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Study the Structural and Optical Properties of Nickel Oxide Nanoparticles

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***Abstract*- Nickel Oxide (NiO) was successfully prepared via the Sol-gel method. NiO nanoparticles was characterized using some comprehensive techniques such as X-ray diffraction (XRD), Fourier-Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy(TEM), and UV visible Spectra (UV-vis). The crystalline nature of the NiO nanoparticles is characterized through XRD. FTIR spectra revealed the presence of oxygen containing functional groups in NiO nanoparticles. The surface morphology was showed from SEM images and the presence of component was observed from Elemental Analyzer (EDAX). TEM image showed that the NiO nanoparticles about 50 nm. The test of UV-vis spectrometer indicated the NiO nanoparticles possessed excellent optical properties and analyzed the refractive index, optical bandgap, optical conductivity, Extinction coefficient and urbach tail. Using these absorption spectra, we further examined the NiO nanoparticle to explore the possibility of using them as a material for applications such as solar cell, active optical filters, Adhesive and coloring agents for enamels.**

Keywords—elemental analyzer, extinction coefficient, diffractio, refractive index.

I. INTRODUCTION

Nanoscience primarily deals with synthesis, characterization, exploration, and exploitation of nanostructured materials. The materials that are characterized by at least one dimension in the nanometer range is named nanomaterials. The process, structure and properties of materials in tens to many hundreds of nanometer vary are research areas of interest over the past years. Areas of physics like nanoelectronics, nanomechanics, nanoionics and nanophotonics have evolved throughout the previous couple of decades to produce the basic scientific foundation of nanotechnology. As researchers realize ways to characterize and pattern materials at the nanometer length scale, resolution is happening in nanotechnology and engineeringto be used in data technology, biotechnology, and energy and environmental applications, new materials with outstanding electrical, optical, magnetic and mechanical properties are being developed quickly. Nanotechnology could also be able to produce several new materials and devices with an enormous range of applications, like in drugs, electronics, biomaterials energy production, and consumer product.

On the opposite hand, nanotechnology raises several problems like issues concerning the toxicity and environmental impact of nanomaterials, and their potential effects on global economics, moreover as speculation concerning varied doomsday scenarios. These concerns have led to have a discussion among support teams and governments on whether or not special regulation of nanotechnology is secured.

Nanotechnology covers each current work and ideas they are additionally advanced that has the projected ability to construct things from the bottom up, using techniques and tools being developed nowadays to form complete, high performance compact products, two main approaches are utilized in engineering. Within the "bottom-up" approach, materials and devices are designed from molecular elements that assemble themselves chemically by principles of molecular recognition. In the "top-down" approach, nano-objects are made from larger entities without atomic-level management. Nanostructure and technology could be a broad and knowledge base area of research and development activity that has been growing worldwide. it has the potential for revolutionizing the ways during which materials and products are created and also the range and nature of functionalities may be accessed. Nanomaterials are employed in variety of applications is being widely researched nowadays across multiple domains. Nickel oxide nanoparticles seem in green powder type, and are ranked as a very toxicant and also they will cause an allergic skin reaction, prolonged harmful effects to aquatic life, and attainable harm to organs as a result of prolonged or continual exposure.

Nanosized metallic powders are used for the production of gas tight materials, dense parts and porous coatings. Cold welding properties combined with the malleability make them appropriate for metal-metal bonding particularly within the electronic industry. NiO semiconductor become a remarkable topic in the new space of research due to the quantity impact, the quantum size effect, the surface effect and also the macroscopic quantum tunnel effect, nano-crystalline NiO is predicted to possess several improved properties than those

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of micrometer-sized NiO particles.NiO may be made ready by multiple strategies. Upon heating higher than 400 °C, nickel powder reacts with oxygen to provide NiO. In some commercial processes, green nickel oxide is formed by heating a combination of nickel powder and water at one thousand °C, the rate for this reaction are often enhanced by the addition of NiO. the simplest and most flourishing methodology of preparation is through pyrolysis of a nickel(II) compounds like the hydroxide, nitrate, and carbonate, that yield a light green powder. Synthesis from the weather by heating the metal in oxygen will yield grey to black powder that indicates nonstoichiometry

NiO is employed as an element within the nickel iron battery, a element in fuel cells, precursor to several nickel salts, as specialty chemicals and catalysts, and it's a flexible hydrogenation catalyst. OptiFDTD may be a powerful, highly integrated, user-friendly software system that enables pc assisted design and simulation of advanced passive photonic element. the method permits for the effective and powerful simulation and analysis of sub-micron devices with terribly fine structural details.

