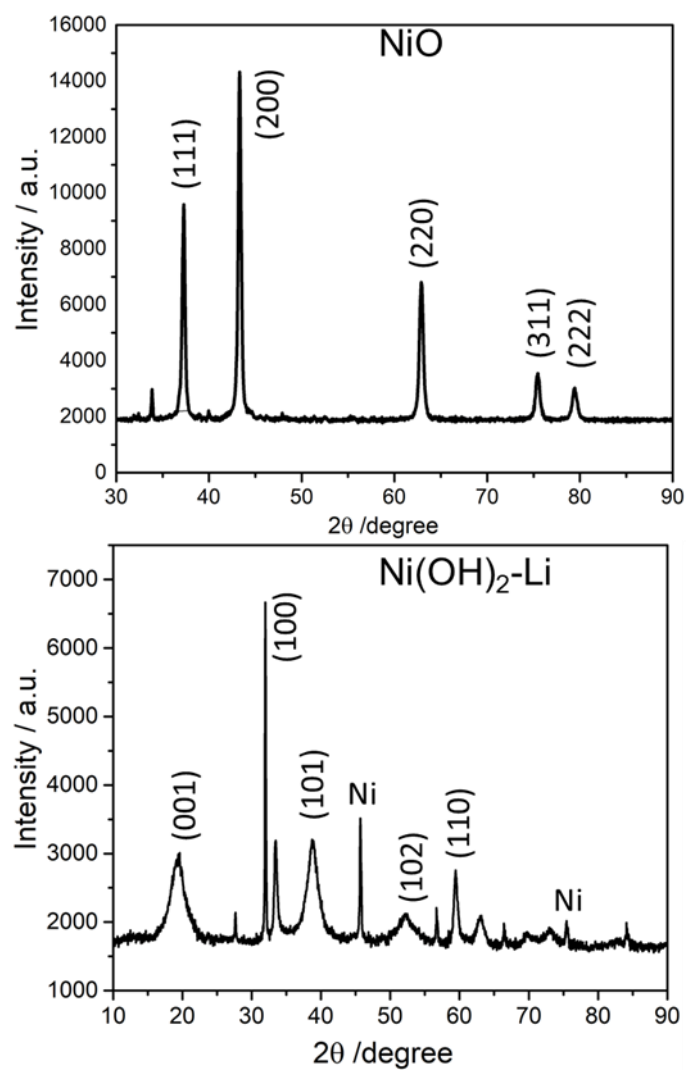


Fig 5.3 XRD of NiO and Li doped Ni (OH)₂

The crystalline size of Lithium doped Nickel hydroxide; It is obtained as was calculated using Debye scherrer equation.

5.2. SCANNING ELECTRON MICROSCOPE:

The morphological differences in the synthesised sample are observed and analysed using SEM.

A SEM analysis of Nickel oxide is shown in Figure 5.4(a). And the images of Li doped Nickel hydroxide nanoparticles synthesised at low temperatures were studied and shown in Figure 5.4 (b)

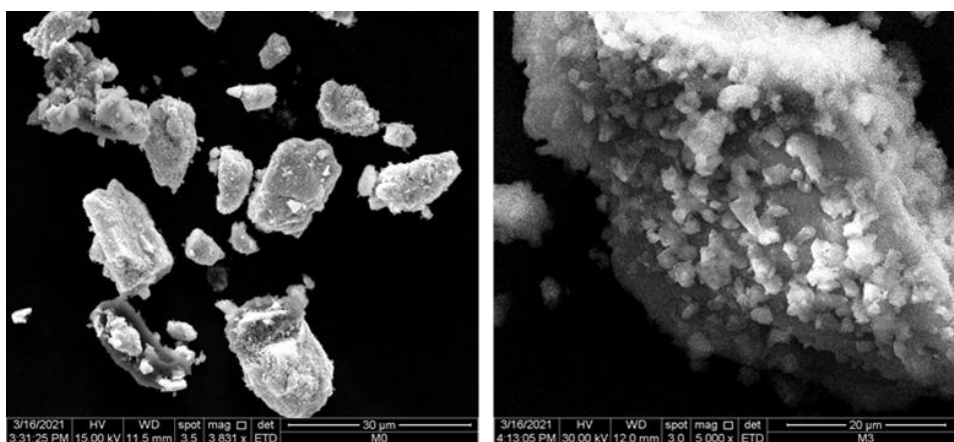


Figure 5.4(a) Scanning electron microscopy (SEM) image of pure NiO nanoparticles and (b) SEM image of Li doped Ni(OH)₂ nanoparticles. The SEM images were measured for powder samples for the both samples (NiO and Li doped Ni(OH)₂). The powder samples were pasted on the carbon tape for measuring the SEM images.

