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Synthesis and characterization of Ni ion doped ZnO nanoparticles UsingEuphorbia abyssinica bark plant aqueous extract To study antimicrobial activity

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Jimma, Ethiopia

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This thesis is Submitted to School of Graduate Studies College of Natural Science Jimma University in partial Fulfillment of the Requirements for the Degree of Masters in Chemistry(Inorganic Chemistry) (M.Ss)

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LIST OF ABBREVIATIONS AND ACRONYMS

Å Angstron

eV Eectron Volt

FT-IRFourierTransformsInfrared

FWHM Full Width at Half Maximum

Nps Nanoparticles

NNNickel nitrate

XRDX-ray diffraction

UV-vis Ultraviolet visible specterometer

ZN Zinc nitrate

Abstract

The green synthesis of metal oxide nanostructure is an interesting subject of nanoparticles. Also, of latest concern is the biosynthesis of metal oxide nanostructure using plants for the large-scale biosynthesis. And doping lead to versatile changed properties as result of slightly changed structure. Therefore, the main aim of the present study was green synthesize of Ni^{2+} doped ZnO nanostructure to evaluate their antimicrobial activity against some selected microbes. The sample was prepared from nickel nitrate hexahydrate and Zinc nitrate hexahydrate with extracted Euphorbia abyssinica bark plant at roomtemperature. This method was interested to synthesize Ni doped ZnO nanostructureusing Euphorbia abyssinica bark plant extract; since the process of synthesis was eco-friendly, low cost, quite suitable for biological and medical application.Finally the synthesized samples had been characterized and examined by UV-Vis spectrum, (FT-IR) Fourier Transforms Infrared and x-ray diffraction (XRD).The UV-Vis maximum absorption peak shows that as doping increase the lambda max increases as result of new atom insertion, from FT-IR the stretching vibration of the Ni doped ZnO ranges between 524 to 439 nm and XRD patters showed the crystallite of Ni doped and un doped ZnO Nps and using Debye-scherrer formula, the size of the sample was determined. As doping increase the size was increased from 8.11 nm to 14.90 nm and the band gap was decreased. in this work using green methods nano sized with simple procedure well crystal sample was synthesized and Lastly it was investigated the antimicrobial activity of Ni^{2+}doped ZnO nanostructure with gram positive and gram negative bacterial strains and antifungal activity by agar well diffusion method and their inhibition zone showed good anti-bacterial and anti-fungal activity.

Key words; Euphorbia abyssinica, UV-Vis spectrophotometer, (FT-IR) Fourier Transforms Infrared-Ray Diffusion (XRD), Nickel doped and undoped ZnO nanostructure, microbial activity

1. INTRODUCTION

1.1 BackGround of the study

Researchers have focused on developing of well-organized green methods for synthesizing of nanoparticles and biosynthesis of metal oxide nanoparticles. Since in the 21st century they have innovated nanotechnology for the process of observing, measuring, manipulating and manufacturing of atoms and molecules at nanoscale size [1].The latest concern of preparing nanoparticles was by using biosynthesis of metal nanoparticles which was more stable and more varied in shape and size.Thusplants contain biological reducing agents like terpenoids, flavonoids, tri-terpenoids and phenollic compounds [2].

Therefore; the researcherused *Euphorbia abyssinica* bark plant as reducing and stabilizing agents and This plant give a better uses for nanoparticles synthesis as they are free from toxic chemicals as well as provide naturalcappingagents[3-4].Nano materials have a wide variety of applications because of their changeable properties from bulk size to smaller size and this increases surface area to volume ratio.From few papers it was reported that biosynthesis of zinc oxidenanoparticles usingplantextractssuchasAloebarbadensismiller,*Physalisalkekengi*, *Azadirachtaindica* [5-7].Andthis green synthesis of metal nanoparticles was interesting issue of nano science and which synthesized by plants are more stable and more varied in shape and size in comparison with those produced by other organisms.

As reported by different literatures;*OcimumbasilicumL.var.Purpurascens,Medicagosativa, Ixora Cochineal*Leaf Extract,*Hibiscussubdariffa* leaf extract, plant-mediated extract, Root Extract of Zingier officinal, [8-13] all credible uses and had been synthesized by green methods.
Since doping is adding intentionally impurities into crystal stricture or to host lattice in small amount and this led to slightly change the properties of the original substances in doped ZnO Npsmanipulate the properties of ZnO Nps and as result of doping, the size of the samples was either increases or decreases based on the reducing power as well as the size of dopantused in the synthesis [14].Also doping slightly can change the properties of the synthesized samples which is constant properties in bulk size. Therefore; the researcher emphasize to synthesis the Nidoped

ZnO Nps by green method which had not reported ahead of by any researcher rather by chemical method. Synthesis of nanoparticles using *E.abyssinica* bark plant extract was not reported before however the chemicals that exist in this plant can enhance the reduction of metal ions to metal oxide nanoparticles. Physiochemical screening test shows most euphorbia species produce a milky latex, which yields a wide range of chemicals such as rubber, oil, hydrocarbons, starches, waxes, resins, balsams and tannins[15].As result of this using this plant for reducing agent and stabilizing agent was preferred by researcher. A doped and undoped ZnO nanoparticles; that was synthesized by green methods has strong anti-microbial activities against bacteria and fungus activity [16]. Green synthesize of zinc oxide and iron oxide nanoparticles using *Sesbania grandiflor*leaf extract provides an effective route for eco-friendly method of synthesis of nanoparticles [17].Also green synthesis of ZnO nanoparticles is much safer and environmentally friendly as compared to chemical synthesis.

Traditionally green plants have been used for different applications since traditional medicine plays a significant role in the healthcare of the majority of the people in developing countries, including Ethiopia, and medicinal plants provide valuable contribution to put into practice [18].As it was reported;*Euphorbia abyssinica* bark used by peoples traditionally for diseases like;gonohhrea, syphilis [19]. ZnO nanoparticles which prepared by *aloe Vera* plant extract shows 340 nm and sensitive to both gram positive and gram negative bacteria and also doping metal oxide nanoparticles by chemical methods such as Ni(II)doped ZnO nanoparticles had been synthesized with zinc acetate dehydrates and nickel acetate tetra hydrate followed by heat action and NI(II)doped ZnO nanoparticles which was prepared is interested due to size and shape controlled oxide nanostructure are very important in controlling their physical and chemical properties.Ni is an important dopant in magnetic/ZnO materials, since Ni^{2+} has (0.69Å) with the same valence as Zn^{2+} and its radius is close to that of Zn^{2+} (0.74 Å) .Threrefore,Ni(II) is easily replaced by Zn^{2+} in to ZnO lattice and the luminescence properties of ZnO NPs varied by doping Ni(II)[20]. ZnO has high electron mobility, high thermal conductivity, good transparency and directed band gap (3.37 eV). Moreover, ZnO is preferentially stable hexagonal wurtzite,zinc blend and rock salt structure at room temperature and each oxygen ion surrounded by tetrahedral zinc ion.

The characterization and synthesis of Ni ion doped ZnO Nps of particles with size 12 to 5 nm reported for the first time by using simple methods of non-basicand the band gap was observed with red shift, this is due to sp-d interaction between bond electron and localized d-electrons of Ni ion substituted in ZnO Nps [21].Colloidal, and aerosol processes are used to prepare nano particles and when we look their importance; they interested by researchers, due to their high reactive to the large surface area to volume ratio. Silver doped with other metal oxides such as Zinc oxide nanoparticles which is used both as a promising, bactericide, a photo- Catalyst, for its Low cost, high catalytic activity and Environmental friendliness. In order to make easy; the separation charge carriers in Zn-nanoparticles and ZnO has been hybridized into complex materials; such that Co-doped ZnO nanorods was reported [22].As it was reported that un doped MgO and MgO; Ni^{2+} 1%, 2 % and 5%Nps were prepared by solution combustion methods in simple, facile, eco-friendly green synthesis and photo catalytic study were by using Ethylene blue as hazardous dye then photo catalytic properties increases as Ni(II)doped ZnO nanoparticles have been done by many experimental studies on the photo catalytic degradation of basic organic dyes with metal ion doped with ZnO which has been reported to possess high photo catalytic activity than undoped ZnO as the doping makes the absorption edges to shift towards longer wavelength[23].Nano-crstallite Ni doped and undoped ZnOnanoparticles with Compositional Formula $Ni_xZn_{1-x}O$(x=0, 1, 3&5mol% were synthesized because doped ZnO has subjected attention as mentioned before, in the process of sol-gel methods using citric acid was used as fuel [24].

There are several report for ZnO nanoparticles from green synthesis, towards their biological applications by using plant extract nanoparticles since using chemical methods have disadvantage due to its toxicity and less eco-friendly, then synthesizing Ni(II) doped ZnO nanoparticles by biosynthesis method have varies advantages than Chemical one. important transition metals for instance Ni nanoparticles has wide ranging application in the fields permanent magnetic, magnetic fluids, recording media, solar energy absorption, fuel cell electrodes dye removal and catalyst [25].Those before prepared metal doped nanoparticles were having eco-friendliness, non-toxicity and low cost at low temperature. Also Fe doping is focused to make available possibility for elucidating the photo catalytic activity of various metal oxides owing to its important role in narrowing the band gap. Zinc oxide has a wide band gap (3.37eV)

therefore used in semiconductors which is an important functional material because of its variety of important properties such as sensors, electrical photovoltaic and photo catalytic properties and ZnO has been found highly attractive because of its remarkable application potential in solar cells, piezoelectric devices, and UV absorbers, pharmaceutical and cosmetic industries[26].

However, Ni doped ZnO naoparticles by biosynthesis of*Euphorbia abyssinica* bark plant extract to study anti-bacteria andanti-fungalactivity was not reported before. nevertheless moderately pollutant free chemicals are used in green synthesis techniques of nonmaterial's and thus, environmentally benign and renewable bark plant extract of Euphorbia abyssinica has effective stabilizing as well as reducing agent for synthesizing of ZnO Nps and Ni^{2+}doped ZnO Nps. Green synthesis methods have several merits such as simplicity, good stability, less time consumption, non-toxic side-effect and large scale synthesis and using plants for the synthesis of nanoparticles is that they are easily available, safe to handle and possess a broad variability of metabolites that may aid in reduction. Many plants are being currently investigated for their role in the synthesis of nanoparticles[27].

Microbial infection and control by drugs available, research reports by metal oxide which is new formulation s against the bacterial strains which are developing resistance for otherantibiotics [28].Undoped ZnO and doped ZnO Nps synthesized by green method showed good antimicrobial activity towards pathogenic microbial strains and strong candidate for therapy against mult-drugresistance of microbial.

Biosynthesized ZnO nanoparticles with different particle size for different applications including antibacterial activity by using extracts from plants such as*Agathosmabetulina,Camellia sine sis,Sesbania grandiflora*and*Euphorbia milli*[29-32], has been reported and the results found are quite conclusive. However, we found as there is no report on the synthesis of Ni ion doped ZnO nanoparticles by using *Euphorbiaabyssinica*.Thus, the main objective of this study is to perform green method synthesis of Ni ion doped ZnO nano particles by using bark extract of *Euphorbia abyssinica* (*Adami in Afaan Oromo)(Qulqwal in Amharic*), and to characterize and evaluate its antimicrobial activity against selected bacterial and fungal strains.

