Investigation of Synthesis, Characterization and Properties of nanosized Nickel doped Tin Oxide and its Applications

Dissertation in partial fulfillment of the requirements for the degree of

MASTER OF TECHNOLOGY

in

NANO-OPTOELECTRONICS

by

PRADEEP KUMAR YADAV

Under the Supervision

of

PROFESSOR KAMAN SINGH

Supervisor

BABASAHEB BHIMRAO AMBEDKAR UNIVERSITY

LUCKNOW

प्रज्ञा शील करुणा

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Department of Electronics & Communication Engineering

University Institute of Engineering and Technology

BABASAHEB BHIMRAO AMBEDKAR UNIVERSITY

(A Central University)

Lucknow-226025, Uttar Pradesh, India

CERTIFICATE

This is to certify that **Mr. PRADEEP KUMAR YADAV** has worked under our guidance and the report submitted in the project title "**Investigation of Synthesis, Characterization and Properties of Nanosized Nickel doped Tin Oxide and its Applications**" is suitable for the award of degree of Master of Technology.

B Yadav

(BAL CHANDRA YADAV)

Coordinator

M.Tech (Nano-OptoElectronics)

University Institute of Engineering and Technology

Babasaheb Bhimrao Ambedkar University,

Lucknow-226025

Kaman Singh

(PROFESSOR KAMAN SINGH)

Supervisor/Director 04/7/18

M.Tech (Nano-OptoElectronics)

University Institute of Engineering and Technology

Babasaheb Bhimrao Ambedkar University,

Lucknow-226025

DECLARATION

I hereby declare that this project report entitled "**Investigation of Synthesis, Characterization and Properties of nanosized Nickel doped Tin Oxide and its Applications**" is an authentic work carried out by me during the session 2016-18, under the supervision of Prof. Kaman Singh, University Institute of Engineering and Technology, Babasaheb Bhimrao Ambedkar University, Lucknow, Uttar Pradesh, India. This dissertation is submitted for the partial fulfillment of the requirement for the award of the degree of Master of Technology in Nano-Optoelectronics.

(PRADEEP KUMAR YADAV)

M.Tech (IV Semester)

Roll No. 116032

Babasaheb Bhimrao Ambedkar University

Lucknow-226025, Uttar Pradesh, India

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PRADEEP KUMAR YADAV

M.Tech (IV Semester)

PREFACE

The design of experiments, data analysis and writing of this thesis was done under the supervision of Professor Kaman Singh. Exclusive the following data collection steps all the other data collection and Master of Technology analysis was performed by the author.

- SEM, EDX and FTIR data was taken by the help of members of USIC, BBAU.
- UV-Vis, BET and ZPC data was collected by the help of Ashok Kumar, Department of Applied Chemistry.
- XRD data was collected by the help of members of ACMS, IIT Kanpur.
- The adsorption of Methyl Orange data was gathered by Ashok Kumar, Ph.D Scholar at Department of Applied Chemistry, BBAU.
- The application data of Opto-Electronic Humidity Sensing was collected by the help of Professor B.C. Yadav and Ekta Singh.

DEDICATED

TO MY

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ABSTRACT

Present work deals with the synthesis, characterizations and applications as adsorbate & optical humidity sensing of nickel doped tin oxide. Nickel doped tin oxide were synthesized by using chemical precipitation method. By using different capping agent and size of nanoparticles can be controlled by PEG. The structure and morphology of these nanoparticles were investigated by SEM. The average sizes of nickel doped tin nanoparticles were found to be 15.182 nm by BET analysis and 21 nm by zeta seizer. The band gap energy was 4.28 eV found by UV-visible spectroscopy. The thin film of nickel doped tin oxide nano was fabricated on glass by spin coating techniques. The nickel doped tin oxide exhibit good sensitivity towards moisture and also has good adsorption of methyl orange. The average moisture sensitivity was found to be 0.66 µW / %RH.

CONTENT

LIST OF FIGURES

LIST OF TABLE

CHAPTER

1

1. INTRODUCTION

1.1 Nanotechnology And Nanoscience

Nanotechnology is made by two words "Nano" and "Technology". Nano is the simple word which means something small. The use of nano in the field of science is called nanotechnology. In those days nanotechnology use in each area of science [1]. Nanotechnology is the art and involves the manipulation of atoms and molecules for developing the materials, which having dimensions in the range of nanometers (1-100 nm), which are synthesized by two approaches: (a) Top-down and (b) Bottom-up. The top-down approach involves the breakdown of bulk materials up to the nanoscale to obtain the nanoparticles, and bottom-up approach involves the gather of the atoms or molecules of bulk material for the fabrication of nanoparticles [2]. Nanotechnology and Nanoscience started by physicist R. Feynman at American Physical Society meeting at California Institute of Technology on 29th December, 1959. In this meeting R. Feynman describes the process to manipulate and control the atoms and molecules. In last 10 years, nanomaterials in the range of 1-20nm become most important area of research interest and small size has potential for the industries, biomedical and electronic applications [3].

This is revolutionary in several fields like material science, optoelectronics, medical, industries etc. Nanotechnology improving the mechanical, physical, chemical and optical properties of material and devices. In this first guideline developed by K.E. Drexler in his book "Engines of Creation". The United State National Human Genome Research Institute proposed a proposal to develop a new technology [4].

The nanomaterials offer unique properties. In sample, atoms building the blocks and measured using nanoscale. Nanosized particles allow to interaction between molecules by increasing the efficiency and attraction to the bulk sized particles. High surface to volume ratio offer unique physical property of nanomaterials, which means that large number of atoms present on the surface than its volume. This is useful for the surface atom, which has unbound surfaces than its volume atoms, with potential to forming strong bond and more reactive than the bulk materials. Nanomaterials are easily arranged in comparison to the bulk material in packing configuration due to its high surface to volume ratio, which easily manipulated and utilized in many fields [4].

Now days, Nanotechnology based approaches have attracted global attention for detection, degradation and removal of hazardous pesticides. There different type of nanomaterials, nanoparticles, nanotubes, nanorods, nanoblades and nanocomposite are used in the different areas through myriad methodologies. The nanoscale size, high surface to volume ratio, physicochemical properties and high target specificity of the material is help in highly sensing of the pesticides [2].

Here Society is the user, funding party and decision maker, attitude of society toward nanotechnology plays a important role in victory and dissappoinment, in different words society is judge and jury. This is ambition through ethics, moral and values. Nanotechnology is currently integrated in fields that directly affect to the public like energy supply, health care, telecommunications and pollution control, with various advantages like low cost, easy to use and ecofriendly [4].

Properties of nanomaterials are special and incredible, to their available equivalents in polycrystalline, which is considered from the chemical composition, grain size, atomic structure, crystallographic orientation, coordination number and dimensionality. It acquire the area of synthesis, characterizations and applications of nanomaterials. Nanomaterials, by good quality of their small size and large surface to volume ratio, also shows great novel properties which include optical behavior, increased mechanical strength, high thermal strength, magnetic behavior and electrical resistivity.

Nanomaterials classified on the basis of their properties:

- Chemical Properties
- Biological Properties
- Optical Properties
- Mechanical Properties

1.2 Optoelectronics

Optical technologies are the future oriented technologies of 21st century. This is the study and application of electronic devices and system which has source, detector and light controller, generally considered as subfield of photonics. In this light include radiations such as gamma rays, X-rays, UV-rays, IR in invisible form to visible form. These devices are electrical to optical or optical to electrical transducer that uses such devices in their operation. Optoelectronics based on quantum mechanical effect of light on electronic materials in the presens of electric field.

Optoelectronics devices are devices that work on light and also on electrical currents. This can be included electrically determined by light sources like LD, LED, and components for converting light to electrical current such as solar cells, photovoltaic cells and devices that can electronically control the propagation of light.

1.3 SEMICONDUCTOR

Semiconductor material has an electrical conductivity between conductor and insulator. Semiconductor material is a material which occupied high energy band, also valence band is completely filled by the electrons and next one is the conduction band which is empty. The resistivity of the semiconductor can be changed by orders of magnitude (up to 10) and also by doping or external biases. If we talk about conductors, have very low resistivity, resistance is very difficult to change, and maximum disposed energy band is partially filled by the electrons and insulator has unconscionable high resistivity. Doping or external fields are difficultly change the resistivity and wide band gap is present between the valence and conduction band. In metal conductors, current passed through the flow of electrons and in semiconductors, current can be passed either by the flow of electron or by the flow of holes in electron structure of the material [3].