Nickel oxide has a greater surface energy than Lithium doped nickel hydroxide because it is NiO is agglomerated [46]. In comparison to pure nickel oxide, Lithium doped nickel hydroxide has fine nanostructure. Because of their high surface area to volume ratio and lower cohesive energy than pure NiO nanoparticles, the synthesised NiO nanoparticles are smooth and spherical in shape, and some are agglomerated. SEM image of Lithium doped Nickel Hydroxide are spherical. Lithium doped Nickel Hydroxide has a smaller size than pure Nickel Oxide due to its high dispersion [47]. Nickel oxide can be de-agglomerated by calcination at a higher temperature of around 160°C [48]. According to SEM images, the size of Lithium doped Nickel Hydroxide may be less than 10nm.

5.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY:

FTIR analysis measures the absorbance of infrared light at various wavelengths in a sample to determine the material's molecular composition and structure. Figure 5.5 (A) On the FT-IR spectrum, the O-H bond is reflected by a peak around 3499cm^{-1} . Because of the absorption at 1650cm^{-1} , the hydroxyl groups are obtained. The absorption bands at 1460cm^{-1} and 1133cm^{-1} confirm the presence of carbonates, while the bond at 2920cm^{-1} corresponds to the CH_2 stretching mode [49]. The Ni-O vibration bond is associated with the absorption bands 550cm^{-1} . Figure 5.5 (B) The stretching vibrational mode (OH) of non-hydrogen bonded hydroxyl groups in Nickel hydroxide is assigned to the sharp peak at 3635cm^{-1} , while the wide band at 3455cm^{-1} is assigned to the stretching mode of hydrogen bonded hydroxyl groups in the same layered structure [50].

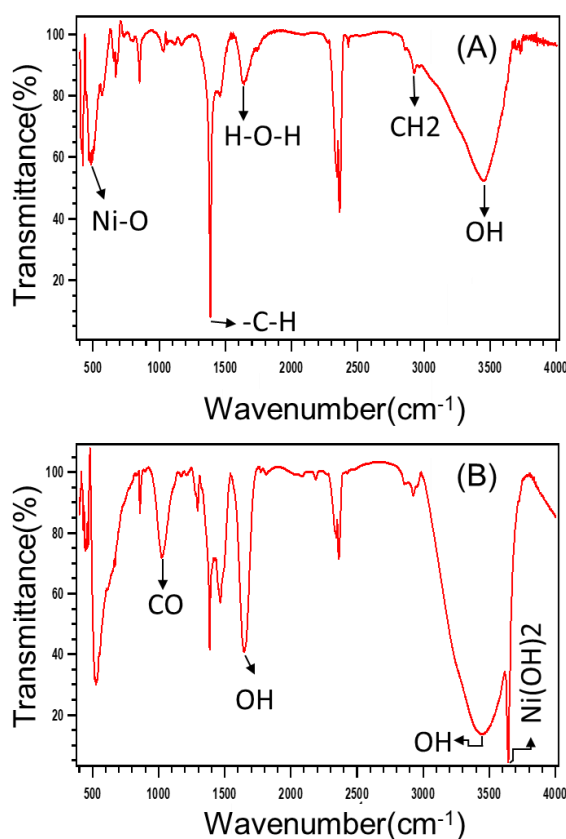


Fig.5.5. FTIR of NiO and Li doped Ni (OH)₂

5.4 PHOTOCATALYTIC ACTIVITY TEST:

Fast green (FG) 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 $0.2 \mu\text{m}$ 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,

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

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 FG as follows;