1.2. Statements of the problems

Nanotechnology is an interesting field in current world; this is due variety of uses uninterested industrial applications. Most of bulk substances are their properties are constant but for Nanostructure the properties can easily change. Nowadays chronic diseases have high resistance towards drugs there for this innovation can solve the problems since mostly small size was preferred for anti-microbial activity. The other attention-grabbing, doping can change on dopinf the activity of the original substance. therefore, synthesis of Ni (II) doped ZnO Npscan be synthesized by chemical synthesis methods.

However, thismethod has mor disadvantage than green methods since reducing and stabilizing agents was obtained from the plant.Therefore,physiochemical screening test confirms E.abyssinica plants are rich of secondary metabolites.As result of this motivated to synthesize Ni ion doped ZnO nano particles by *Euphorbia abyssinica/adaami*/ bark extract since Ni doped ZnO Nps by any plant also undoped ZnO Nps by Euphorbia*abyssinica*in different applications was never reported before. Though in different industries increasing surface area to volume ratio was interested as well as doping metals in metal oxide at nanoscale have advantages in current technology and synthesizing small size of undoped ZnONps and Ni doped ZnO Nps using green methods is novel innovated and can be used in different fields area and using this method was enhance metal ion doped to ZnO Nps in further synthesis.

1.3 Objectives

1.3.1 General Objective

To study the synthesis and characterization of Ni (II) doped ZnO nanoparticles and their anti-microbial activity using *Euphorbia abyssinica* bark aqueous extract.

1.3.2 Specific objectives

- To extract *Euphorbia abyssinica* bark plant
- To synthesize Ni (II) doped and undoped ZnO nanoparticles.
- To characterize the synthesized samples byFourierTransformsInfrared (FT-IR) X-ray diffraction (XRD), andUV–Vis spectrometer.
- To evaluate anti-microbial activity of *Euphorbia abyssinica* plant extract of doped and undoped ZnO Nps.
-

1.4. Significant of the study

Synthesis of zinc oxide and nickel doped zinc oxide by green synthesis encompasses various significant and in different field of studies it reduce side product which released to environment.Researcherwanted to study the anti-microbial activity ofundoped and Ni ion doped ZnO Nps by *Euphorbia abyssinica* bark plant extract which was synthesized by non-toxic, low cost, at low temperature and easily obtained from local environment. This work solvesside effect of chemical synthesis and synthesized Ni doped ZnO nanoparticles which would enhance the anti-bacterial activity also it uses in different industrial applications and in different of fields. And doping can be enhance the properties of the orginal substances.

2. LITERATURE REVIEWS

2.1 Concept of Nanotechnology

In 21st century, the new technological innovation is Nanotechnology which has the capability to observe measure, manipulate, and manufacture things at the nanometer scale size of atoms and molecules. The development of this field is growing rapidly throughout the world and this field of science is a major contribution of science for the development of new materials in the nanometers scale. These are usually particulate materials with at least one dimension of less than 100 nanometers (nm), even the particles could be of zero dimension in the case of quantum dots. Nanoparticles exhibit completely new or improved properties with larger particles of the bulk materials and these novel properties are derived due to the variation in specific characteristics such as size, distribution and morphology of the particles. Since Nanoparticles has a higher surface area to volume ratio with decrease in the size, distribution and morphology of the particles [33].Doping nanoparticles can be either chemical or green methods; chemical doping is the process of doping through which metals (laboratory level synthesized) precursors are used as dopants and which toxic substance released to the environment, these metal ions come from laboratorial synthesized compounds or elements. However green methods both reducing and capping agents are from plant as result of this it was pronounced for various purpose consequently, we researcher focused on green methods of synthesis.

2.2 Application of Nanotechnology and synthesis

2.2.1 Green synthesis of doped and undoped ZnO nanoparticles

Zinc oxide nanoparticles were synthesized by both chemical and green methods as it was reported before, but currently researchers interested to prepare by green methods due to less cost, low time consumption, environmental friendliness, and the plants renewable after they used also even if, the organic molecules exist in plants are un controlled; they are enrich of reducing substances rather than chemically prepared reducing agents then synthesizing ZnO using green method was preferred. Using green synthesis of plant *per sea Americana*/avocado fruit/extract ZnONps was prepared by sol-gel methods which are biological method were reported [34] and this synthesis methods of diverse zinc oxide nanostructures can largely be classified as two most important categories namely gas phase synthesis and solution phase synthesis. The sol–gel method has gained much interest among researchers as it offers controlled consolidation, shape

modulation, pattern of the nanostructures. In the synthesis of ZnO Nps using *Aloe vera* leaf extract, ZnO Nps with average size 25–40 nm was synthesized and ZnONps has Physical appearance of; white, colorless, odorless solid crystalline structure and Wurtzite fine metal colloids have the optical, catalytic electromagnetic properties which are dependent on size and shape of the particles also for the anti-bacterial andanti-fungal activity.Using green method synthesis avoids use of harm full and toxic reducing and stabilizing agents. ZnONps exist in ions only in the presence of strong oxidizing substances and anti-bacterial activity increased with increasing in surface to volume ratio. As result of decrease particle size, nanoparticles showed most effective against both gram positive and gram negative bacteria [35].*B.glabra* leaf extract for synthesis of ZnO Nps has better choice due to eco-friendliness than chemically prepared one and also antibacterial resistance of it was modified as result of smaller distribution with respect to chemical methods [36].

Thesessynthetic processes or the preparation of colloidal nanoparticles with controlled
Morphology are crucial and is well known that impurity was added to a given substance by doping and changes arise in optical, electrical, and magnetic properties to ZnO nanoparticles to expand the band gab semiconductors as reported for transition metal doping to ZnO at room temperature. Due to this result Ni is important doping for ZnO and Ni doped nanoparticles have wide ranging application in the fields of permanents, magnetic fluids, magnetic recording media, solar energy absorption and fuel cell electrodes etc.

ZnO and Ni doped ZnO thin films were synthesized by using spray phyrolysis methods, this Ni doping reduce the bad gab, for concentration of Ni at 4% or under, the Ni substituted into Zn site at the lattice, but for higher concentration led to the formation of NiO like phase. And band gab of Zn_1-xNi_xO is strongly reduced for higher doping. X-ray Spectroscopy at low doping explained by Ni 3d&O 2P hybridization acting to raise the valence band maximum and at higher doping, the removal of Zn 3d states acts to relax the valence band, and change Ni coordination counter acts the lower of conduction bands this gives the ability of Ni (II) doping revolving the ZnO band gap between 2.8 and 3.4 eV [37].Therefore; synthesis of Ni doping nanoparticles was given attention. Ni doped MgO nanoparticles were synthesized by solution combustion methods using *S.grantii* plant extract as fuel by using magnesium nitrate hex hydrate with nickel nitrate hex

hydrate at temperature of 500 ^{0}C by simple eco-friendly green synthesis and these Ni doped ZnO NPs increases surface area to volume ratioad considered to be a technologically prodigious material having a wide spectrum of application such as that of a semiconductor (Eg =3.37 eV), magnetic material, electro-luminescent material, piezoelectric sensor and actuator, nanostructure field emission displaying material, thermoelectric material, gas sensor, constituent of cosmetics etc[38-39].As reported doped and undoped metal oxide Nanoparticles such as $Zn_{1-x}Ni_xO$ Nanoparticles was synthesized using either of roots, bark, leaves, stem extract using distilled water as solvent followed by adding of mole percent of Ni ion to Zn ion and Lastly it was calcinated for 2 hr. at 500^{0}C in muffle furnace then $Zn_{1-x}Ni_xO$ Nps was synthesized and these methods had reported [40].

2.2.2Application of ZnO nanoparticles

Nonmaterial are being used in a wide variety of applications due to its changeable properties on scaling down from bulk size to nanometer size (10^{-9}m) since nanometer substances ranges (1-100 nm) Zinc oxide is an inorganic compound with the formula ZnO and It usually appears as a white powder, it insoluble in ethanol and methanol but nearly insoluble in water. The surface area to volume ratio plays an important role in nanoparticles and appropriate to which they become more reactive bulky zinc oxide (ZnO), thismetal oxide is semiconductor, non-toxic and bio-compatible which has also a band gaps of about 3.37eV.Most commonly, ZnO nanoparticles are produced through chemical methods like sol-gel processing and this nanotechnology can find application almost in all branches of human activities [41].The application of nanotechnology in agricultural and food production, Nps and/or controlled-release and targeted delivery.

Nanoformulations are broadly used for agrochemicals (*e.g.*nano pesticides Nano fertilizers) that were primarily designed to reduce the amount of applied active ingredients by means of their enhanced bioavailability and protection against degradation, which finally resulted in a decrease of dose-dependent toxicity for non-target organism ‘sand environmental burden.Nanoparticles can be used as vectors for gene transport. The application of nanotechnology in the areas such as food packaging, food security, detection of pathogens and contaminants by using nanosensors and indicators, encapsulation of nutrients and development of new functional products is growing rapidly. Nano scale food packaging materials may extend food life, may improve food safety, may alert consumers that food is contaminated or spoiled, repair tears in packaging and

even may release preservatives to extend the life of the food in the package. As mentioned above, nano-size materials change their physical and chemical properties in comparison with bulk materials and can become toxic when reach nano-size. Therefore increased attention must be devoted to the impact of risk factors associated with their usage on the environment and possible adverse effects on non-target organisms and mammals, especially humans. [42].

ZnO usually has crystalline Wurtzite structure composed of stacking Zn(II) and O^{2-} ion alternatively al the ion in the crystalline structure are four fold coordinated due to lack of center of inversion and has polar surface. This lack of center of inversion leads to instability of crystal structure. According to the definition of anti-bacterial activity is the action through which bacterial growth is disrupted then antibacterial agents are basically some drugs which are capable to inhibit or destroy the bacterial and fungal cell and must not be harm full to the host cell. Therefore, nanoparticles were prepared by environmental friendly green method approach exhibit excellent biocompatibility and antimicrobial activity [43].Also ZnO Nps by Precipitation and electron deposition methods since ZnO acts as a physical barrier to the Ultraviolet (UV) radiation; they are being used in transparent sunscreen applications. Nanoparticles had being used in different fields including electrical, biological, textile and chemistry in which shape and size of colloidal metal particles play crucial role in different application including preparation of magnetic, electronic devices, cut curing, antimicrobial gene expression and they have wide band gap and large excitation energy. Flavonoids are forceful water soluble super anti-oxidants and the fruit may also act as free radical scavengers.