Recently there has been enough interest in the synthesis, Characterization, properties and application of semiconductor nanomaterials that plays very important role in various advance technologies. When the size of the semiconductor materials is decrease to nano range, their physical, chemical and mechanical properties would be change radically, resulting in unique properties due to their large surface area. The conductivity and optical properties (absorption coefficient and refractive index) of semiconductor materials can be altered. Semiconductor materials and devices in nano range are still under research but it promising for wide applications in different fields, such as electronic devices, solar cells, LEDs, Laser technologies, sensors, food technologies, automobiles, catalysts, electrodes and many more [3, 5, 6,]. Some of the semiconductor materials are Silicon, Germanium, Gallium Arsenide, Aluminum Gallium Arsenide, Indium Phosphate, Indium Gallium Arsenide, Zinc Sulfate, Tin, and Antimony etc., reveal excellent application in the field of computers, laptops, mobiles, televisions, remotes, satellite, optical fibers etc. Most of the semiconducting materials, such as II-VI or III-VI compound show quantum confinement behavior [3].

Semiconductor nanoparticles are made from array of different compounds. They are referred to as II-VI, III-V or IV-VI semiconductor nanoparticles on periodic table into which these elements are formed such as silicon, germanium, gallium arsenide, indium phosphate, Zinc, Tin, Titanium and many more elements are present [3].

In nanomaterials, electrons are confined from all three dimensions when relative dimension is equivalent to the de Broglie wavelength. The nanosized semiconductor having z direction below critical value (thin film, layer structure, quantum well) are defined as two dimensional nanostructure. When dimension of nanosized semiconductor in x and z direction is below critical value (linear chain structure, quantum wire) are defined as one dimensional nanostructure and when y direction is also below threshold (cluster, colloid, nanocrystal, quantum dot) is defined as zero dimensional nanostructured [3].

1.4 SYNTHESIS MATERIAL

Tin Oxide belongs to organic compound group and the mineral form of tin oxide is known as Cassiterite. Tin oxide is colorless with diamagnetic and amphoteric solid material. Commonly tin oxide has tetragonal structure in which have six co-ordinate of tin atom and three co-ordinate of oxygen atom [5]. Tin have two oxidation states +2 and +4, whom two oxidization stannous oxide (SnO) and stannic oxide (SnO_2) are possible. Stannic oxide is also known as tin oxide. Tin oxide is more stable than the SnO [7] and tin oxide is n-type semiconducting material. Researchers shows their interest on tin oxide due to their wide band gap energy (E_g) varying from 3.6eV-3.8eV at different temperature[5, 6, 7] and have unique properties like highly transparency in visible spectra, operate at low temperature, high thermal stability, high sensitivity, strong chemical and physical interaction etc. in low cost[6, 8].

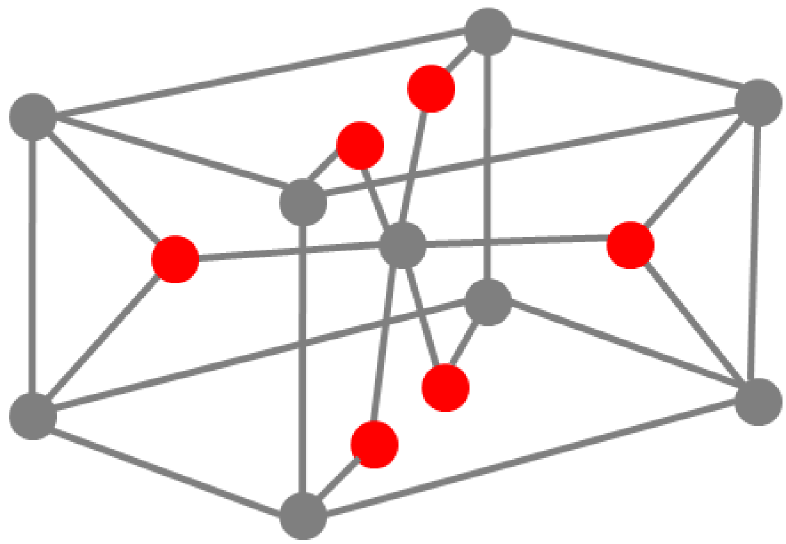


Figure 1: Structure of Tin Oxide

Table 1: Properties of tin oxide

S. No.	Properties	Values / Specification
1.	IUPAC Name	Tin (IV) Oxide
2.	Other Name	Stannic Oxide, Flowers of tin, Cassiterite
3.	Chemical Formula	SnO_2
4.	Molar Mass	150.71 $gmol^{-1}$
5.	Appearance	White or light grey powder
6.	Odor	Odorless
7.	Density	6.95 g/cm^3 (at 20°C) or 6.85 g/cm^3 (24°C)
8.	Melting Point	1630°C
9.	Boiling Point	1800 to 1900°C
10.	Solubility	Hot Concentrated alkalis, Concentrated acids
11.	Magnetic Susceptibility	-4.1×10^{-5} cm^3/mole
12.	Crystal Structure	Rutile Tetragonal
13.	Optical Band Gap Energy	3.4 to 3.6 eV

Nickel Oxide belongs to chemical compound and the mineral form of nickel oxide is known as bunsenite, which is very rare. Nickel oxide is regarded as p-type semiconductor with wide band gap [5].

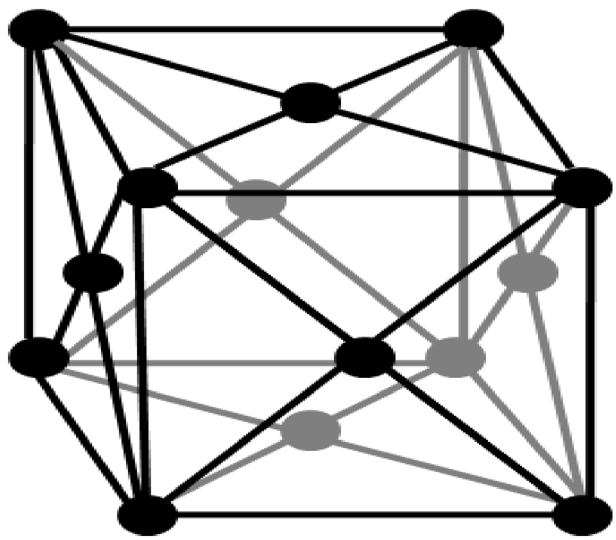


Figure 2: Structure of Nickel Oxide

Table 2: Properties of nickel oxide

Sn. No.	Properties	Values / Specifications
1.	IUPAC Name	Nickel (II) Oxide
2.	Other Name	Nickel Mono-Oxide, Oxonickel
3.	Chemical Formula	NiO
4.	Molar Mass	74.69 g/mol
5.	Appearance	Green crystalline solid
6.	Odor	Odorless
7.	Density	6.67 g/cm^3
8.	Melting Point	1955^{o}C
9.	Flash Point	Non-Flammable
10.	Solubility	KCN
11.	Magnetic Susceptibility	+ 660 x 10^{-6} cm^3/mol
12.	Crystal Structure	Monoclinic
13.	Optical Band Gap Energy	Approx 3.6 eV

Many doping process are developed using n-type semiconductors like zinc oxide, tin oxide, titanium dioxide etc. and p-n type semiconductors are developed by using nickel, copper, ferrite, silver, antimony etc[8]. There present many materials to doped in tin oxide but nickel has extra motivating significant like electronics, optical, magnetic, electrochemical etc. The introduced nickel can be easily conjunct with tin oxide and form p-n junction because nickel is p-type material. After mixing metal-oxides, shows unique physical and chemical properties, which used as sensors, solar cells, batteries, catalysts, electrodes etc[5].