II. MATERIALS AND METHODS

1) Synthesis Of Nickel Oxide Nanoparticles

i) Combustion Method

In combustion method, two compounds of nickel can be prepared via two approaches as given as follows.

a) Preparation of Nickel oxalate

Polyethylene glycol of molecular weight 6,000 was obtained commercially (Merck Chemicals). The double distilled water is used is for preparation of solution.

The NiO is synthesized through self-propagating low temperature combustion route, employing nickel oxalate as precursor. This precursor is prepared by dissolving equimolar quantity of Nickel sulphate heptahydrate and oxalic acid in minimum amount of water. This mixture was well stirred in a three-necked flask. The Light green precipitate of nickel oxalate dehydrate obtained was filtered through sintered glass funnel and washed with double distilled water. Finally it was washed with dry acetone and dried under vacuum for some hours.

b) Preparation of Nickel Oxide (NiO)

Thermal decomposition of Nickel Oxalate precursor with a fuel leads to the formation of high surface area NiO. The above prepared Nickel Oxalate was mixed with Polyethylene glycol (PEG) in the weight ratio 1:5 and ground well in a pestle and mortar. The resultant solid was placed in a crucible and heated in air. It was observed that initially PEG melted, then frothed and finally ignited to give NiO as a residue.

On cooling to room temperature, no traces of carbon impurities were observed in the final residue of NiO. As the reaction is fast, i.e. going to completion within 10 min, and ignites auto-catalytically, the exact temperature of the reaction could not be measured. However, using a thermocouple the highest temperature of the reaction was found to be around 500°C.

ii) Thermal Decomposition

The mononuclear 2,9-dimethyl-1,10-phenanthroline-nickel(II) complex was isolated in excellent yield by stirring equivalent amounts of 2,9-dimethyl-1,10-phenanthroline in distilled water with NiCl2·6H2O in ethanol [33,34]. NiO nanoparticles can be successfully synthesized through thermal decomposition of the (2,9-dimethyl-1,10-phenanthroline)NiCl2 complex precursor at 400 °C. NiO is formed via decomposition of 2,9-dimethyl-1,10-phenanthroline organic and chloride ligands in open atmosphere to NiO powder product and COx, NOx, ClOx as expected gases bi-products. Uniform and spherical NiO nanoparticles with weak agglomeration were collected.

iii) Sol-gel Method

NiO nanoparticles can be amounts of water (H2O) (0, 10, 20, 40 mass%) were added to dilute the ethanol solution. 0.01 mole of nickel nitrate (Ni(NO3)2·6H2O) was then added to the F108 ethanol solution and stirred vigorously for 1 h. The role of the block copolymer in the as-made sample was used to control the growth of nanoparticles and coat the nanoparticles to prevent them from further oxidation and aggregation. These nanoparticles were easily dispersed in ethanol to form a homogeneous colloidal solution. The resulting sol solutions were aged and dried at 343K in an oven for 48 h.

iv) Anodic Arc Plasma Method

The material includes the stainless steel vacuum chamber, the gas supply device, the DC power supply, the plasma generator with a high frequency initiator, the vacuum pump, the water-cooled collection cylinder, and the water-cooled copper crucible. The bulk pure material to be evaporated was laid on the water-cooled copper crucible, which served as the anode. Ni rod with 10 mm diameter is mounted in an insulated and axial manner, which was also water cooled and served as the cathode.

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In the process of preparation, the vacuum chamber was pumped to Pa and then a mixture of O_2 and Ar (1 : 4 by volume ratio) was backfilled as a reactant gas to reach the desired pressure. The electric arc in the inert environment was automatically ignited between the Ni electrode and the nozzle by high-frequency initiator, which was maintained by the current source at the pre-established values of the voltage and current. Under argon pressure and electric discharge current, the ionized gases were driven through the nozzle outlet and form the plasma jet. The bulk metal nickel (purity 99.99%) was heated and melted by the high temperature, and metal atom detached from the metal surface when the plasma jet kinetic energy exceeds the metal superficial energy, and evaporated into atom soot. Above the evaporation source was a region of supersaturated metal vapor, where metal atoms diffused around and collided with O atom to form NiO at high temperature due to the oxidation reaction.