They prevent oxidative cell damage; have strong anti-cancer activity [44].*Euphorbia abyssinica* (Adaamii in Afaan Oromo) Qulqwal in Amharic) which In Ethiopia, It usually grows in steep rocky hill sides, and sometimes used for live fencing at high altitudes. Itperformswellin dry, moist and Wet Weyna Dega Agro climatic zones in Tigray, Gonder,Gojam, Shoa,Harerge andWollega and it was 1,400–2,400meter.Usuallyabove1, 900meter. Euphorbia abyssinica species name its family is *Euphorbiaaceae.* Looking for medicinal usage was, for external injury (kusil) (madaa) and for internal; worms, internal diseases, kintarot, undefined swelling and neck cancer. E.abyssinica, milky latex was extremely poisonous and dangerous irritating and specially can cause complete blindness (Oromo say" ijaaf adaamii") means opposite [45].

2.2.3Virtues of ZnO and doped ZnO Nps

Zinc oxide nanoparticles (ZnO Nps) are a class of inorganic metal oxides available and exhibit a wide range of nanostructures, Photo catalytic and photo oxidizing ability against chemical and biological species are used to characterize these metal oxides, as administration of food and drug have recognized, ZnO Nps looked as harmless and also lower cost, UV excess numbers property high catalytic activity, large surface area, white outward validate and their important applications in the field of medicine and agriculture are the advantages of ZnO particles [46] .Photo catalytic activity of ZnO nanoparticles offers a talented method for waste water treatment that of toxic water pollutants released from textile and dying industries by utilizing natural source of energy, sunlight are degraded by ZnO and demonstrate photochemical reactivity.

This could be because of the existence of many active sites and production of hydroxyl radicals on ZnO, Zinc oxide has enormous applications in optical, piezoelectric, magnetic, and gas sensing. ZnO nanoparticles is also used in manufacturing sectors including environmental, synthetic textiles, food, packaging, medical care, healthcare, as well construction and decoration. The physical properties of ZnO molecular weight 81.38 g/mol electron effective mass 60 melting Point 1,975°C, density 5.47 g/m^2,isoelectric point 9.5–10,standard molar entropy 43.9 $J{\cdot}K^{-1}mol^{-1}$, standard enthalpy of formation −348.0 kJ/mol,ZnO was prepared by cost effective plate form in large scale with high productivity yield by chemical methods and it hasdisadvantage due to its toxic precursors which restrict for use of biological applications therefore green synthesis of ZnO nanostructure is suitable for biological application [47].

2.2.4 Ni (II) doped and undoped ZnO Nanoparticles

Impurities and dopants may have various effects on crystals. Interstitially dissolved foreign atoms can normally affect the strain in the lattice. When ionized their charge affect the electro neutrality condition. Substitution ally dissolved impurities or dopants would also affect the properties of the host compound; if they exhibit a difference in size or charge compared with the host atoms. Since doping can slightly change the chemical and physical properties of the original substance therefore we researcher, interested for synthesis of Ni ion to ZnO Nps. Here are many experimental studies on the photo catalytic degradation of basic organic dyes with metal ions doped with ZnO Nps which has been reported to possess high photo catalytic activity than undoped ZnONps as the doping makes the absorption edge to shift towards longer wavelength

[48].The synthesis of Fe nanoparticles from natural or biological sources has received greatawareness due to its superior aspects of low-cost, rich biochemical constituents and high activity. Formation of Fe nanoparticles from agro residues and plant extract has been explored recently for its non-toxic nature and environment-friendly disposal of photosensitized degradation on semiconductor catalytic surfaces can be a remediation for colored organic pollutants. The photo catalytic activity of ZnO could be well improved by band gap narrowing, which usually requires a high doping level in the semiconductor, principal to a different material from the close relative oxide. Numerous researchers have shown that proper quantity of Fe doping could improve the photo catalytic property of semiconductor catalyst under light introduction conditions that could partially prevent the recombination of the e/h pair. Thus, Fe doping is focused in this study as it could provide feasibility for elucidating the photo catalytic activity of various metal oxides owing to its significant role in narrowing the band gap .Zinc Oxide has a wide band gap (3.37eV) semiconductor is an important functional material for the reason that of its variety of important properties such as sensors, electrical photovoltaic and photo catalytic properties [49].

Ni-doped ZnO nanoparticles has the average size approximation of 61 nm and Uv-emission peak was 384 nm when it synthesized by simple electrochemical route at room temperature. Ni doped ZnO nanoparticles were also prepared by adding equal mole of zinc nitrate and nickel nitrate using urea'sprecursors and its size was calculated by Deybe Scherer's for formula was determined with grain size of 32 nm. ZnO nanoparticles has been found highly attractive because of its remarkable application potential in solar cells, piezoelectric devices, and UV absorbers, Pharmaceutical and cosmetic industries. ZnO nano fibers which is doped with Aluminum oxide was prepared by sol-gel processing and electro spinning techniques [50].Aluminum (Al^{3+}) content increases in the processor solution, then the average diameter of nano fibers to be decreased. Liquid phases are interesting as low cost and was synthesized at low temperature. Those methods are sol-gel processor, co-precipitation, Hydrothermal or Solvo-thermal synthesis, template and Micro wave synthesis are some examples and Cu doped ZnO nano powder has been synthesized by co-precipitation methods [51]. And nitrogen double bond of the azo dye is the most active site for oxidative section. Therefore,Ni doped ZnO rods for the degradation of an azo dye from aqueous solution method was reported [52].

Since green synthesis techniques make use of modern relatively pollutant free chemicals to synthesis nano materials and seek to reduce pollution at source. ZnO and Ce doped ZnO nanoparticles are synthesized by simple and cost effective using aqueous leave extract of *Sesbeniagrandifloria* and bio-ZnO nanoparticles have efficient to degrade Bismarck brown dye under solar irradiation. Doping of transition metal onto ZnO nanoparticles lattice can modify its optical electrical magnetic properties as examples the micro structure of Ni doped ZnONps has irregular micro-spheres with hollow space at center, and a red shift in the band gab had been observed due to attributed to sp-d ZnO exchange interactions between band electrons and localized d electros of N^{2+} is substituting in Zn ion and clearly indicating the incorporation of Ni ion into the Zn site of ZnO lattice [53]Ni doped ZnONps by using co-precipitation method.

The physicochemical characterizations have revealed the morphology and particle size of these NPs.The substitution doping of Ni in ZnO latticeatlowtemperaturehas positive influenceon ZnO electronic structure and improvedas result no catalytic activity [54].Ag-doped ZnO nanoparticles better bactericide than commercial ZnO nano crystals and enhance anti-bacterial activity since doping increase charge transfer resistance. Nano crystallite size was synthesized by pure and nickel doped zinc oxide ($Zn_{1-x}Ni_xO$, x = 0.00, 0.01) powder are successfully synthesized by a simple and low-temperature auto-combustion method. The optical absorption measurements indicate the red shift in the absorption band edge upon nickel doping and band gap energy decreases from 3.21 eV to 3.17eV [55]. Generally, ZnO nanoparticles shows good anti-bacterial activity and doping Ni ion into ZnO Nps had change the Morphology of the ZnO nano particles and then using green synthesis, the reducing agents obtained from plant extract had the power to reduce the synthesized nanoparticles, this enhance both anti-bacterial and surface area to volume ratio[56].even though green synthesis of ZnO Nps has greater merits than Chemical method and doping of Ni into ZnO can change the properties of the sample it was not reported by green method synthesis therefore the researcher was interested to synthesize Ni(II)doped ZnO Nps by *E.abyssinica* bark plant extract for future application

3. METHODOLOGY

3.1 Materials and Methods

3.1.1 Chemicals and Materials

The Chemicals and apparatus that had been used in these synthesis processes were: Nickel nitrate(NN)($Ni(NO_3)_2.6H_2O$),Zinc nitrate(ZN)$Zn(NO_3)_2.6H_2O$;Aluminumfoil, DMSO,gentamacin,80 ml beaker, 250 ml beaker, rod stirrer, magnetic balance, What man no.1 filter paper, funnel large, measuring cylinder, thermometer, Ceramic crucible, Cubits and etc.

3.1.2 Preparation of plant extract

3.1.2.1 Collection of plant for extraction

The bark of the plant *Euphorbia abyssinica (Adaamii in Afaan Oromo) (Qulqwal in Amharic)*was collected in the month of March, 2017 and the plant was collected from; KiremuDistrict, East Wollega, Ethiopia. This is located from Addis Ababa 255 km and 222 km from Jimma. (**Appendix.**5)

3.1.2.2 Preparation of bark extract

The collected *Euphorbia abyssinica*bark plant cleaned with running tap water thoroughly to remove debris and other contaminations, followed; dried in the sun shade and powdered using mortal and pistil. Then in 300 ml beaker, 60 gm of sample was dissolved in distilled water and kept for 24 hrs for extraction under suns hade. Then the extracted filtered by using filter paper (What man number1.filter paper), and stored for further experimental analysis.

3.1.3 Preparation of Nanoparticles (ZnO and Ni ion doped ZnO)

In 75 ml beaker, filtered bark extract (50 ml) and zinc nitrate hexahydrate (ZN) stirred well for 30 minute using magnetic stirrer .Then the mixture was kept for 12 hrs.For partial precipitate and to make paste which was collected through ceramic crucibles. The paste was calcinated for 2 hrs in a muffle furnace at 500 ^{O}C to remove organic impurities. The result of calcinations was collected and stored for characterization this procedure was for synthesis of ZnO Nps. For Ni ion doped ZnO Nps the above procedure was adopted using zinc nitrate hexahydrate with 1-5 % mole of nickel nitrate hexahydrate .This method was developed by optimizing; mass, volume of extract and temperature of calcinations. Thus parameters were selected based on

theirlowestwavelength with sharp peak by Uv-visible spectroscopy and assigned for Ni doping process. Temperature was optimized at 700 ^{0}C and 500^{0}C based on reported literature [57].The volume of extract was 30 ml, 40 ml, 50 ml, 60 ml, and 70 ml. Then 500°C for 2 hrs and 50 ml of extract in 2 gram of Zinc nitrate hexahydrate was selected for all procedure had used in this work. This calcination was done at Jimma University in laboratory of Environmental science department.

3.1.4 Characterization of doped and undoped ZnO Nanoparticles

The synthesized doped and undoped ZnO nanoparticles were characterized by the instruments like UV-visible spectroscopy, FT-IR and XRD.he UV-Vis absorption spectrum was recorded using (Shimadzu UV-Vis 2600) spectro-photometer in the wavelength range of 230 -800 nm and the nano sized doped and undoped of ZnO nanoparticles were determined based on the maximum wavelength and sharp peaks. Therefore, in our work the ZnO and Ni doped ZnO nano particles were same with reported different literatures. The functional group of compound was examined using Fourier transform infrared spectroscopy(FT-IR)and theexistence of different functional groups left after calcinations as capping and stabilization agent those obtained from; alcohols, carboxylic acid, esters in proteins also in metabolism such as terpenoids which the synthesized nano particles was surrounded[58].Structural parameters of doped and undoped ZnO nanoparticles synthesized using *Euphorbia abyssinica* bark plant extract was calculated from the XRD pattern using Scherer equations and instrumentation was done at Addis Ababa University.Andthesamples calcined at 500^{0}C are essential for relatively, complete removal of water and organic molecules from the extractto obtain higher crystallite.