1.5 SYNTHESIS TECHNIQUES

1.5.1 Two major methods to synthesis nanomaterials

The nanomaterials grasp subfields which would study materials having important properties from their nanoscale dimensions. Nanoelectronics are relevant with fast ion transport of nanomaterials. Innovation in application organizes semiconductor nanomaterials to be used in 4^{th} generation products such as lighting, solar cells and biological imaging.

1.5.1.1 Top-Down Method

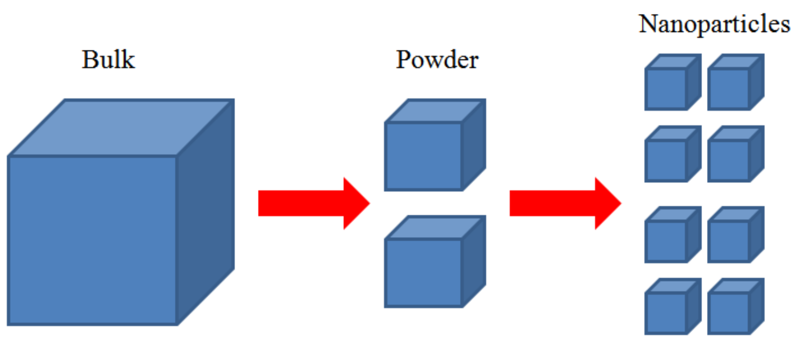


Figure 3: fabrication process of Top-Down Method

Figure 3 shows the fabrication process in top-down method is succeed by reducing the size of bulk material into nanoscale range. This method links the assembling of parts over some methods such as cutting, molding etc. This method is preferred for design the variety of machinery and some electronic devices.

- Ball Milling

- Chemical Etching
- Laser Ablation
- Sputtering

1.5.1.2 Bottom-Up Method

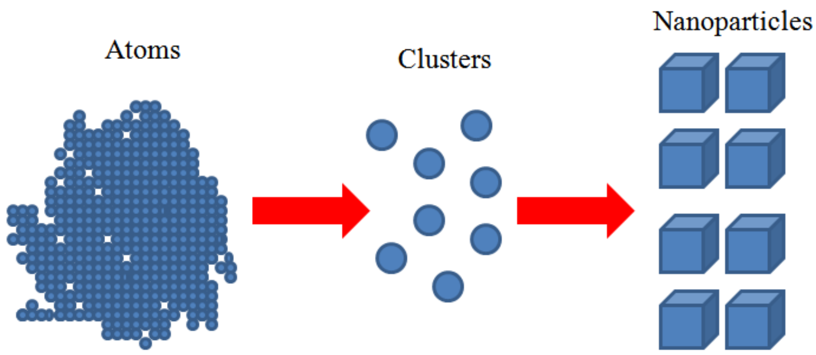


Figure 4: Fabrication process of Bottom-Up Method

Bottom-up method provides better structure by accumulating the atom with atom and molecule with molecule as shown in Figure 4. The mechanisms of molecules are filled simultaneously by covalent force, which is stronger than the forces performing on macro-scale components. Because of this, the quality of information has been stored in devices which made by bottom-up method which are as follows:

- Chemical Synthesis
- Self-assembly
- Positional assembly

1.6 SENSORS

The word "Sensor" derived from the Latin word "Sentire", which means that "to preserve". Sensor is a device that responds to the physical and chemical stimulate such as heat, light, sound, pressure and many more, that transmit the variable impulse. Sensors can detect an input signal and convert into an appropriate output signal. Interaction between prepared film and test gas is dependent upon the number of nanostructure and composition variables which are thickness of prepared film, crystalline size, porosity, surface, as well as content of doped element [9]. Sensors can be classified on the basis of principle as physical sensors, chemical sensors, optical sensors etc.

1.6.1 Opto-Electronic Humidity Sensor

Opto-Electronics sensors are the systems that use optical technology to transport a light as a input signal that modulated from the light source according to the magnitude of the measured object and collect the output through the detector [10, 11]. The origin of the opto-electronics sensors with the invention of the LASER in 1960. In 1963 observed that the optical signals could be transmitted from the glass with slighter loss that of copper cables [12].

The sensitivity of the sensor element is defined as the slope of the curve between percentage of Relative Humidity (%RH) and output power [11]. The formula for calculation of sensitivity is:

$$Sensitivity = \frac{Change\ in\ input\ power}{Change\ in\ \%RH\ values}$$

Where RH = dimensionless quantity

CHAPTER

2

2. EXPERIMENTAL TECHNIQUES

2.1 SYNTHESIS PROCESS

Nickel doped tin oxide nanomaterials were successfully synthesis by using Chemical Precipitation method and using capping agent for reducing size and Sonication. So here also using similar process, first of all we dissolve 0.1M of tin chloride ($SnCl_2.2H_2O$) and 2wt% of nickel chloride ($NiCl_2.6H_2O$) in distilled water and Stirring for an hour, after that start doping nickel in the tin oxide during stirring process which is done in approx 2hrs. After finishing the doping of nickel, adding the capping agent into the solution, here I am using PEG (Polyethylene Glycol). Adding 10-12 drops of PEG, which is use to reduce the size of the prepared sample. Then adding sufficient amount of ammonia to the solution during stirring and dropping rate of PEG and Ammonia must be well controlled. The resulting precipitate were collected, washed with distilled water, and then dried in oven at 100°C for an hour, after that put on furnace for 2h at 350°C, and finally got nickel doped tin oxide nanosized particles.

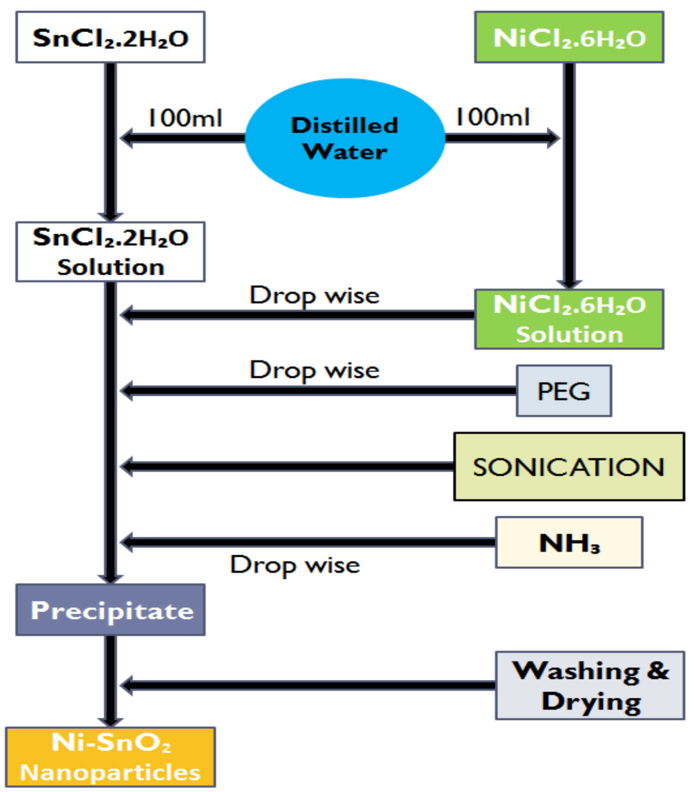


Figure 5: Flow chart of synthesis Nickel doped tin oxide

2.2 CHARACTERIZATIONS TOOLS

2.2.1 Scanning Electron Microscope

For morphological study using JSM-6490LV, JEOL, Japan Scanning Electron Microscope, which gives black & white color and 3D image. The prepared sample mounted on carbon stab with the help of double sided tape. Mounted stabs were coated by palladium to analysis using a Polaron sputter coater [13].

Figure 6: SEM Setup

2.2.2 Energy Dispersive X-Ray Spectroscopy

EDX is use for the confirmation of the presence of the chemicals in the prepared sample. EDX is attached with the SEM. High energetic backscattered electron (BSE) from the sample was filtered by using traps of magnetic electron at front of EDX detector. BSE are filtered by using filter foils which prevent to electron when transmitting the X-ray photons. The use of foils, constant composed X-ray sensitivity losing to carbon and boron can be achieved.