When the vapor was supersaturated, a new phase was nucleated homogeneously out of the aerosol systems. The droplets were rapidly cooled and combined to form primary particles by an aggregation growth mechanism. The particles were transported from the nucleation and growth region to the inner walls of the cylinder by the free inert gas convection between the hot evaporation source and the cooled collection cylinder, the loose nanoparticles could be obtained.

v) Chemical Precipitation Method

NiO/MWNTs nano composites have been prepared by chemical precipitation method with the sodium dodecyl sulfate (SDS). Chemical precipitation is the approach utilized for the production of NiO; Materials mainly used nickel nitrate hexahydrate, sodium hydroxide, and polymeric (PVP, PEG) and cationic (CTAB) surfactants. The Ni(OH)2 precursors were prepared via the precipitation transformation method, which originated from Na2C2O4, NiSO4 ·6H2O and urea. The NiO samples were successfully obtained by calcining the Ni(OH)2 precursor with different calcinations methods.

vi) Chemical Methods

Single crystalline, defect free metal oxide (NiO) nanoparticles with diameter ~40nm was synthesized through chemical synthesis route. Crystalline cubic NiO rods with diameter ranging from nanometer to few hundred nanometers and lengths up to 10mm have been realized from a simple chemical route. The chemical reaction, of aqueous solutions of nickel chloride and sodium hydroxide with different molar ratios of NiCl2 and NaOH, formed the nickel hydroxide precursor which on thermal dehydration resulted in NiO nano crystals with rod like morphology.

The Nano crystalline nickel zinc ferrite was prepared via chemical synthesis; Zinc nitrate, Nickel nitrate, iron nitrate, citric acid and ethylene glycol were used as precursor materials. Nano crystalline NiO has been prepared successfully by a simple chemical route using NiCl2·6H2O and NaOH aqueous solution at a temperature of 70°C. Face centered cubic Nano crystalline nickel nanoparticles prepared at 60ºC from NiCl2 precursor using hydrazine hydrate as reducing agent and EG as capping agent. A nickel salt–urea–H2O ternary system has been developed for the large-scale synthesis of hierarchical α-Ni(OH)2 microspheres, the solid precursor for the subsequent topotactic transition to NiO upon calcination.

In this facile synthetic system, hierarchical structure is self-assembled under the cooperative direction of urea and anions in nickel salts. Thus, simply tuning the Ni salts leads to the selective construction of urchin and flowerlike hierarchical α-Ni(OH)2 and NiO microspheres consisting of radial 1D nano wires and 2D nano plates, respectively. NiO nanoparticles have been prepared by the decomposition of the hydroxide.

vii) Polymerized Complex Methods

Synthesizing the spherical, size tunable, well dispersed, stable nickel and nickel oxide nanoparticles by reduction of nickel nitrate at room temperature in a TX-100/n-hexanol/cyclohexane/water system by a reverse micro emulsion route. Nano particles of fcc-NiO phase were obtained by heating the dried resin resultant of a mixture of gelatin and NiCl2.6H2O in aqueous solution.

Nickel oxide (NiO) nano crystallites with a crystal size of around 54 nm have been synthesized via the polymerized complex method. Nanoparticles of nickel oxide have been prepared through a new mixed reverse micro emulsion route. Quaternary micro emulsion (water/surfactant/co surfactant/ oil-phase) was used to synthesize nickel oxide nanoparticles. The micro emulsion was prepared by Tween-80, Aerosol-OT, n-Propanol, Cyclohexane, and Nickel Chloride.

viii) Thermolysis Methods

Using hydrothermal method, nano structures of various kinds of oxides: ZnO, CuO, NiO, and Ga_2O_3 were fabricated from aqueous solutions of the respective metal nitrate hydrate and hexamethylenetetramine by hydrothermal method. The hydrothermal calcinations method is used for the synthesis of NiO nanoparticles by using Ni (NO3)2.6H2O, HMT and NaOH as starting materials. Nanoparticles of NiO with average diameters in the 3–24 nm range have been prepared by the decomposition of nickel cupferronate or acetate under solvothermal conditions.