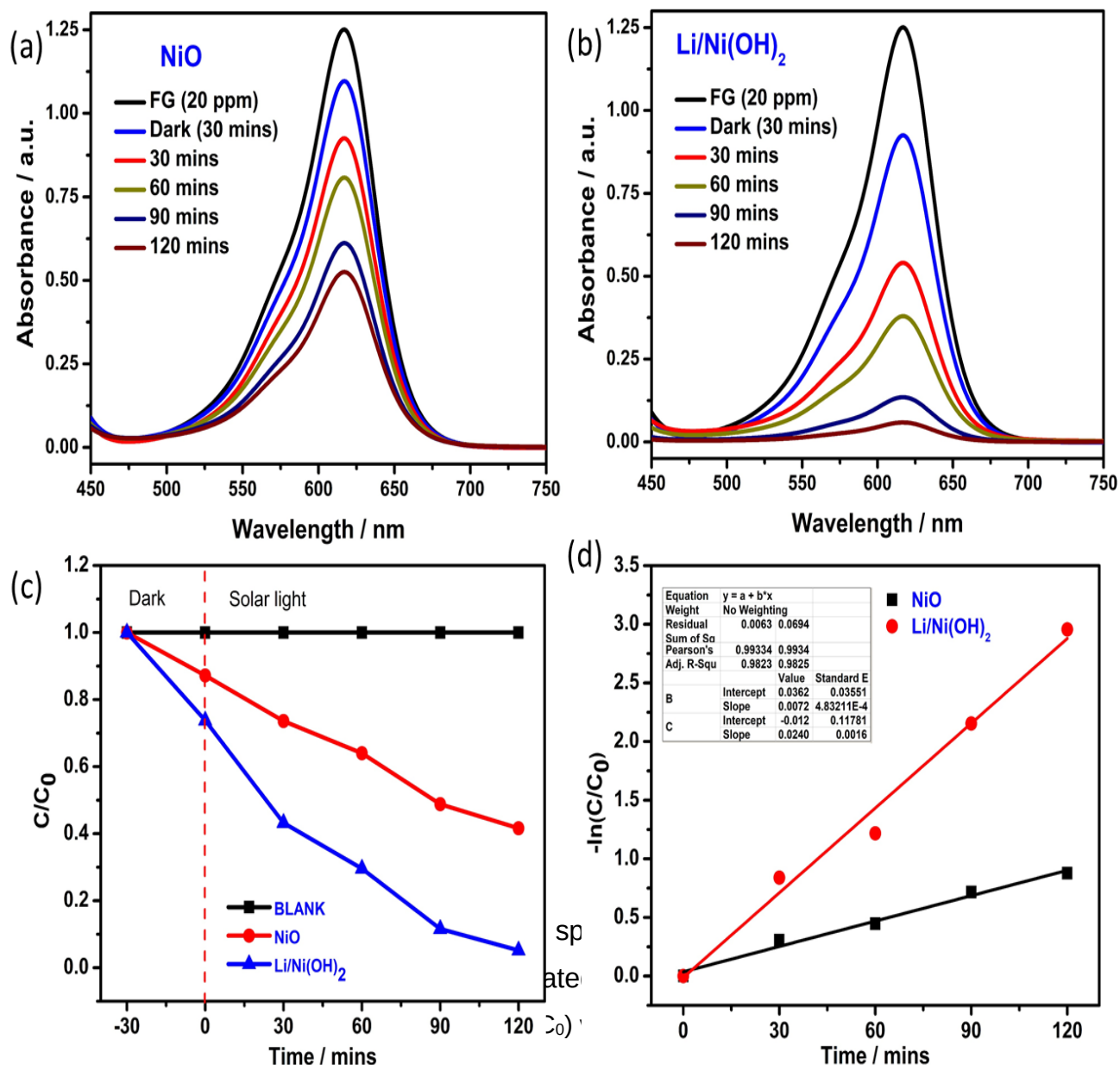
$$-\ln(C/C_0) = kt \quad (2)$$

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 $1.00 \times 10^5 \text{ lux}$.

Photocatalytic activity

The photocatalytic degradation efficiency of as prepared catalysts of NiO, and Li/Ni(OH)₂ nanoparticles were resolute by the deprivation of FG dye under irradiation of Sun light. When irradiating the FG 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 58.4% within 120 mins. The photocatalyst experiment was further extended to doped samples. The degradation reaction of $\text{Li}/\text{Ni}(\text{OH})_2$ sample was 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, $\text{Li}/\text{Ni}(\text{OH})_2$ catalysts reached high efficiency of degradation of 94.8% respectively [51].



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

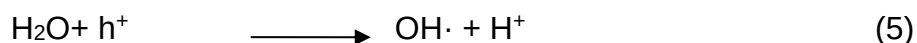
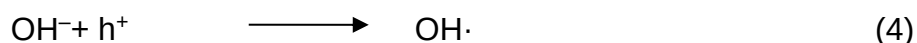
Figure.5.6c depicts the changes in initial and final concentration of dye in the presence and absence of photocatalyst against the light irradiation with respect to time (t) for the FG dye solution. The Li/Ni(OH)₂ catalyst shows highest degradation ability due the fact that sub energy-level act as traps 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.5.6d the degradation rates were 0.0112 min⁻¹ and 0.0375 min⁻¹ for NiO and Li/Ni (OH)₂ nanoparticles against FG dye, respectively. From the studies rate constant value and R² values are listed in the Table 1. The Figure 5.7 showed schematic diagram illustrating the mechanism of charge separation, generation of reactive oxygen species (ROS) and photocatalytic activity of photocatalyst under the sunlight irradiation [52].