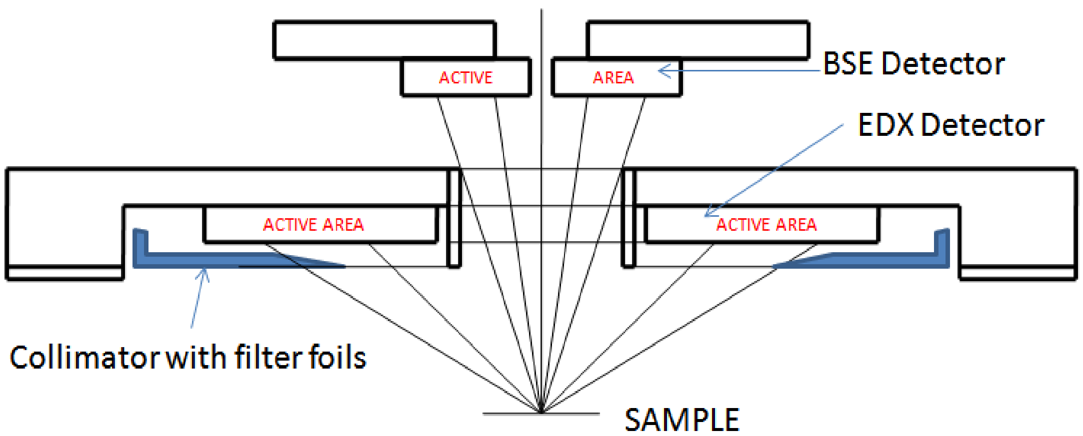


Figure 7: Energy Dispersive X-ray Spectroscopy

2.2.3 X-ray Diffraction

Structural characterization of prepared sample was obtained by the PANalytical X'Pert Powder X-ray Diffraction from ACMS, IIT Kanpur. XRD is one of the most non-destructive tools to analyze all kinds of matter ragging from fluids, to powders and crystals. It is the indispensible method for characterization of structural materials and quality control which make useful to Debye-Scherrer formula. This characterization technique uses X-ray by diffraction on powder or samples, where every possible crystalline course is represented equally.

This method gives ability to easily analyze the unknown materials and characterizations in the field of metallurgy, mineralogy, archeology, biology, pharmaceutical and forensic sciences.

Powder X-ray diffraction patterns were recorded on a Rigaku Miniflex II desk top X-ray diffractometer at 30kV and 15mA using CuK radiation [13].

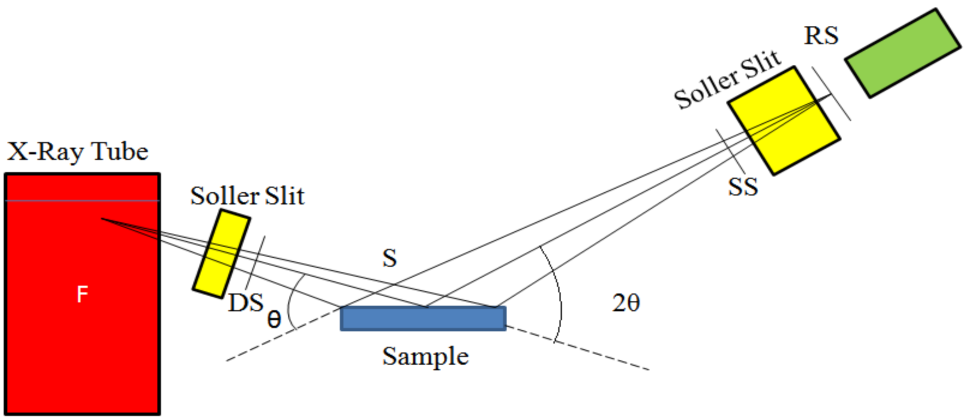


Figure 8: Setup of XRD

2.2.4 Brunaur-Emmett-Teller (BET)

The surface analysis, size and pore volume of materials was performed by the surface area analyzer BEL Sorp-mini, Japan. A definite amount of the synthesized materials nickel doped tin oxide (actual amount = 0.2782 gm) were heated at 400°C under vacuum and nitrogen flow approximate for 5-6 hrs. After the pretreatment of the material, the process of nitrogen adsorption and desorption were performed. The surface and pore volume were calculated using BET equation and pore size was determined by employing a method BJH (Barrett-Joyner-Halenda) [13].

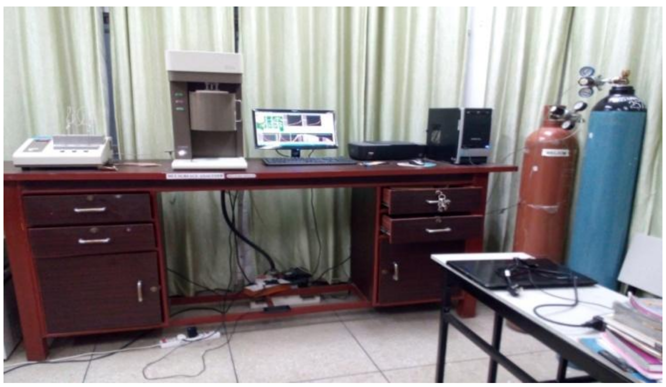


Figure 9: BET surface analysis setup

2.2.5 UV-Visible Spectrophotometer

Absorbance spectra recoded by Agilent Technologies, Cary Series UV-Vis Spectrophotometer, which seen in Figure 10.

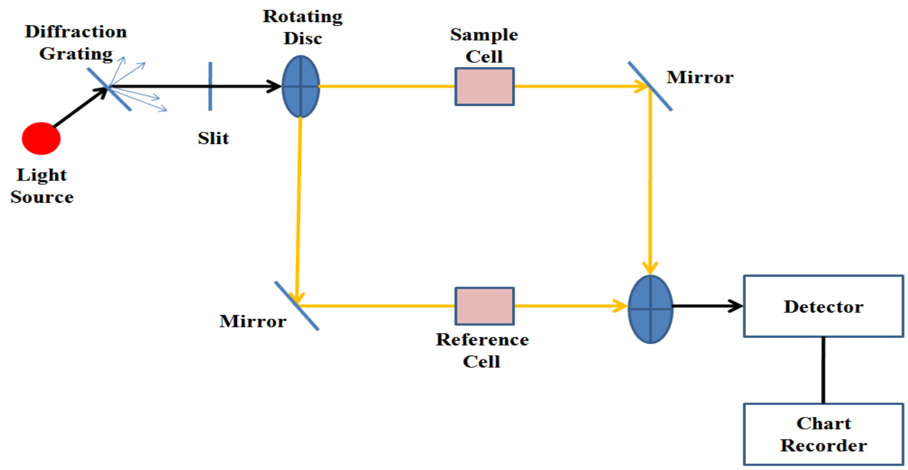


Figure 10: UV Visible Spectroscopy setup

2.2.6 Fourier Transform Infrared Spectroscopy

For infrared analysis using Nicolet™6700, Thermo Scientific, U.S.A. Fourier Transform Infrared Spectrometer. The prepared sample was mixed with KBr and formed palette. KBr is use to reduce moisture present in prepared sample. The IR spectra of the pellet were recorded [13].