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ix) Laser Ablation Methods

Nickel oxide nanoparticles were fabricated by a laser ablation technique using the third harmonic of an Nd:YAG laser and sintered NiO targets in an on-axis configuration under argon pressures of 0.67, 1.33, and 2.00 Pa . Pulsed laser ablation in liquid (PLAL) has been widely applied for the generation of nanoparticles. The generation of NiO nanoparticles using a high-power, high-brightness continuous wave (CW) fiber laser source at a wavelength of 1,070 nm. NiO nanoparticles with average particle size of 25 nm were prepared by anodic arc method

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x) Microwave-Assisted Preparations

By using a domestic microwave furnace and depending on the nickel precursor used, either tetra hydrated nickel acetate or dehydrated nickel formate, different nano sized materials- Ni/NiO composites, Ni metal, or NiO are obtained. NiO has been synthesized by microwave induced chemical synthesis route using metal organic complex of nickel in a domestic type microwave oven (2.45 GHz).

Nanoparticles of nickel oxide with an average crystalline size of 45-55nm have been prepared by microwave irradiation using nickel nitrate and sodium hydroxide solutions as the starting materials. The precipitation of nickel hydroxide after dry was irradiated by microwave radiation for short time. Nickel oxide (NiO) nano-particles were produced via a rapid microwave-assisted method using $Ni(OH)_2$ precursor which is obtained from nickel nitrate and sodium hydroxide. Nano-sized NiO was synthesized by microwave firing through the thermal decomposition of nickel carboxylate precursor employing glycine as a fuel. Nanoparticles of ZnO, MgO and NiO were produced from droplets of aqueous salt solution in the flame spray pyrolysis reactor.

xi) Chemical Vapour Deposition Methods

The ultrahigh pseudo capacitive nickel oxide nano particulate films on the nickel foils are prepared through a two-step in which nickel hydroxides are electrodeposited on the nickel substrate in 0.08 M $Ni(NO_3)_2$ aqueous solution and then they are thermally transformed into uniform nickel oxide nanoparticles [89]. Ni/NiO nanoparticles were synthesized by metal organics chemical vapor deposition of nickel acetylacetonate in an externally heated tube flow reactor at moderate temperatures, up to 500°C.

xii) Solution Methods

Nickel oxide (NiO) nano wire was synthesized by a simple aqueous solution method with urea as precipitant. A size series of ligand-stabilized Ni nanoparticles with diameters between 8–24 nm was prepared by solution chemistry, followed by solution-phase oxidation with atmospheric oxygen at 200°C to form Ni(core)/NiO(shell) nanoparticles with shell thicknesses of 2–3 nm. The nano crystalline nickel oxide (NiO) particles can be successfully prepared by a simple, fast, economical and eco -friendly solution, combustion method using $Ni(NO_3)_2$ .6H2O (oxidizer) and sugar. Nano crystalline metal oxides (ZnO, NiO, and SnO2) powders with an average particle diameter of 18–55 nm is prepared with the surfactant-mediated method.

All the chemical reagents utilized in this experiment were of analytical grade and were used as received while not further purification. NiO nanoparticles were successfully synthesized by using 1 g of nickel nitrate hexahydrate (Ni(NO3)2•6H2O) and 0.5 g of sodium hydroxide (NaOH) dissolved in 90 ml of absolute water. after five minutes of smartly stirring the answer, then it absolutely was cooled to room temperature. The greenish precipitates were washed with distilled water and completely ethanol for six times to get rid of impurity. the final products were dried in vacuum at 120ºC for 6h. After that, the sample was kept underneath sintering at 400 ºC for 3h. the ultimate product of the particle was obtained in black colour.

a) Characterization of synthesized NiO nanoparticles

The synthesized NiO nanoparticles were characterized by the subsequent. X–ray powder diffraction (XRD) by employing a Seifert (JSO-DEBYEFLEX 2002) diffractometer with Cu-Kα1 radiation (λ=0.1540 nm). Fourier transformed infrared (FT-IR) spectroscopy by using NICOLET 205 spectrometer (as pellets in KBr). Scanning electron microscopy (SEM) and Energy dispersive X-ray (EDX) analysis (accompanied) performed on a Hitachi S- 4500. Transmission electron microscope (TEM) was taken with a JEOL-3010 operating at 200KV. Ultraviolet – visible (UV-vis) absorption spectra were obtained from a Varian Cary 5E spectrophotometer.