3.1.5Anti-microbial activity

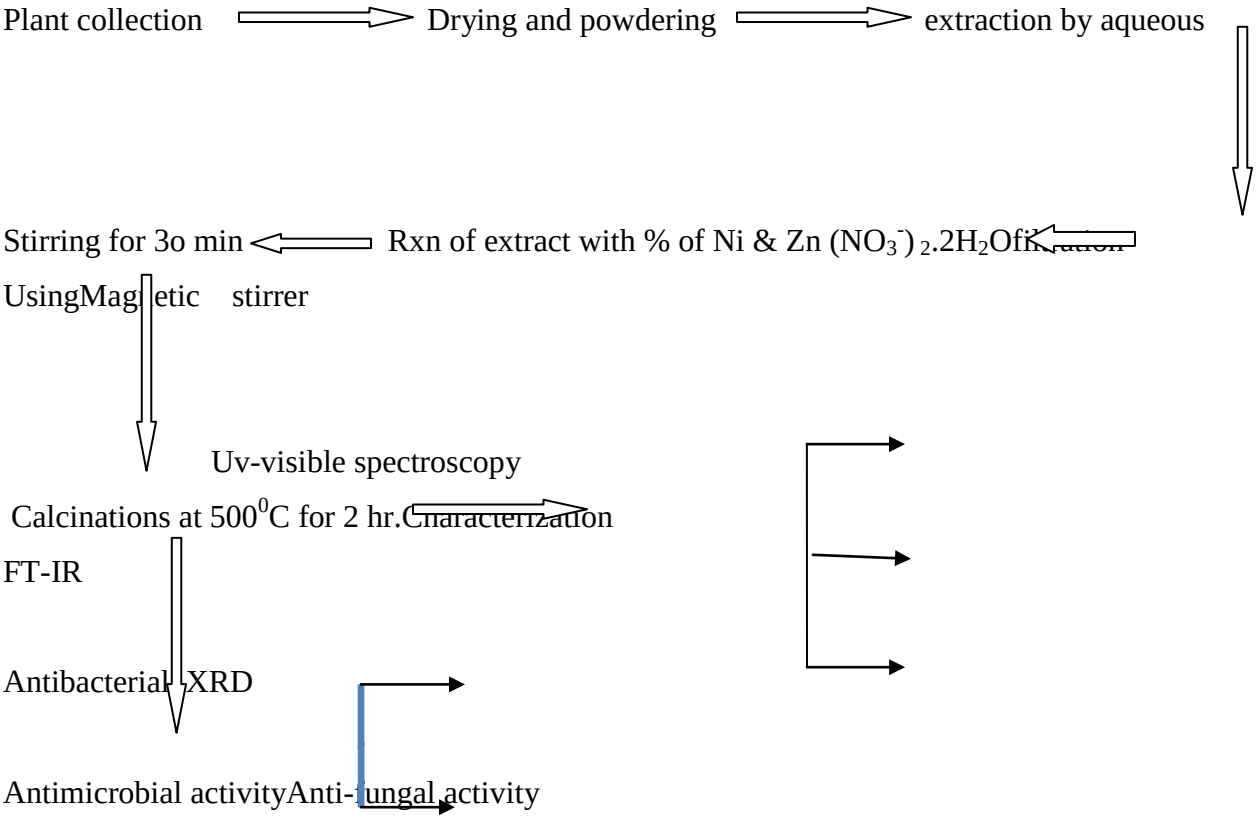
Anti-microbial studies were done at Jimma University in laboratory of Biology Department.

The antibacterial antifungal activity study was conducted using agar disc diffusion method as described byMackeen[59]. The biological screening effects of the synthesized nanoparticles was tested against the bacteria (*staphylococcusaureus*), (*Escherichia coli*), (*Bacillus subtitles*) and (*aspergillum) and (fusariums)* by disc diffusion method using nutrients agar as the medium. All the blank discs was moistened with the solventDMSO was used as negative controls the sample solution of 20 mg/mL each test compounds has been prepared by dissolving the compounds in distilled water and the solutions will be loaded on the wells of the culture and in culture

incubated at 37 °C for 20- 24 hrs for the bacterial culture. During this period, the test solution was diffused and the growth microorganisms were affected.

Hence, the inhibition zones were developed on the plate around the discand the ant microbial activity of Nps was demonstrated by the diameter of the zone of inhibition developed around the sample then the zone of inhibition was measured.Gentamicin and DMSO had been used as a positive control and negative control respectively. The nickel doped and undoped zinc oxide was does partially soluble in DMSO and also in distilled water this solubility was affect themicrobial activity of the samples.

The follow of chart of green method synthesis and characterization of Ni doped ZnO Nps



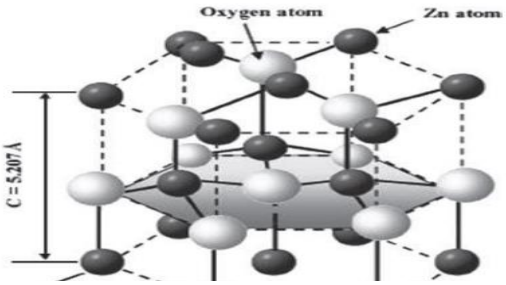


Figure 1. Structure of ZnO Nps adopted [60]

4. RESULTS AND DISCUSSION

In synthesis of doped and undoped ZnO Nps the structural determination was by XRD and Compositional analysis confirmed by FT-IR spectroscopy. Following the procedure listed under methods: nanoparticles were synthesized by the researcher. Thecolour of the sample was changed after calcinations at 500^0C for 2 hrs and in the processes the impurity of organic maters were removed and the white colour of ZnO Nanoparticles for undoped with somewhat greenish white powder of doped ZnO Nanoparticles was prepared .

4.1 Physiochemical screening of *Euphorbia abyssinica* plant barks extract.

4.1.1. Alkaloids test

The acid layer was used for chemical tests of alkaloids Wagner's Tests(solution of Iodine in potassium Iodide):Few drops of Wagner's reagent was added to *Euphorbia abyssinica* bark plant extract and partially brown coloured precipitate was formed which confirms the existence of alkaloids.[61]

4.1.2Flavoinods carbohydrates test

Base solution of 5 ml was added to a portion of the crude extract followed by addition of concentrated H_2SO_4. A yellow coloration had observed in each extract this confirm the presence of flavonoids. The yellow coloration disappeared on standing [62].Also by Alkaline Reagent Test**:** Extracts were treated with few drops of sodium hydroxide solution. Formation of intense yellow colour, which becomes colorless on addition of dilute acid, indicates the presence of flavonoids[63]. Both Fehling A and Fehling B solution were mixed in equal volume. These

reagents are added in crude extract and smoothly boiled. A brick red precipitate is appeared at the bottom of the test tube and indicates the presence of reducing sugar

4.1.3. Phenols test

Ferric chloride Tests; *E.abyssinica* extracts were treated with 3-4 drops of ferric chloride solution and it showed that bluish black colour which indicate the presence of phenols[64]

4.1.4 Saponoids test

Foam Test; Small amount of *E.abyssinica* extract was shaken with little quantity of distilled water and the foam produced with persistence of 10 min confirms the presence of saponoid.

4.1.5 Steroids test

E.byssinica extracts of 5 ml was added in 2 ml of chloroform and followed carful addition of 3 ml conc.H_2O_4 layer of reddish brown color formed which confirms the presence of steroids [65].

4.1.6Tannins Test

E.abyssinica extract of 1 mL was mixed with 10 ml of distilled water and 3 drops of ferric chloride ($FeCl_3$) reagent was added with the filtrate then blue-black coloration green or brownish green colour was formed that indicates the existence of strong tannins [66].Carbohydrates Cardiac glycosidesantimicrobial compounds such as flavonoids and terpenoids are polar constituents which can be extracted using polar solvent systems like those which were used in this study. In this study, flavonoids, together with tannins, alkaloids, saponins, glycosides, Cardiac glycosides, anthrax cenelycosides, amino acids were found in extracts of *E.abyssinica*stembark and latex.Flavonoids, tannins, saponins, sesquiterpenes, and alkaloids are known to be synthesized by plants in response to microbial infection and they serve as defense mechanisms against predation by many microorganisms, insects and herbivores [67-68]. The presence of these metabolites in other*Euphorbia species* has also been reported by [69 -70].

Table 1. Phytochemical screening of *Euphorbia abyssinica* plant bark extract

Number	Chemical constituent	Result of *E.abyssinica* plant barkextract

1	Alkaloids	-
2	Flavoinods	+
3	Carbohydrates	+
4	Phenols	++
5	Saponoids	+
6	Steroids	+
7	Tannins	++

Presence of chemical compounds is: (++) =the most presence; (+) =Less presence; (-) = absent

As it was observed from the above table 1; the *Euphorbia abyssinica*bark extract has mostly;phennolic, tannins and phenols and steroids and saponoids are also found as it was tasted by different physiochemical screening reagents.

Then the existence of these functional groups have used for reducing agent by acting as fuel to synthesis both undoped and doped ZnO nanoparticles

4.2. Synthesis of Ni doped zinc Oxide Nanoparticles

4.2.1 Optimizations of sample.

The parameters of desired procedure was optimized for each of samples like mass, temperature and the volume of extract by measuring the wavelength of the sample for maximumabsorbance and sharpest peak.

4.2.1.1 Optimization of mass

The mass of zinc nitrate hexahydrate used for synthesis was optimized by taking different masses and measuring their respective wave length using Uv-visible spectroscopy and relatively the sample which has less wave length was taken for further synthesis since nano sized structure with less wave length was more interested then we synthesis with different mass of analysts using bark extract.

Table 2. Uv-visible spectroscopy Data of Maximum wavelength for different masses of ZnO nanostructure

Number of samples	Mass of $Zn(NO_3)_2.6H_2O$(in gram)	Mole(mol)	Polarity (in M/L)	Maximum wavelength
1	0.25	0.000084	0.00168	377
2	0.50	0.00108	0.034	366

3	0.75	0.0025	0.05	365
4	1.00	0.0034	0.067	366
5	2.00	0.0067	0.134	358

The values which recorded in table 2 and 3 are measuring the wavelength at highest absorbance for each mass of the sample at constant volume.

Table.3.Maximum absorption of wavelength for different masses of ZnO nano particles

Number of samples	Mass of $Zn(NO_3)_2.6H_2O$ (in gram)	Mole(mol)	Molarities (in M/L)	Maximum wavelength
1	1	0.0034	0.067	376
2	2	0.0067	0.134	366
3	4	0.13	0.268	368
4	6	0.02	0.403	367
5	8	0.027	0.54	373
6	10	0.34	0.67	374

From two trails 2 gm of zinc nitrate was relatively has good lambda max. Also as reported [71].Since as wave length decreased and the graph become sharp the nano size prepared was preferable therefore; after identifying the mass of sample used in synthesis the next step was optimizing volume of extract by taking the mass of the sample as constant then we optimizes the volume of the extract this was optimized by recording the maximum wave length from Uv-vis.