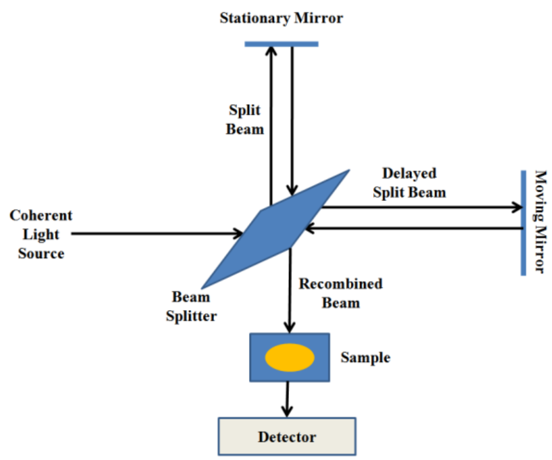


Figure 11: FTIR Setup

2.2.7 Zero Point Charge (ZPC)

The ZPC characteristic of nickel doped tin oxide was determined by solid addition method using 0.1M KCl and 0.02M citramide solution. 40ml of KCl and citramide solution was prepared in 100ml stoppered conical flask. The initial pH value of the solutions were adjust between 2.0 to 12.0 by adding either 0.1N HCl or 0.1N NaOH. The total volume of the solution was adjust to 40ml by adding 20ml of KCl and 20ml of citramide solution. 0.1 gm of nickel doped tin oxide was added to each flask. The suspensions were shaked and allow equilibrating for a day. The final pH values of the suspernatant liquid were noted [13]. The ZPC value determined by using following formula:

ZPC = (Initial pH of Solution) – (Final pH of the solution)

CHAPTER

3

3. RESULTS AND DISCUSSION

3.1 Scanning Electron Microscope

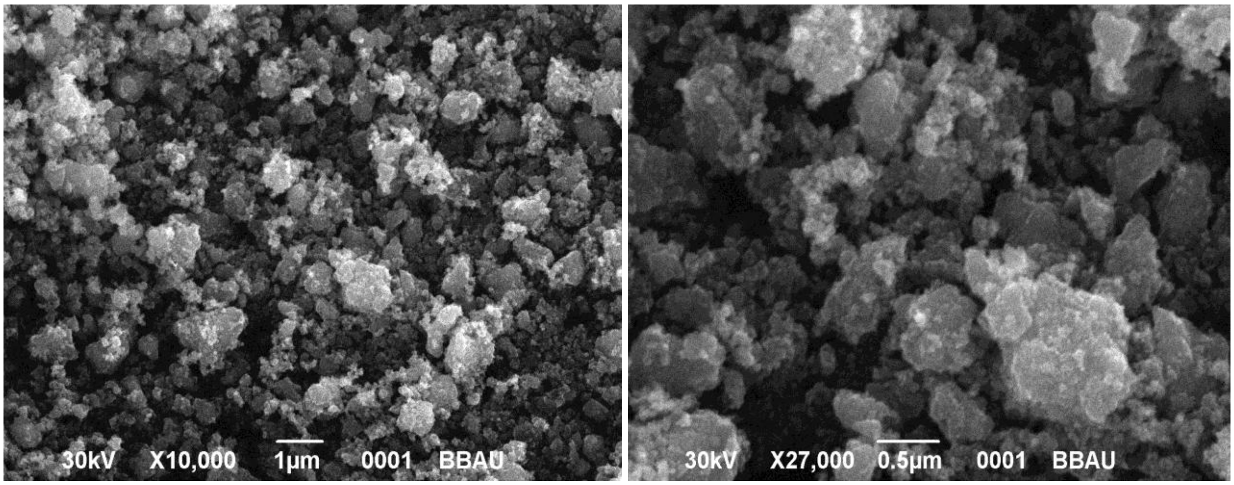


Figure 12: Morphological study of Nickel doped Tin Oxide

Nickel doped tin oxides are successfully synthesized by Chemical Precipitation Method and Morphology of prepared nanostructured samples are characterized by Scanning Electron Microscope (SEM), which we can see in Figure 12. After characterizing the nickel doped tin oxide nanoparticles found high porosity, so these types of nanoparticles are used in different application such as sensors, solar cells. The morphology of prepared sample contain two type of chemical structure because of doping of nickel, so we can see in Figure 12 particles are agglomerated and in such case cluster is found.

3.2 Energy Dispersive X-ray Spectroscopy

After the study of morphology of prepared sample, go for checking the purity and contents materials in the prepared sample, which analyzed by the EDX and it give peaks, these peaks define the presens and amount of materials present in the prepared sample. On analyzing the EDX data of prepared sample, found that one large and one small peak of Sn nanoparticles, three small peaks of Ni nanoparticles and single peak of O and C. We can observed that in Figure 13 present desired contents Sn, Ni and O but C is also got which shows the Carbon. Here carbon content is present in prepared sample is mounded on Carbon before the SEM Characterization.

Table 3 Percentage of present chemicals compound in prepared sample

Element	Weight%	Atomic%
C K	26.11	48.55
O K	30.95	43.20
Ni K	0.91	0.35
Sn L	42.03	7.91
Totals	100.00	

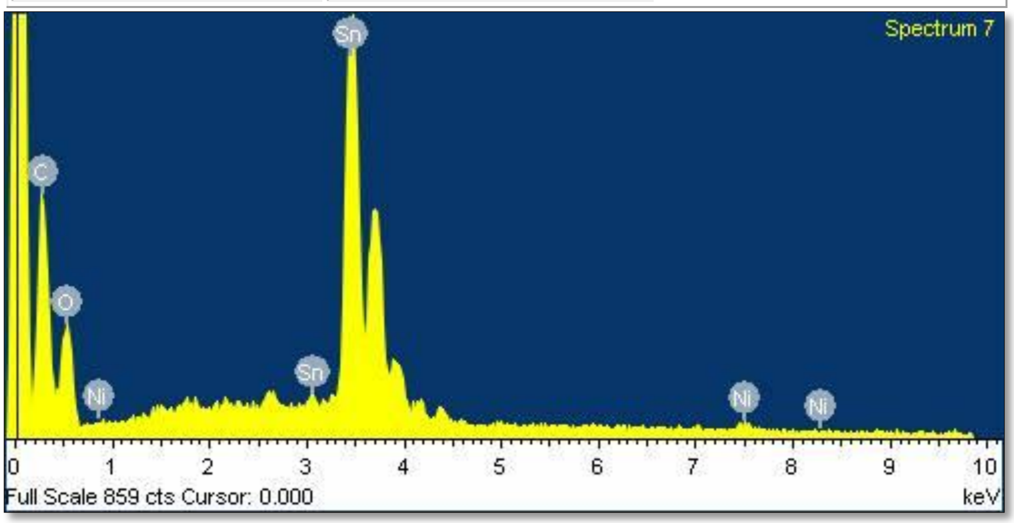


Figure 13: Chemical composition in prepared sample

3.3 X-ray Diffraction

To study the crystalline structure and size of the prepared two materials was examined by XRD in powder form. Figure 14 shows the pattern and result of the prepared sample P1 and P2, which annealed at 400^{o}C. XRD using CuKa as source, which gives highest peak at 26.55^{o} at plane (110). The crystalline size of the prepared samples was calculated by Debye-Scherrer's formula is given below;

$$D = \frac{n\lambda}{\beta Cos\theta}$$

By this formula got that 21nm size of sample Prepared sample. No reflection peaks of impurities, which indicate that samples have high purity.