The purity and crystallinity of the as-synthesized NiO nanoparticles were examined by using powder X-ray diffraction (XRD) as shown in Fig.1. The XRD patterns of the product ensure that the shaped material is nickel oxide. It is seen from the XRD patterns that the diffraction peaks are low and broad owing to the tiny size effect. The peaks positions showing at 2θ = 37.200, 43.200, 62.870, 75.200, and 79.380 is promptly indexed as (111), (200), (220), (311), and (222) crystal planes of the majority NiO, respectively. Of these diffraction peaks is absolutely indexed to the face-centered cubic (FCC) crystalline structure of NiO, not solely in peak position, however additionally in their relative intensity of the characteristic peaks, that is in

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accordance with that of the quality spectrum (JCPDS, No. 04-0835).

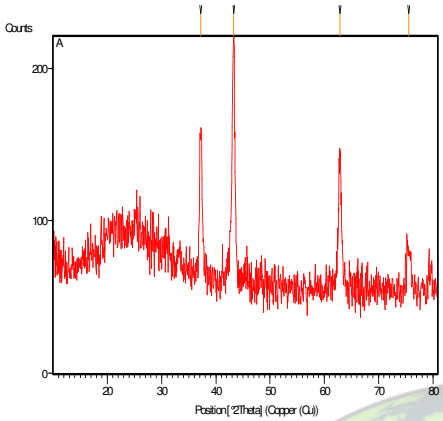


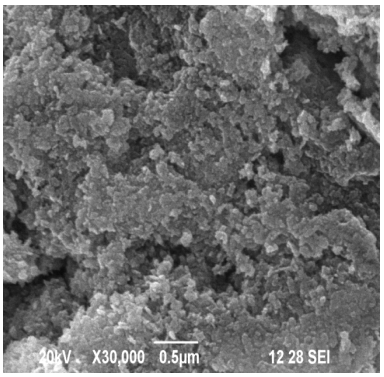
Fig. 1 Characterization of XRD Pattern spectra

The XRD pattern shows that the samples are single phase and no the other impurities distinct diffraction peak except the characteristic peaks of FCC phase NiO was detected. This result shows that the physical phases of the NiO nanoparticles have higher purity ready during this work. The crystal size of the as synthesized nickel oxide nanoparticles calculated from the diffraction peaks using the Debye- Scherrer's formula:

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

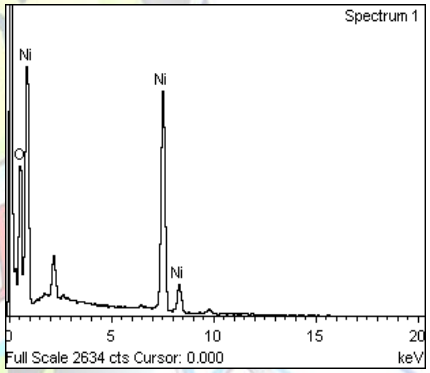
where K is a constant (0.9), λ is the X-ray wavelength employed in XRD (0.15418 nm), θ is the Bragg angle, β is the FWHM (full width at half most intensity), that is, broadening owing to the crystal dimensions. The diameter of the nanoparticles calculated by the Debye- Scherrer's formula is 27 nm of NiO nanoparticles.

The surface morphological studies and composition analysis of the nickel oxide nanoparticles were allotted using SEM and EDX images shown in Fig. 2 a-b. From the SEM analysis, one will conclude the formation of nanoparticles granular structure. The EDX spectrum for NiO nanoparticles shown in Fig. 2(b) discovered that the presence of Ni and O because the solely elementary species within the sample. Moreover, no further peaks similar to any other components except Ni and O were determined.



a) SEM image

Use of TEM for determining the particle is most popular over X-ray line broadening. This technology is more direct and less probably to be suffering from experimental errors and/or alternative properties of the particles like strain or a distribution in the size.



b) EDX pattern

Fig. 2 Characterization of SEM image and EDX pattern

Fig. 2a depicts the well distributed NiO nanoparticles size and morphology of primary nanoparticles will be further confirmed from imaging by TEM analysis. Chosen area electron diffraction pattern of the samples shown in Fig. 3b.

The figure indicates that almost all particles are fine and spherical whereas some are elongated. The typical particles size was measured to be 27 nm that is in good agreement with calculated particles size by XRD analysis. A typical SAED patterns taken from the individual nanoparticles was shown in Fig. 3b, for NiO nanoparticles are polycrystalline structure.