4.2.1.2 Optimization of volume of plant extract

The extracted sample was used for synthesis of pure and doped ZnO using different volume and measuring their respective wavelength and which has sharp peak in Uv-vs spectroscopy was used for further synthesis.

Table4. Uv-visible spectroscopy data for different Volume of extraction by using 2 gram of zinc nitrate hexahydrates

Number of samples	Mass of $Zn(NO_3)_2.6H_2O$ (in gram)	Mole(mol)	Molarities (in M/L)	Maximum wavelength	Volume of Extract
1	2	0.0067	0.22	375	30 ml
2	2	0.0067	0.168	373	40 ml
3	2	0.0067	0.134	372	50 ml
4	2	0.0067	0.112	371	60 ml
5	2	0.0067	0.096	370	70 ml

As it have observed from the maximum wavelength ad their sharpness of peak 50 ml of the sample was selected with average lambda =372 nm for synthesis.

Based on different literatures;500°Cand 700°C for 2 hrs in furnace was selected and the samples were calcinated in this two temperature ranges and their respective wavelength with intensity of peak was recorded by Uv-vis.and sharp peak appearance and relatively small wave length was used for furthermethodsof synthesis. Since as the temperature of calcinations increases the sizes of the sample was increased this may be due to nanopowder agglomeration [72].

During calcinations the colour of brown black samples changed to white powder for undoped ZnO Nps and for nickel doped white brown powder. After the temperature reached 500 °C then heating continued for 2 hrs these heating were used to remove impure organic materials or groups.

Table 5.UV-visible spectroscopy data for different percent by mass of doping nickel nitrate hexahydrate with zinc nitrate hexahydrates

Percent ofdoping (NN)	Mass of $Zn(NO_3)_2.6H_2O$	Mole (ZN)(mol)	Molarities (inM/L)(ZN)(M)	Mass of(NN)	Mole of NN(mol)	Molarities of NN(M)	Maximum wavelength	Volume of Extract
0%	2000mg	0.0067	0.134	0.00 mg	0.00	0.00	351 nm	50 ml
1%	1980 mg	0.00666	0.133	19.5 mg	0.00671	0.00134	352 nm	50 ml
2%	1600 mg	0.0054	0.017	38 mg	0.00013	0.0026	355 nm	50 ml
3%	1940 mg	0.0065	0.13	58 mg	0.00012	0.00392	356 nm	50 ml

4%	1920mg	0.00645	0.129	78.5mg	0.00034	0.0054	357 nm	50 ml
5%	1898 mg	0.0064	0.127	99 mg	0.00034	0.0068	358 nm	50 ml

The UV-Vs. of the extract was measured and its wavelength was recorded and labeled in the following figure and The absorption peak of *Euphorbia Abyssinica* bark extract shows the lambda maximum at 288 nm this was the under finger print region also for zinc nitrate salt the peaks was 242 nm this is may be due to hydroxyl and nitrate ion in solution then after synthesis was done the peak of ZnO nanoparticles and its doping was differ in absorption of Uv-visible spectroscopy.

4.3. Discussion of instrumental analysis for synthesized samples

4.3.1 UV-visible Spectroscopy of undoped and Ni doped ZnO nano particles.

From the UV-VIS spectra (**Appendix1**; 1.1-1.8), different value of wavelength was observed that indicates as Ni doped and undoped ZnO was synthesized. It was reported that as the physiochemical,like phenols, steroids and tannins were working as reducing agent and responsible for conversion of salt of zinc into ZnO nanoparticles as reported [73]and during exposure to plant extracts and calcinations were observed as result the colour change is due to surface Plasmon resonance phenomenons. Hence the metal nanoparticle has free electrons which gives SPR absorption bands ; due to the combined vibrations of electrons of metal in the resonance with light wave that the sharp peak for zinc oxide nanoparticle observed at 351 nm. Also from different literature it was found that sharp peak for ZnO nanoparticles at 358 nm and 350 to 380 nm [74] we confirm that *Euphorbia abyssinica* bark plant extract has more power to reduce zinc oxide ion into zinc oxide nanoparticles.With increasing the mole of the doping, absorption peak was slightly decreased and the peak which appeared at 288 cm^{-1} for extract was completely disappeared and the synthesized sample has with similar of reported before.

Table 6.Uv-isible spectroscopy of doped and undoped ZnO Nps with their plant extract

Samples of doped and undoped ZnO nps	0 %	1 %	2 %	3%	4 %	5 %	extract
Wavelength In nm	351	352	355	356	357	358	288

This changing in wave number and peak sharpness were as result of doping. Generally the wavelength confirms that doping was succeeded with replacement of Zn ionby Ni ion with to some extent differs of wavelength. From different literature, it was observed that asNi doping concentration were increased; wave length decreased and increasing more Ni ion decrease the wavelength of the sample [75]. The band gap for synthesized doped and undoped ZnO Nps were tabulated in table 9 below, indicating that as concentration of doping increase the band gap decreased.

4.3.2 FT-IR Interpretations

FT-IR analysis confirms the functional groups for different organic and inorganic compounds. The metal oxides absorption peaks range less than 1000 cm^{-1} and for doped and undoped Zn-O Nps the bands were between 400 $cm^{-1}$to 400cm^{-1}as reported [76].As it was labeled in (**Appendixb**2:2.1-2.6) their stretching vibration of different functional groups was observed.

FT-IR result was observed clearly that the extract had varies functional groups in alcohol, carboxylic acid, ether, phenols, tannins and ketone which act in capping and stabilizing agents of synthesized nanoparticles[77].The weak stretching of Zn-O and Ni-O Nps were support by[78].Generally due to inter ionic vibrations, Metal oxide give bands below 1000 $cm^{-1}$and in this work the peak appears between 400 cm^{-1} and 600 cm^{-1} as shown in stretching of Zn-O with doped and undoped nanoparticles [79-82].Therefore; as it was observed; doping nickel ion their absorption peaks were between 444 to 457 cm^{-1}.This indicate that the undoped ZnO and doped ZnO Nps has relatively similar bands and we confirm that Ni ion was inserted to Zn lattice or replaced by Zn metal since there size was almost similar .Then this peak shifts to higher frequency side for 1% and 5% Nidoped ZnO Nps then for 3% and 4% Ni doped ZnO Nps shifts toward lower frequency this was due to Ni doped into ZnO and leads some structural changes by doping Niion [83] and the doped zinc oxide nanoparticles are different in absorption band since Ni ion and Zn ion was slightly differ in size.

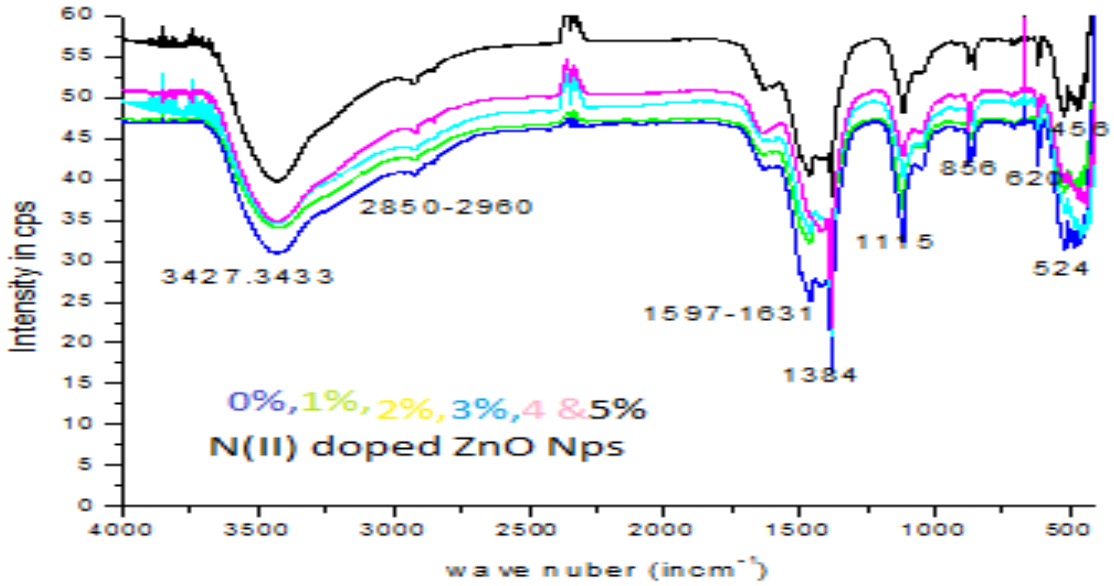


Figure 2. FT-IR spectra for (0% - 5%) Ni doped ZnO Nps

The spectra shows the stretching of some functional groups almost the same for all but there was some change in wave length because of doping new substance.

Table7. FT-IRInterpretations of Ni ion doped and Undoped ZnO Nps

Functional groups or bond vibrations	Wave number in cm^{-1}	explanation for stretching and vibrations
O-H	3584-3700	• Observed for 0%, 4% and 5% doped ZnO Nps Stretching in alcohol free[84]
O-H	3427-3433	• Due to stretching vibration of hydroxyl groups in alcohol intermolecular bond and water absorbed by sample it exist in all samples [85]
C=C	2850-2960	• Stretching in all samples as result of conjugated alkene
C-H	1597-1631	• Stretching vibration in alkane
C-N NO_3^-	at 1384(1350-1450)	• Stretching in amine and nitrate groups
C-C	1114-1117	• Stretching in alkane
C=C	817-873	• bending in alkene

Zn-O Ni-O	619-717	• Weak stretching in ZnO and NiO Nps
Zn-O	524	• Except 95 %,stretching vibration of ZnO Nps[86]
Ni-O	444-457	• Stretching vibration of Ni ion doped

Different literatures support that as Ni-O stretching observed for the ZnO peaks at 480 cm^{-1}dueto Ni ion present in the samples and 481 cm^{-1} stretching vibration with average crystalline size is 11.9 nm for ZnO Nps[87-88]

4.3.3. X-ray diffraction study (XRD)

X-ray diffraction study confirms that the synthesized material doped and undoped ZnO Nps was with hexagonal wurtzite phase and the entire diffraction peaks are in agreement with the standard JCPDS data (card No. 36-1451). The X-ray diffraction data were recorded by using Cu Kα radiation (λ=1.5406 A°).The average crystallite size was calculated from the Debye–Scherer formula [89-90].

Grain size calculation and d-spacing or d (hkl)

Scherrer equation = $D = k\lambda/\beta_{1/2}\cos\Theta$ ---------- (1)

Where*k* denotes the Scherer constant (the shape factor of the average crystallite and can be considered $k = 0.90$ [91] $\lambda = 0.15406$ nm is the wavelength of the incident Cu $K\alpha$ radiation; *r*epresents the full-width at half-maximum (FWHM) of the respective peak.