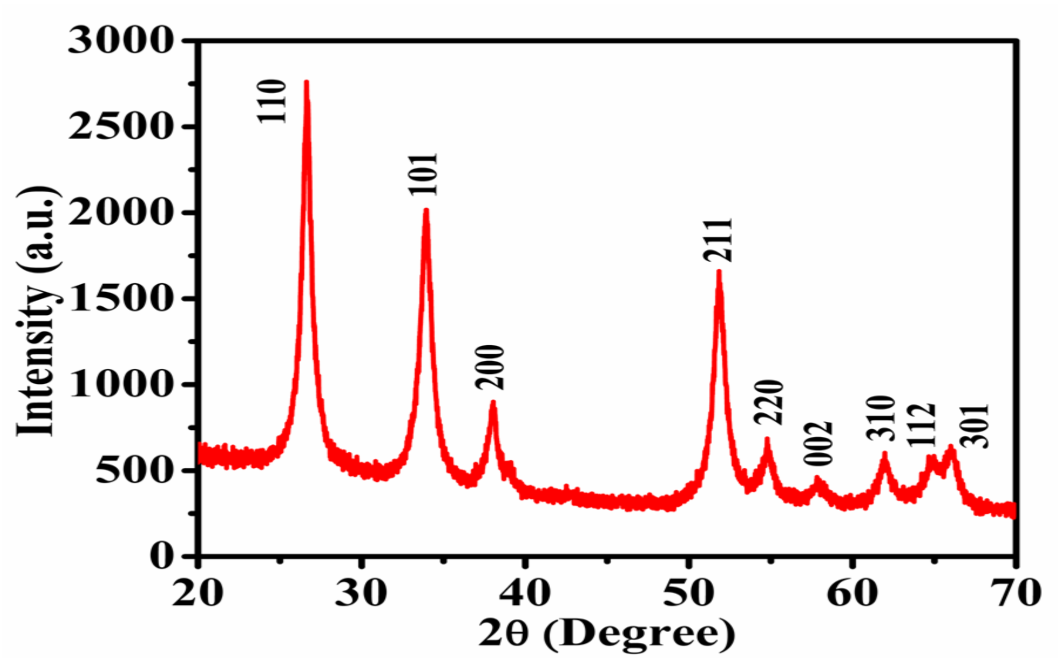


Figure 14: XRD graph of Nickel doped tin oxide

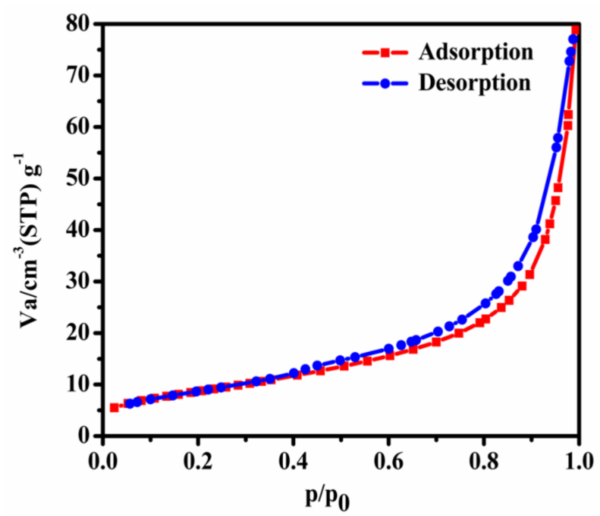
3.4 BET Surface Area

BET analysis confirms that the nickel doped tin oxide had a specific surface area 30.74 nm^2/gm. The average pore diameter of both samples was found to be 15.182 nm, which is an indicative of its mesopores character as defined by Dabrowskye. According to him pore diameter is ranges as follows:

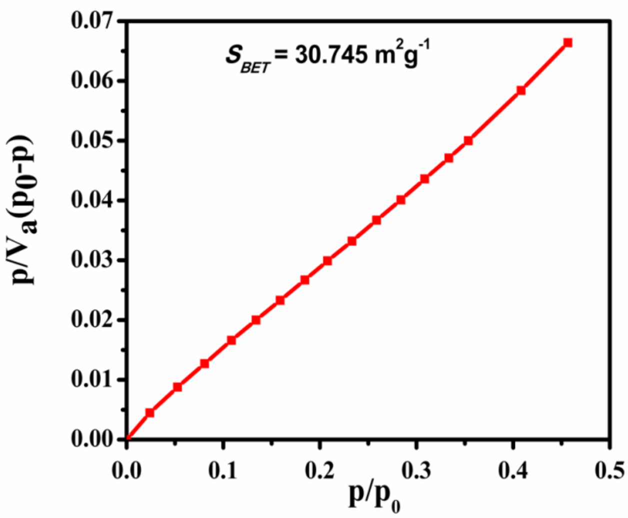
- Micropores; < 2nm
- Mesopores; 2 to 50nm
- Macropores; > 50nm

Nitrogen adsorption-desorption isotherm of nickel doped tin oxide at 77ᵒK was recorded (Figure 15a). The hysteresis that appears as the difference in the two curve at P/P_0 at approx 0.1167 cm^3g^{-1} is due to nitrogen condensation, a typical phenomenon shown by mesopores material (Figure 15b).

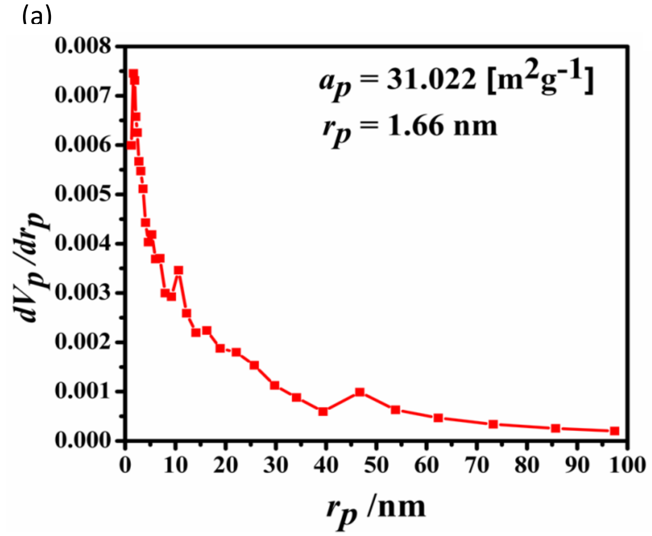
The pore size distribution of these particle shown in BJH plot (Figure 15c), which clearly shows that mesopores nature of the material.



(a)



(b)



(c)

Figure 15: BET Surface analysis; (a) Adsorption Desorption, (b) BET graph, and (c) BJH graph

3.5 UV-Visible Spectroscopy

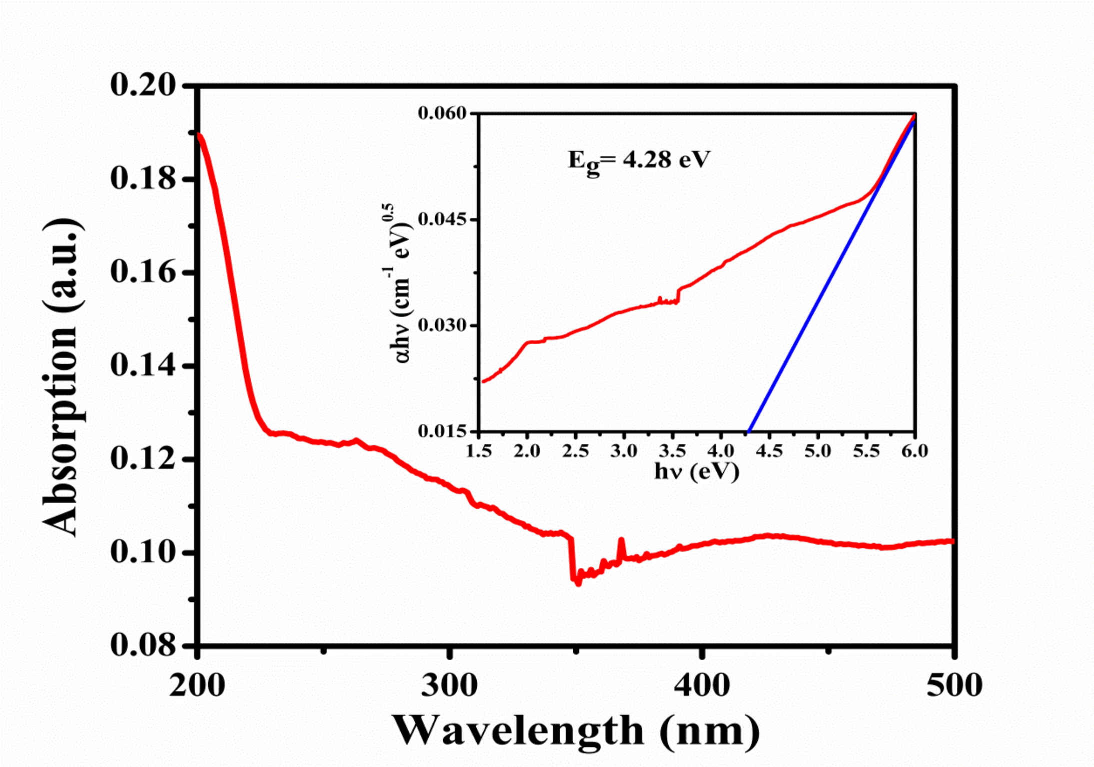


Figure 16: Measurement of Optical property of nickel doped tin oxide

UV-Visible spectroscopy refers to absorption spectroscopy in UV-Visible spectral region. It measures the radiation percentage which absorbed at each wavelength. This is done by recording the absorbance and wavelength. This is widely used for the study of optical band gap of the semiconductor material Figure 16 shows the UV-Visible absorption spectra of nickel doped tin oxide with PEG. The absorption wavelength for nickel doped tin oxide was found to be at 200nm and corresponding band gap energy was 4.28eV.