The mechanism of FG dye degradation for the Li/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



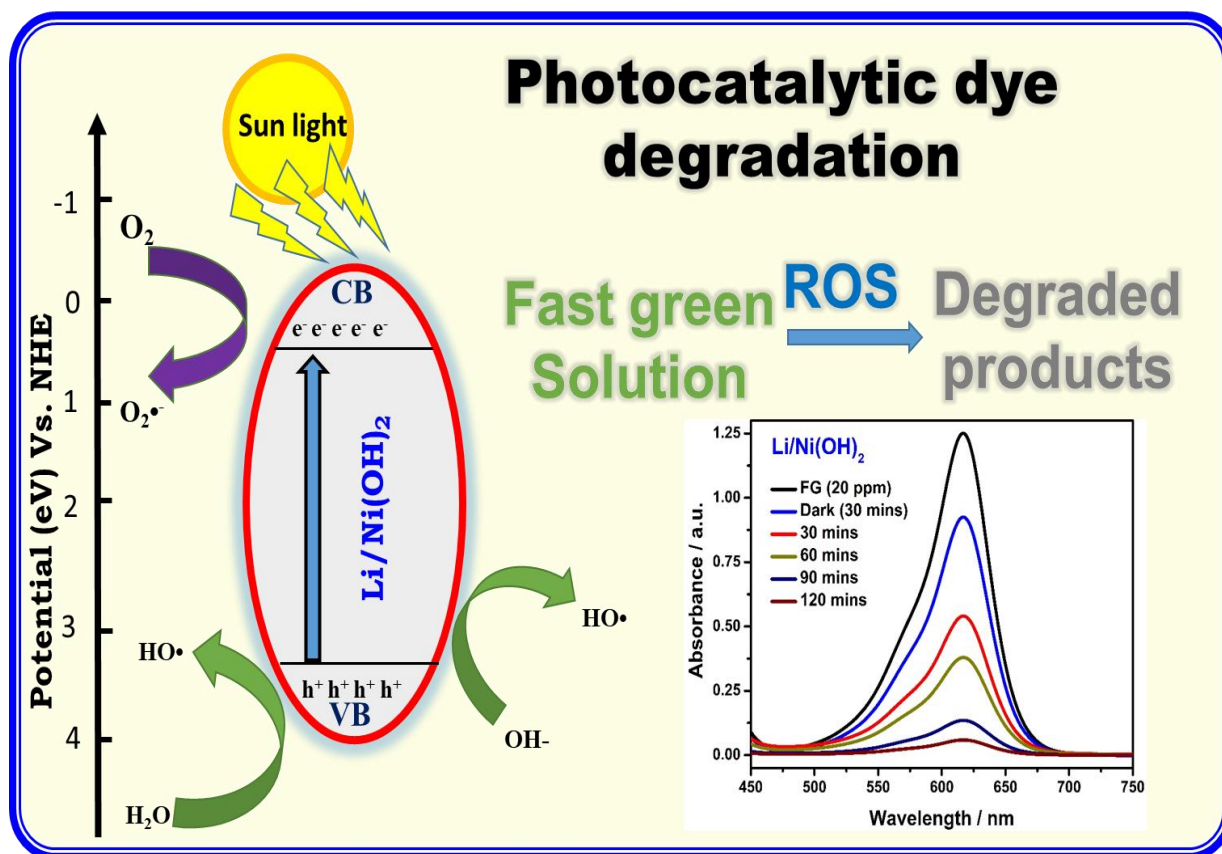


Figure. 5.7 Schematic diagram illustrating the mechanism of charge separation and photocatalytic activity of photocatalyst under the sunlight irradiation

Table 1 photocatalytic activity result of NiO and Li/Ni(OH)_2 nanoparticles.

S.No	Catalyst	Band gap (eV)	Degradation efficiency (%) (120 minutes)	K (min^{-1})	R ²
Fast Green (20 ppm)					
1	NiO	3.21	58.4	0.0165	0.9823
2	Li/Ni(OH)_2	2.82	94.8	0.0552	0.9825

CHAPTER-6

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 by 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 Li (5%) ion doped Ni $(\text{OH})_2$ 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 8.4 nm for NiO and Li 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 Li doped Ni(OH)_2 showed as excellent dye degradation of Fast Green (FG) 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 Li doped Ni(OH)_2 could be the suitable candidate for water purification, through the photocatalysis process.

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