The XRD pattern of synthesized sample of doped and undoped ZnONps was in the range of 2Θ=20°to 70°their grain size were shown in (**Appendix3:** 3.1.1-3.6.1 &3.1.2-3.6.2.).All evident peaks could be indexed as the ZnO wurtzite structure (JCPDS Data Card No: 36-1451). Wurtzite lattice parameters such as the values of d which is distance between adjacent planes in the Miller indices (hkl)

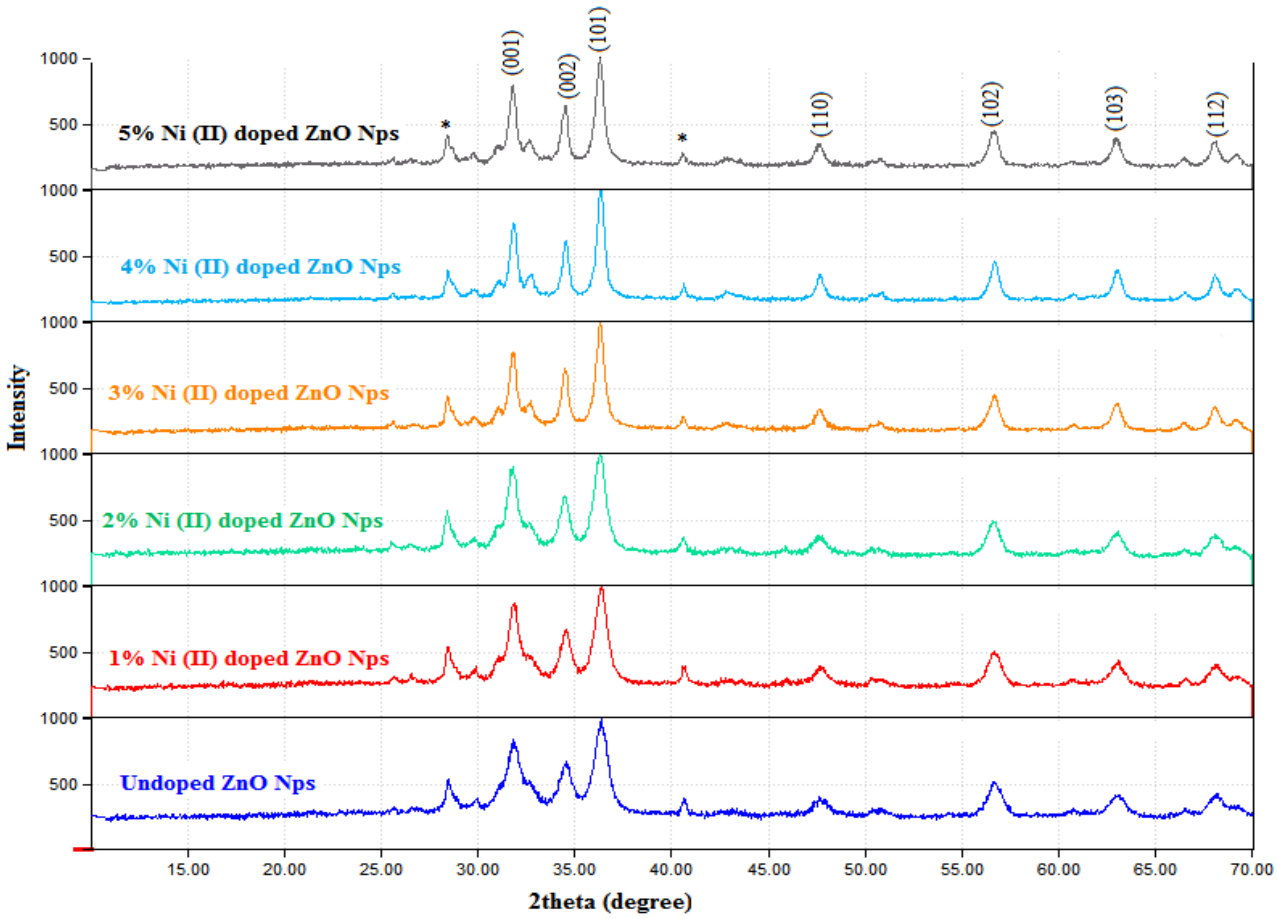


Figure 3.XRD spectra of Ni (II) doped and undoped ZnO Nps

Table 8 . Crystal size calculated for doped and undoped ZnO Nps

% Ni(II)doped ZnO Nps	2Θ(degree)	cos Θ	*B*, FWHM (radian)	D = kλ/$\beta_{1/2}$cos Θ (in nm)
0%	36.37	0.95	0.018	8.11
1%	36.52	0.949	0.017	8.60
2%	36.21	0.95	0.016	9.10
3%	36.34	0.95	0.0105	13.89
4%	36.36	0.95	0.0105	13.90
5%	36.33	0.95	0.0098	14.89

(Calculated from the Bragg Equation, $\lambda=2d\sin\Theta$), lattice constants *a*, *b*, and *c*, inter- planar angle and unit cell volumes are calculated from the Lattice Geometry equation [92].

The lattice parameters of the powders calcined at 500°c.

$$\sin\theta = \lambda/2a\,(h^2+k^2+l^2)^{1/2} \text{----------} (2)$$

$$a=\lambda/(3)^{1/2}\sin\Theta hkl. \text{ ----------} (3)$$

$$c= \lambda/\sin\Theta \text{----------} (4)$$

$$d_{hkl} = 1/((4/3)^{1/2}(h^2+k^2+hk)/a^2+l^2/c^2) \text{ ----------} (5)$$

Also The XRD patterns of the precursor of powder were corresponded the patterns of JCPDS CardNo.891397. The precursor of powder has been the lattice parameters *a* = *b* = 3.2481 Å, *c* = 5.2049 Å indicate hexagonal structure for (100) and (002) was reported[93],Then based o the above formula We calculate the grain size, or d-spacing and hkl value and it was filed in the following table with their respective figures. As it was observed from 2ɵ angle Ni ion was doped to ZnO nanoparticles hence with no change in hexagonal Wurtzite structure of ZnO nanoparticles because no other peaks for Ni metal or NiO nonoparticles were observed for NiO nanoparticles average grain size 44 nm and for Ni metal synthesized by *A. indicia* and *p.guajava* plant extract[94].The Bragg's diffraction angle at intensity (101) reflection observed slight shift toward higher waves relative to un doped ZnO nanoparticles which has been that indicated that N^{2+} doped to ZnO lattice and Ni metal may be doped into ZnO nanoparticles [95].

We can calculate the bond length (L) = $((a^2/3 + (1/2-u)^2{}_C{}^2)^{1/2}$ ---------- (6)

And (u) positional parameter in Wurtzite structure which related to c/a ratio

$$u=a^2/3c^2 + 0.25 \text{----------} (7)$$

It is the measure of the amount by which each atom is displaced with respect 'c' axis.Since the two main properties extracted from peak width analysis are the crystallite size and lattice strain. Crystallite size is a measure of the size of coherently diffracting domain. The crystallite size of the particles is not generally the same as the particle size due to the presence of polycrystalline aggregates therefore, the peaks observed shows that the different size of particles but we focus on the largest intensity.

From theXRDspectrum the parameters of unit cell at miller index of (101) was calculated as the following for % of Ni doped and undoped ZnO Nps.

0% Ni doped ZnO nanostructure, at 2θ= 36.37(deg) a=λ/(3)1/2sinθhkl. When λ=1.5406Å,

1.5406 Å/ $(3)^{1/2}$ sin18.2 deg = 1.5406Å/0.541

a =2.848 Å

c =λ/sin**,** 1.5406 Å/sin18.2 deg. = 1.5406 Å/0.312

a = 4.935 Å

1 % Ni doped ZnO nanostructure at 2θ = 36.52 (deg)

a and care; a=λ/ (3)1/2sinΘhkl. When λ=1.5406Å, 1.5406 Å/ $(3)^{1/2}$ sin18.26 deg

a = 1.5406 Å/0.541

a = 2.848 Å

c= λ/sin Θ, 1.5406 Å/sin18.26 deg. = 1.5406 Å/0.312

c = 4.935 Å

2% Ni doped of ZnO nanostructure at 2θ = 36.32(deg) the parameters are;

a=λ/ (3)1/2sinΘhkl. When λ=1.5406Å, 1.5406 Å/ $(3)^{1/2}$ sin18.17 deg

= 1.5406 Å/0.3118

a = 2.852 Å and

c= λ/sinΘ, 1.5406 Å/sin18.17 deg. = 1.5406 Å/0.3118 c = 4.940 Å

3 % Ni doped ZnO nanostructure2θ = 36.52(deg)

The unit cell parameters was a and c are;

a=λ/ (3)1/2sinΘhkl. When λ=1.5406Å, 1.5406 Å/ $(3)^{1/2}$ sin18.08 deg = 1.5406 Å/0.296

a = 2.999 Å and c= λ/sinΘ, 1.5406 Å/sin18.08 deg. = 1.5406Å/0.296

c = 5.195 Å

4% Ni doped ZnO nanostructure2Θ = 36.36 (deg)When λ=1.5406Å, 1.5406 Å/ $(3)^{1/2}$ sin18.18 deg = 1.5406 Å/0.296

a = 2.999 Å and c= λ/sinΘ, 1.5406 Å/sin18.08 deg. = 1.5406Å/0.296

c=5.195Å

And for 5% Ni doped ZnO nanostructure of 2θ = 36.16(deg)

a=λ/ $(3)^{1/2}$sinΘhkl. When λ=1.5406Å, 1.5406 Å/ $(3)^{1/2}$ sin18.16 deg = 1.5406 Å/0.3117

a = 2.854 Å and

c= λ/sinϴ, 1.5406 Å/sin18.16 deg.

= 1.5406Å/0.3117, c = 4.943Å

For sample 1 the volume and band gap of sample is calculated by $V= (3)^{1/2}/2 \text{ X } a^2c = 34.69Å^3$, band gap (eV) = 1240/λ = 1240/351nm = 3.53 eV.

Then for samples; 0%,1%,2%,3%,4, and 5% are 3.53,3.52,3.49,3.48,3.47 ad 3.46 eV respectively. These decreases in band gab may as the result of less quantum confinement within the nanoparticles [96]

Also the band gap can calculated using the formula (eV) = h*C/λ---------- (8)

Where; h = Planks constant = 6.626 x 10-34 Joules sec, λ = wavelength each samples in nm

C = Speed of light = 3.0 x 108 meter/sec and eV = 1.6 X 10^{-19} Joules (conversion factor

The following calculation was at miller index (101)

For sample 2the volume and band gap of sample is calculated by $V= (3)^{1/2}/2 \text{ X } a^2c$ = (0.866 X 8.116 X4.935) $Å34.68Å^3$ and band gap was 3.47 eV then for sample 5 the volume and band gap of sample is calculated by $V= (3)^{1/2}/2 \text{ X } a^2c$ =0.866 X 8.134 X 4.94 =$34.8Å^3$ and band gap = 3.48 eV. Also the amount by which each atom is displaced with respect 'c' axis was calculated for the above table 9 for sample 1; $u=a^2/3c^2 + 0.25$, $(2.849)^2/3X (4.935)^2 +0.25 = 0.361$.