3.6 Fourier Transform Infrared Spectroscopy

In the present thesis, FTIR analysis was performed to identify the presence of certain functional groups in the LZF nanoparticles and confirm the purity of it. Hence, the FTIR spectrum of the sample was recorded in the range of 450-4000 cm^{-1}. The weak absorption bond observed at 1025 and 1330 $cm^{-1}$is due to the bending vibration of absorbed water and the absorption bond at 672 cm^{-1} is because of the O-H stretching mode. However, be main band that characterizes the formation of spinal phase is located at 3385 cm^{-1}. Which are associated with the vibrations of Ni-O and Sn-O bonds, respectively.

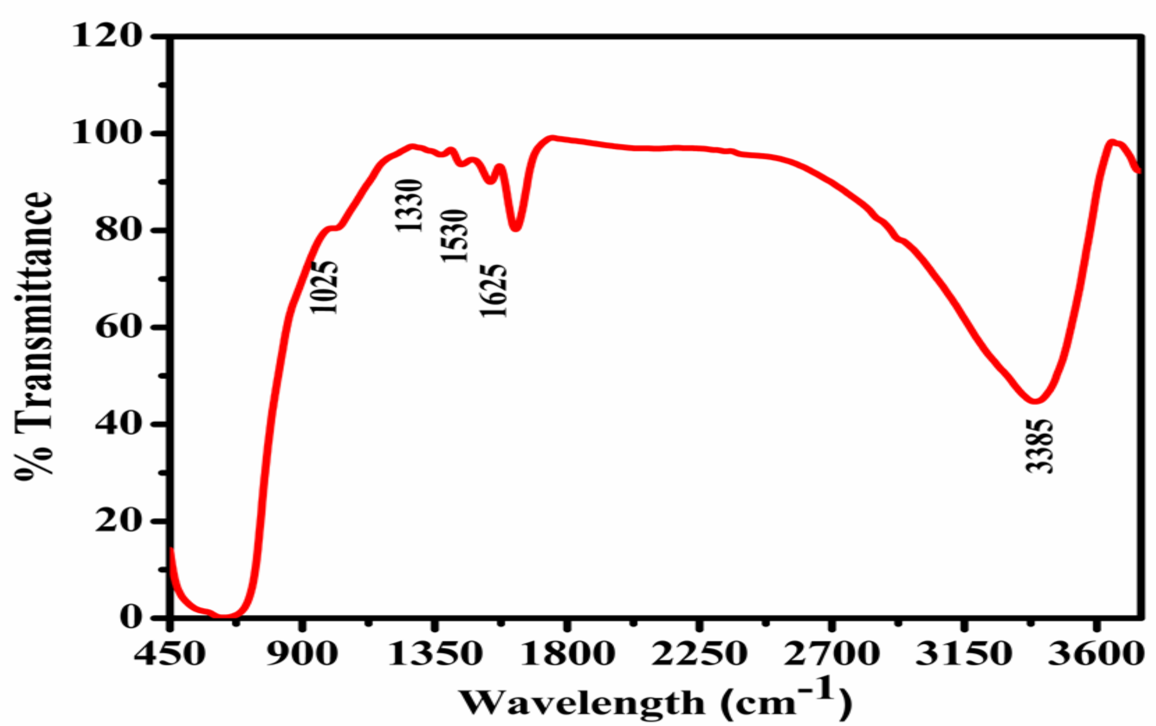


Figure 17: FTIR spectra of prepared sample

3.7 Zero Point Charge

The ZPC characteristic of nickel doped tin oxide was determined by solid addition method using 0.1M KCl and 0.02M citramide solution. 40ml of KCl and citramide solution was prepared in 100ml stoppered conical flask. The initial pH value of the solutions were adjust between 2.0 to 12.0 by adding either 0.1N HCl or 0.1N NaOH. The total volume of the solution was adjust to 40ml by adding 20ml of KCl and 20ml of citramide solution. 0.1 gm of nickel doped tin oxide was added to each flask. The suspensions were shaked and allow equilibrating for a day. The final pH values of the suspernatant liquid were noted [14]. The ZPC value determined by using following formula:

ZPC = (Initial pH of Solution) – (Final pH of the solution)

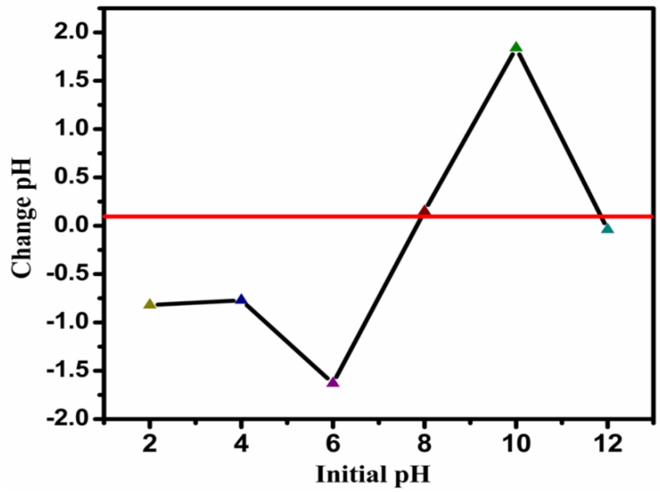


Figure 18: Zero point charge of prepared sample

CHAPTER

4

4. APPLICATIONS

4.1 Optoelectronics Humidity Sensing

4.1.1 Limitation

This is important to understand the sensing limitations for good fabrication of a sensor. Which are as follows:

- Sensitivity
- Reproducibility
- Hysteresis
- Operating temperature
- Response and recovery time

4.1.2 Humidity Sensing

Figure 19 sows the experimental setup for the optoelectronics humidity sensing [11]. The prepared thin film of nickel doped tin oxide placed inside the glass chamber. Put saturated solution of potassium sulphate (K_2SO_4) to increase the humidity continuously inside the chamber, which increases %RH from 10 to 100%. However to decrease the humidity continuously inside the chamber by using saturated solution of potassium hydroxide (KOH), which decreases %RH from 100 to 10%. 2 mW and 632 nm wavelength He-Ne laser was used as a light source.

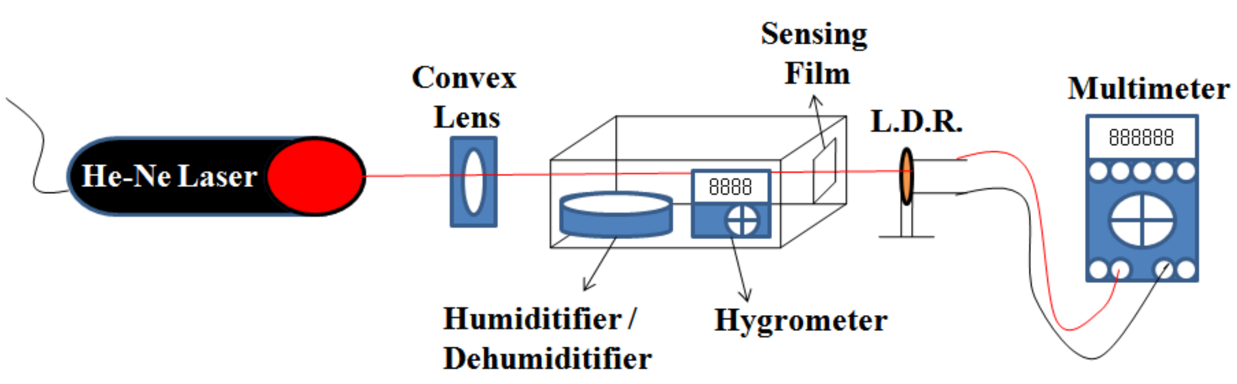


Figure 19: Setup of Opto-Electronics Humidity Sensing

Opto-Electronics humidity sensing of nickel doped tin oxide film was prepared by using spin coater variation in output power with %RH were recorded by multimeter and plotted as Figure 20.

The change in the power of sensing materials depend upon the number of pores exist in the material. Nickel doped tin oxide at all concentration shows a linear trend with increase in power with increase in %RH value. Nickel doped tin oxide showed good humidity sensitivity ability.