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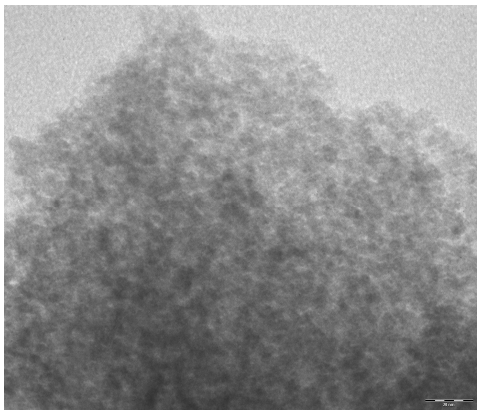
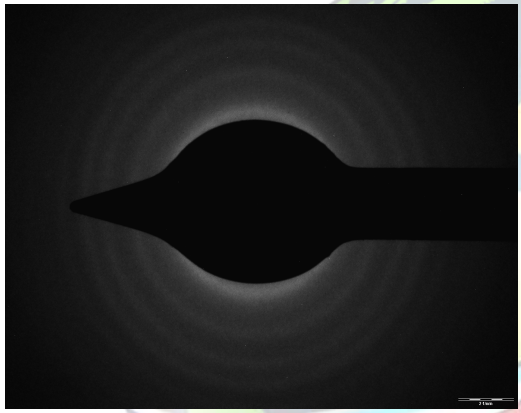


a) TEM image



b) SAED pattern

Fig. 3 Characterization of TEM image and SAED pattern

Fig. 3a shows the UV-Vis absorption spectra of the as-synthesized NiO nanoparticles. The band gap is set by using extrapolating the linear portion of the plot shown in Fig. 3b-c. the worth of the absorption edge of the samples is 318 nm. According to the data of the absorption spectra, the optical band gaps (Eg) of NiO nanoparticles can be predicted by using the following equation:

$$(Ahν)^n = B(hν - E_g)$$

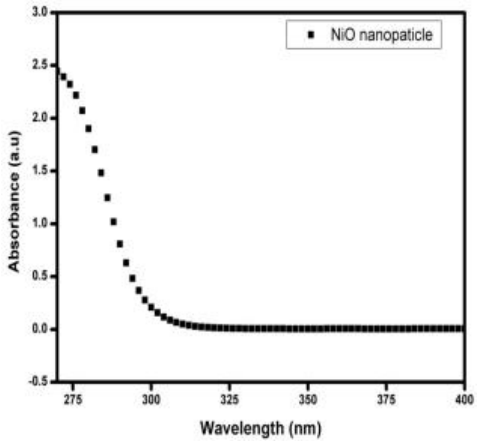
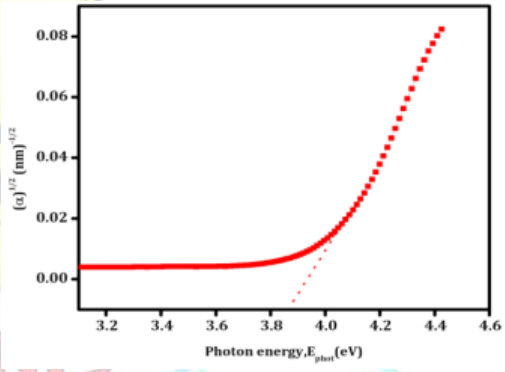
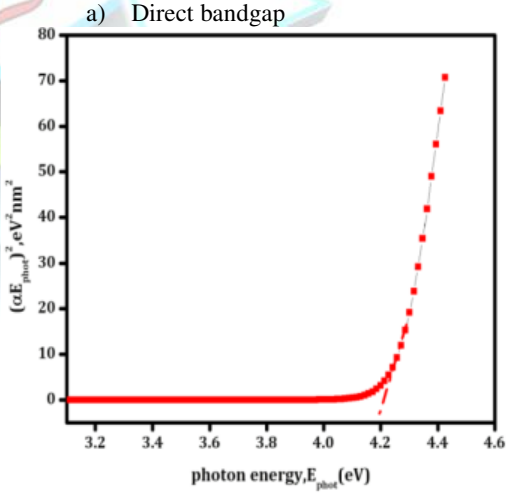


Fig. 4 Characterization of UV spectra



a) Direct bandgap



b) Indirect bandgap

Fig. 5 Direct and Indirect Bandgap

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