Forsample 2.The same since their& c are equal. For sample 3, u = 0.3611 and for sample 4,

$u = (2.999)^2/ 3 \text{ X } (5.195)^2 + 0.25 = 0.261$ and for sample 6,

$u = (2.854)^2/ 3X (4.943)2 +0.25 = 0.2611$ and the bond length for sample1 was based on equation (6) above for Zn-O the above calculation was for peak 101 which shows highest peak from the other peaks.

$(L) = ((a^2/3 + (1/2-u)^2{}_C{}^2)^{1/2}$

$L= ((2.849)^2/3 + (4.935)^2 \text{ X } (0.5 – 0.361)^2)^{1/2}$

L = 3.763.

Band gap (eV) = 1240/ λ ---------- (6)

Where, λ = maximum wavelength absorbed in nm

the values are calculated, for undoped ZnONps 4.53 eV and for doped ZnO Nps 3.52 eV,3.49eV,3.48 eV,3.47eV and 3.46eV and found for 1%,2%,3%,4% and 5% Ni doped ZnO Nps respectively. And as doping increase the band gap of sample was decreased. This is may be due to distortion in the host ZnO lattice by foreign impurities of Ni (II) and quantum size effects on the electronic energy bands of semiconductors [97].

Table 9. Data of bandgap and crystal structure for their unit cell parameters of Ni (II) doped and doped Un doped ZnO Nps at miller index (101)

#samples	Percent of Doping	Grain size in(nm)	wavelength in nm)	Lattice parameters in Å	u in ((KJm^{-3})	c/a	Band gap in (eV)	V (Å^3)
1	O%Ni doped ZnO Nps	8.11	351	a=2.849 c=4.935	0.361	1.73	3.53	34.69
2	1%Nidoped ZnO Nps	8.60	352	a=2.849 c=4.935	0.361	1.73	3.52	34.68
3	2% Ni doped ZnO Nps	9.10	355	a=2.852 c=4.940	0.361	1.73	3.49	46.40
4	3 % Ni doped ZnO Nps	13.89	356	a=2.999 c=5.195	0.261	1.73	3.48	40.46
5	4%Ni doped ZnO Nps	13.90	357	a=2.852 c=4.94	0.361	1.73	3.47	34.80
	5 % Ni doped ZnO Nps	14.90	358	a=2.854 c=4.943	0.261	1.73	3.46	34.87

This is bond length of Zn-O for sample 1. For the other samples it seems the same procedure. The approximately constant ratio of Ni doped ZnO of a/c= 1.73 as tabulated in table 9, that suggests, there is negligible variation in the ratio of c/a with increasing dopant concentration. this suggests ions are incorporated in to ZnO lattice without altering over all structure[98]The prominent peaks obtained for doped and undoped samples corresponding to the diffraction planes {001}, {100), {002},{101}, {102}, {110}, {103} and {112} agree well with the JCPDS Card No. 36-1451.The prepared doped and undoped material was of hexagonal wurtzite crystalline structure. Presence of several peaks indicates random orientation of the crystallites, confirming the hexagonal Wurtzite structure the crystal phase information was characterized from2Θ= 20°-70° by XRD (Shimadzu 610) with Cu Kα (λ = 0.15406 nm) radiation

Table 10.JCPDS Card No. 36-1451

JCPDS	Hkl	100	002	101	102	110	103	112
36-1451	2Ɵ(0)	31.77	34.422	34.353	47.539	56.603	62.864	67.961

JCPDS Card No. 36-1451 as it was reported by [99] then in our work the two theta values were similar with reported value.

4.4Anti-microbial activity

Enterococcus facials,*Bacillussubtitles*, and*Escheichia acoli*micro-organisms by disk diffusion method in accordance with the procedure reported and described by [100].This method is a mean of measuring the effects of an antimicrobial agent on bacterial and fungal growth in a culture. Muller-Hinton Agar (MHA) powder was used as a culture medium for microbial. Culture medium was prepared using 20 g of agar was dissolved into 500 ml of distilled water, and then a transparent brown solvent was achieved via boiling the solution. The MHA medium 20 ml was sterilized at 120 °C for about 1 hr in autoclave, cooled to room temperature, and then poured into sterilized Petri dishes (15–90 mm). After cooling over 24 hr, the bacteria and fungus were swabbed uniformly across the culture plate. To evaluate the microbial activity, 40 μL of each of the samples was dropped moderately on disks' surface using a sampler, then no colonies would grow and size of inhibition zone measures the efficiency of sample. A more effective sample produces a larger clear area around the disk. All tests were done under laminar flow hood. Finally, all Petri dishes containing bacteria and antibacterial reagents were incubated at 37 °C for 24 h. At the end of incubation period, the diameters of inhibition zones formed around disks were determined and measured in mm. The results concerning microgram activity were expressed as strong activity (>11 mm), moderate activity (7–11 mm), weak activity (6 mm), or no activity (inhibition zone < 6 mm) and the diameter of inhibition zone was as observed in table below the positive control was gentamicin have the minimum 13mm and maximum 15 mm and from the gram positive bacteria *Staphylococcus*

*aureus andAspergillum and fusarium*fungus,was the highest inhibited for doped zinc oxide nanoparticles and for gram negative also doped one with *Escherichiacoli* bacteriain both bacteria and 5 % Ni doped ZnO Nps show good antibacterial activity with greater than 11 mm inhibiter for 4%, 3%and 2%Ni (II) doped ZnONpswas show good microbial activities and generally doped ZnO Nps had improved antibacterial activity and antifungal activity.

Table11. Antibacterial activities with their inhibition zone of undoped and doped ZnO nanoparticles

% of Ni doped ZnO Nps	Enterococcus Faecalis	*Staphylococcus aureus*	*Bacillus subtilis*	*Escherichia coli*
0 % ZnONps	11	11	10	11
1 % ZnO Nps	10	10	9	9
2 %ZnO Nps	12	12	12	10
3 %ZnO Nps	12	12	12	12
4 %ZnO	13	14	12	11
5 %ZnO	13	13	13	12
Gentamicin	18	19	18	16
DMSO	NI	NI	NI	NI

Table12. Antifungal activity with their inhibition zone of undoped and doped ZnO Nps

% of Ni dopedZnONps	***Aspergillum***	***Furasium***
0 %ZnO Nps	10	11
1 %ZnO Nps	12	11
2 %ZnO Nps	11	10
3 %ZnO Nps	12	12
4 %ZnO	13	13
5%ZnO	13	15

Gentamicin	18	17
DMSO	NI	NI

NI =no inhibiter

5. Conclusion and Recommendation

5.1 Conclusion

In the present work, the synthesized Ni (II) doped ZnO Nps; by simple green methods of down to top process which is eco-friendly, inexpensive, non-toxic, less time consumption and has no environmental side effect product. The synthesized sample was characterized and instruments confirm that Ni doped and undoped ZnO Nps by green method; FT-IR spectra, the stretching peaks appeared between 3433-817 $cm^{-1}$and 439 cm^{-1} to 524 $cm^{-1}$for organic maters and Ni doped and undopedZnONps respectively which used for stabilizing and capping agents.

From XRD spectra the size and unit cell parameters of sample was calculated and the structure of sample agree with reported literature and possess hexagonal wurtzite with grain size of 8.11,8.60, 9.10,13.89,13.90 and 14.90 nm for 0%,1%,3%,4%, and 5% of Ni(II) doped ZnO Nps

respectively this was calculated at miller index of (101)Ni ion doped and undoped ZnO Nps as it was observed from XRD, the structure of the sample were hexagonal Wurtzite and the synthesized has white for undoped ZnO Nps and white brown colour for N(II) doped ZnO Nps.
The band gab of the sample was calculated and showed, as concentration of Ni (II) doped increased the band gab was decreased this enhance the conductivity of the original substances
Also the diffraction peak of each sample was strong sharp and narrow indicating that good crystallite of Ni doped ZnO Nps [101] this increase of concentration of the sample decrease band gab leads to transfer electrons from valance band to conduction band.

Finally the antimicrobial activity of sample was studied and for 4% and 5% doped ZnO Nps show good anti-fungal activity and for 2%, 3%, 4%, & 5% doped ZnO Nps the activity of them to strains of microbial was showed in good manner. Therefore, Euphorbia abyssinica as both reducing and stabilization or capping agent, Ni (II) doped ZnO Nps was synthesized and their size was in the range of reported by different literatures beforeand with good antimicrobial activity.

5.2 Recommendation

For further work it was recommended that additional characterization for further size determination and morphology of the sample using instruments; SEM, TEM and EDX.
The application of Ni doped ZnO Nps further study for dye degradation, adsorption, semiconductors, forchronic diseases, conductivity etc.
Since Euphorbia abyssinica plant was enriching of secondary metabolites, further study will be recommended by researcher.
While working with this plant, safety will be recommended since its latex cause eye blindness

Reactionmay proposed to synthesize doped and undoped ZnO Nps

Ni doped ZnO Nanoparticles was prepared using Euphorbia *abyssinica* bark plant extract the extract used as reducing agent and stabilization agent or capping agent

Mole % of Nickel and Zinc nitrate hexahydrate react with *Euphorbia abyssinica* bark plant and annealed or calcinations at 500^0C for 2 hr gives the product pure and doped ZnO nanostructure and the proposed step was shown figure 4 below