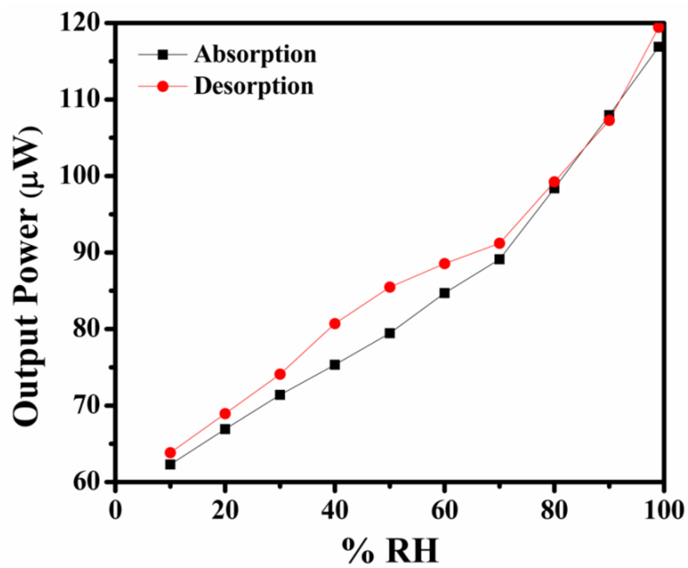


Figure 20: Humidity Sensing graph

The variation of output power (μW) of LASER light source show % relative humidity through the film of nickel doped tin oxide was observed and corresponding data were plotted as Figure 20. The Curves show the behaviour of output power for absorption and desorption of relative humidity. The sensitivity calculated from absorption curve of Figure 20, was found to be 0.66 μW / %RH. Here two different levels of physisorptions were observed. At the first level (30-80 %RH), more decrease of power was taken place. This might be due to the reason that here lower physisorption, large changes in refractive index and hence more bending of light took place. After first level physisorption, the output power became approximate constant for 80-90 %RH. At the second level of physisorption (80-90 %RH), less bending of power took place. This is again due to the reason that in this case, less physisorption, a less change in refractive index and hence small bending of light was taken place.

4.2 Adsorption of Methyl Orange

4.2.1 Measurement

The stock solution of desired concentration was prepared (25 ppm). The standard solution prepared using stock solution for adsorption measurments. In known solution of methyl orange, 0.5 gm of nickel doped tin oxide was loaded and left for 4 to 5 hrs. The residual concentration of methyl orange was determined after filtration in UV spectroscopy (α_{max} = 464 nm). The adsorption density of methyl orange on nickel doped tin oxide was calculated using following equation:

$$q = \frac{(C_0 - C)V}{m}$$

Where, C_0 and C_e are initial and equilibrium concentration (mg^{-1}) of methyl orange in solution, V is the volume, m is the weight of nickel doped tin oxide and q_e is the amount of methyl orange adsorbed by the nickel doped tin oxide ($mg\ g^{-1}$).

4.2.2 Result

- **Effect of Concentration of adsorbate-**

Figure 21(c) clearly illustrate the effect of concentration on adsorption isotherm of methyl orange on nickel doped tin oxide, with gradually increases as the concentration of methyl orange increases.

- **Effect of dose of adsorbent-**

The effect of dose of adsorbent on adsorption isotherm of methyl orange was studies the obtained result shows increases as the dose of nickel doped tin oxide increases.

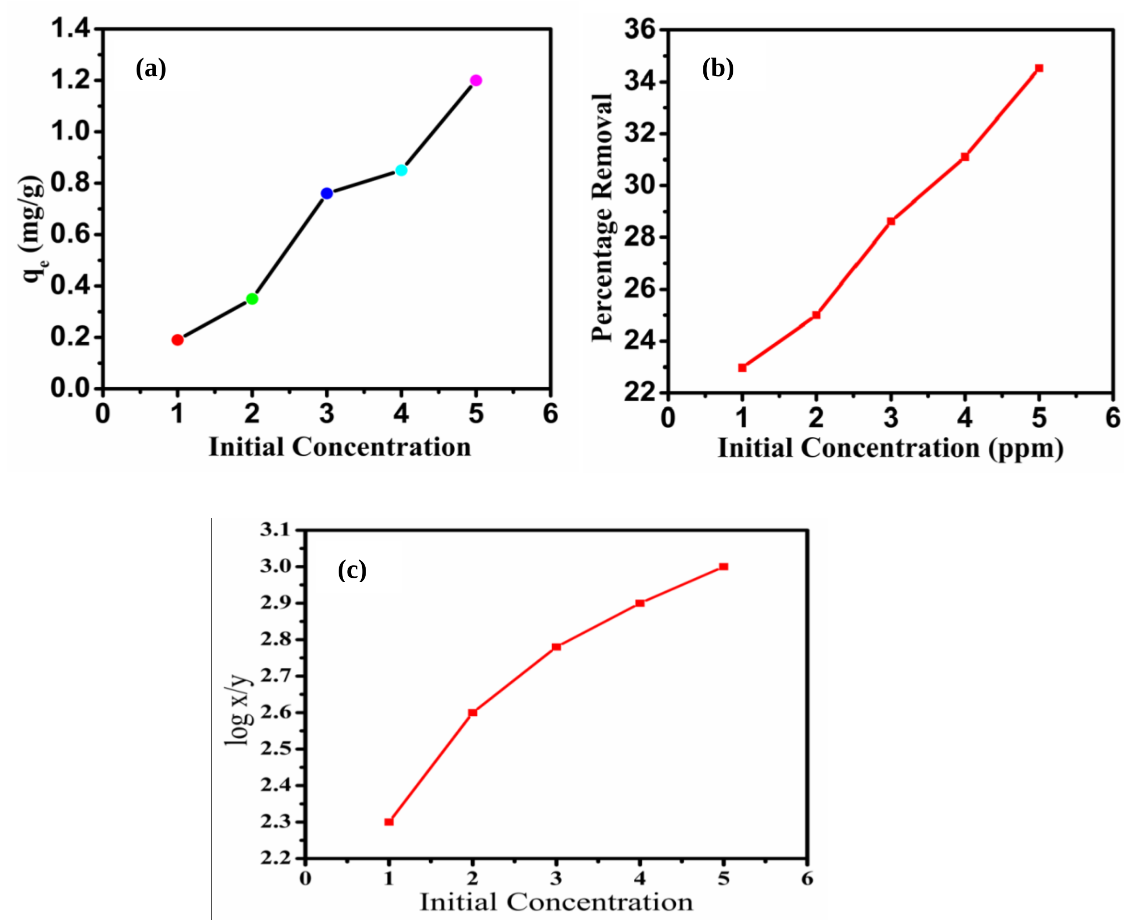


Figure 21: Adsorption of methyl orange (a) adsorption density, (b) percentage removal efficiency, and (c) Freundlich adsorption isotherm

Table 4: Observation of Freundlich adsorption isotherm

Bottle No.	Initial Concentration (C_1)	Residual (C_2)	Final Adsorbed ($C = C_1 - C_2$)	Amount of MO adsorbed (x)	Amount of absorbent used (m)	x/m	log x/m	log C
1.	5	3.2	1.8	50	0.05	1000	3.00	0.26
2.	4	2.6	1.4	40	0.05	800	2.90	0.15
3.	3	2.15	0.85	30	0.05	600	2.78	-0.07
4.	2	1.35	0.65	20	0.05	400	2.60	-0.18
5.	1	0.75	0.25	10	0.05	200	2.30	-0.60

Conclusion

Nickel doped tin oxide is successfully synthesized by chemical precipitation method and characterized by SEM, EDX, XRD, UV-visible spectroscopy, FTIR, BET and ZPC. The average sizes of nickel doped tin nanoparticles were found to be 15.182 nm by BET analysis and 21 nm by zeta seizer which confirm that prepared sample is range in nanoscale. The absorption wavelength for nickel doped tin oxide was found to be at 200 nm and corresponding band gap energy was 4.28 eV found by UV-visible spectroscopy. Opto-Electronics humidity sensing investigated by prepared thin film of nickel doped tin oxide which shows good sensitivity towards moisture and this prepared sample was also used for removal of methyl orange, which give good adsorbate result. Pores on prepared sample will play important role for sensing and adsorbate. The average moisture sensitivity is 0.66 μW / % RH.

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