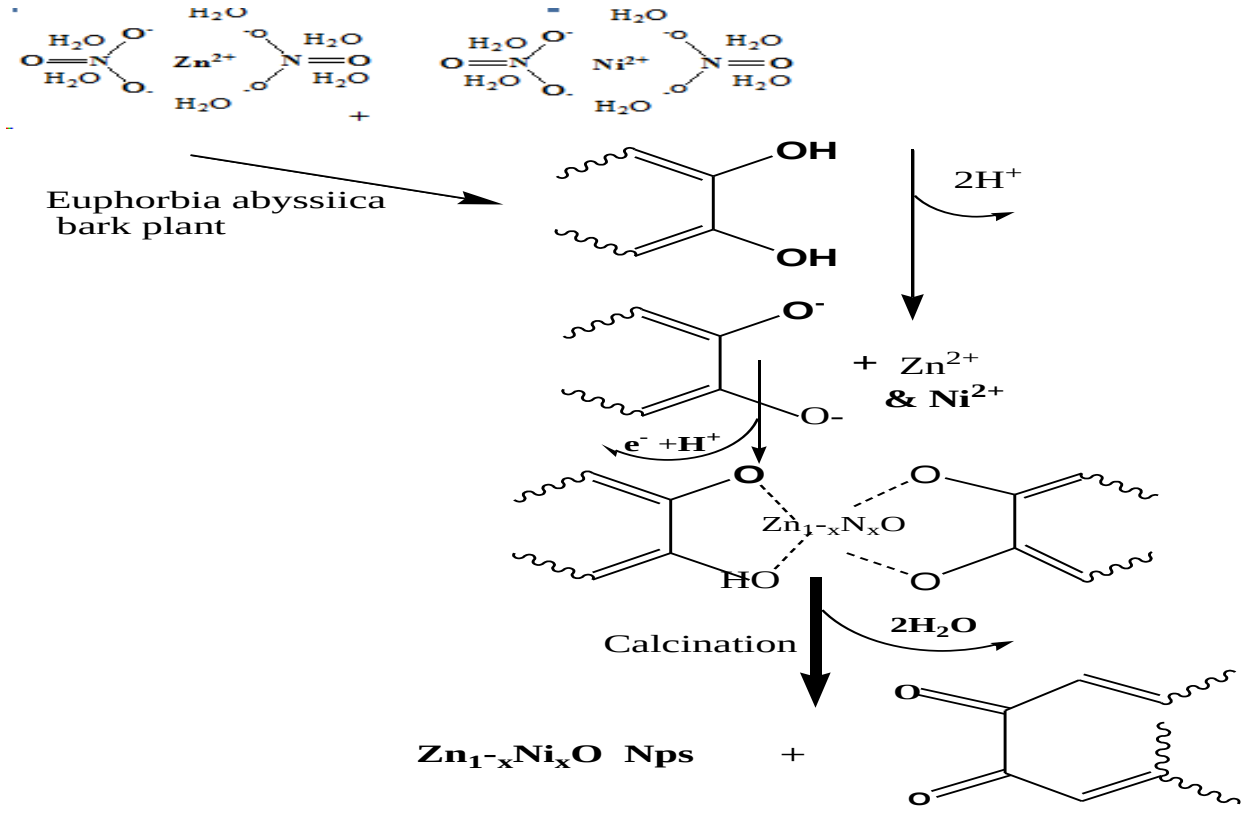


Figure 4. Steps may proposed to synthesize Ni (II) doped and undoped ZnO Nps

This Possible chemical constituent of plant extract responsible for the bio reduction of metal ion was reported [102-104] and chemical constituents of plant extract and uses as stabilization and capping agent in the reaction and used as fuel.

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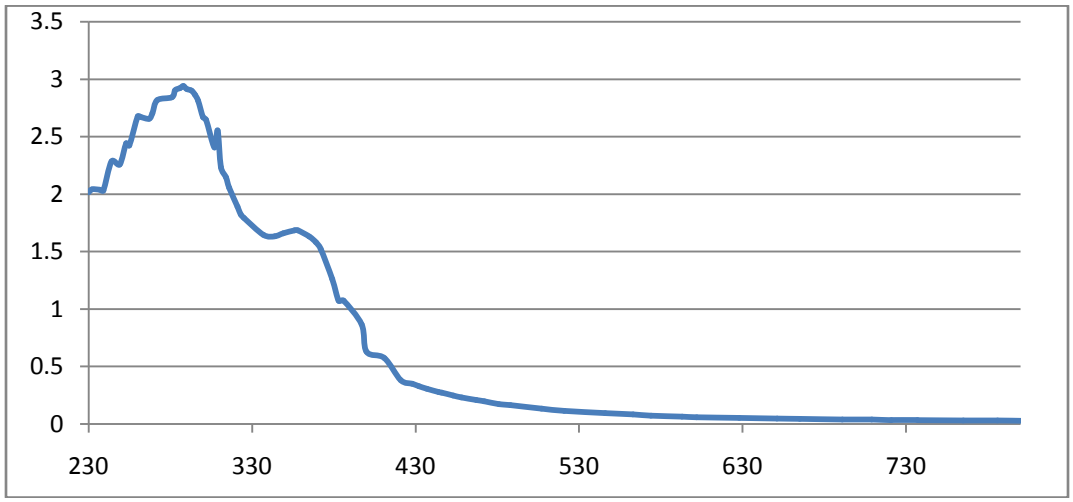
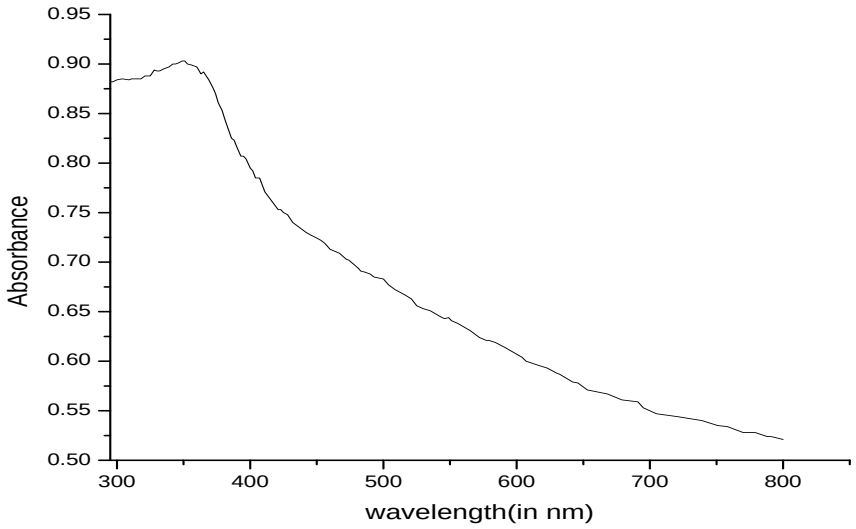
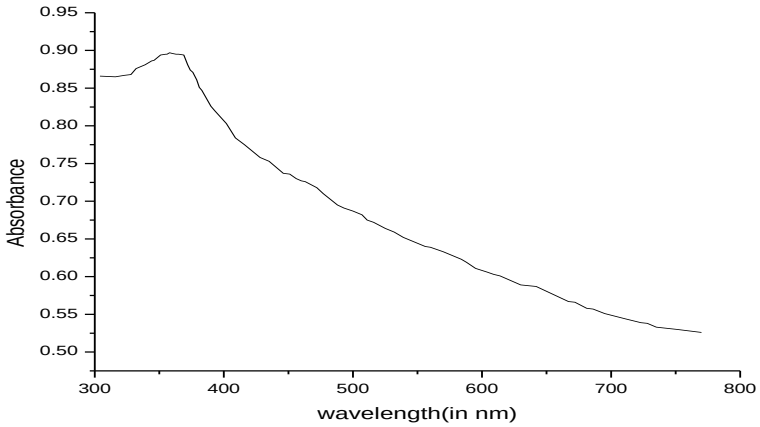
APPENDEX1:



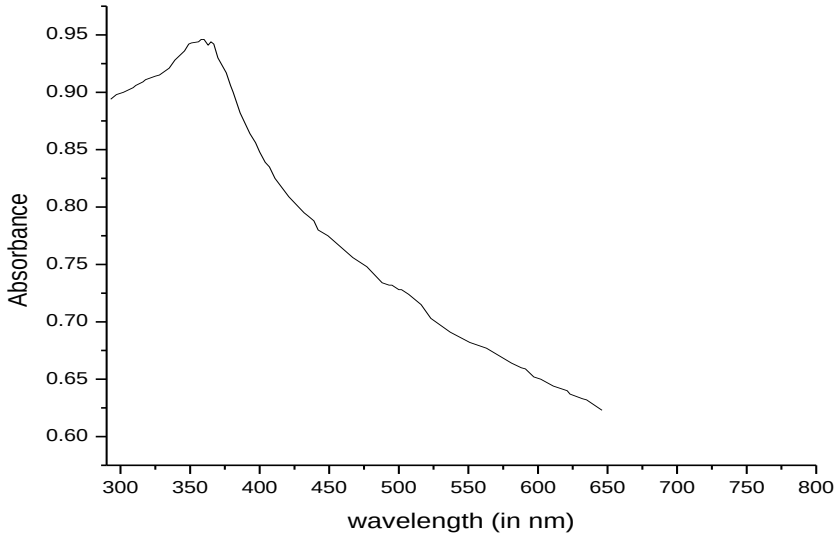
1.1. Uv-visible spectrum of E.abyssinica barks plant extract



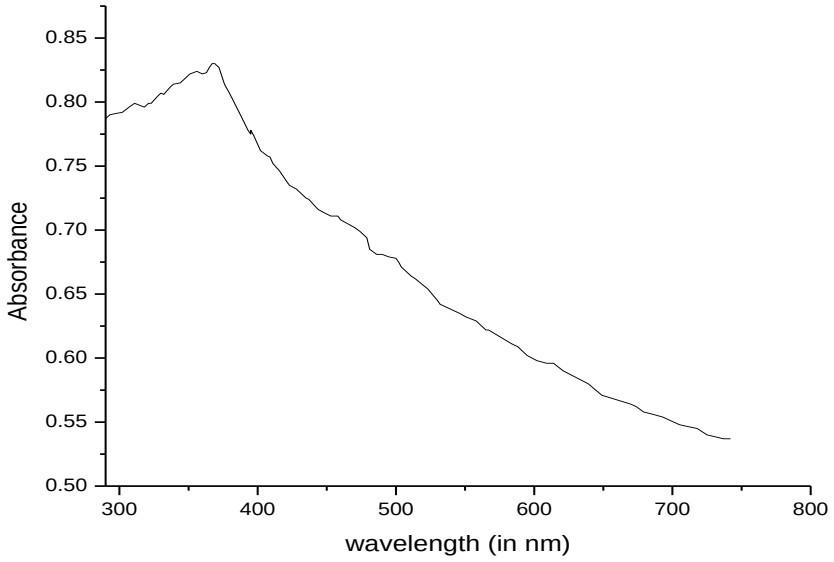
1.2.UV-Vis spectrum of synthesized zinc oxide nanoparticles.of 100 % ZnO nanoparticles



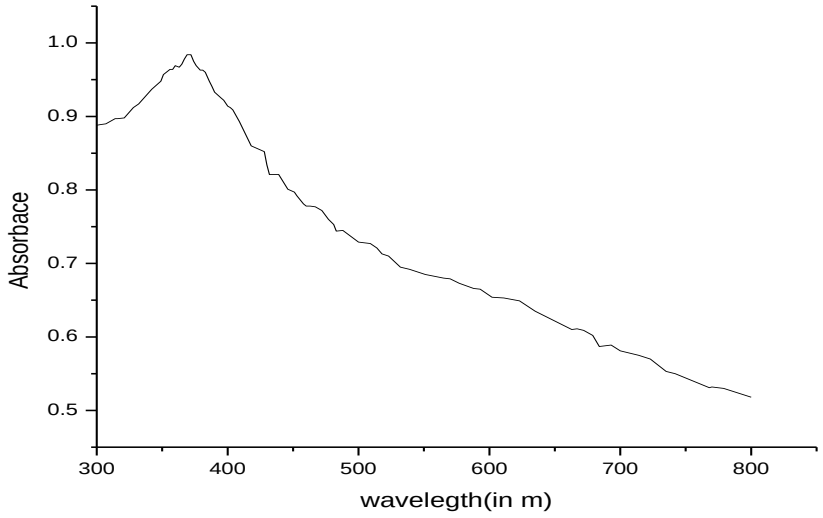
1.3.UV-Vis spectrum of synthesized zinc oxide nanoparticles.of 99 % ZnO nanoparticles



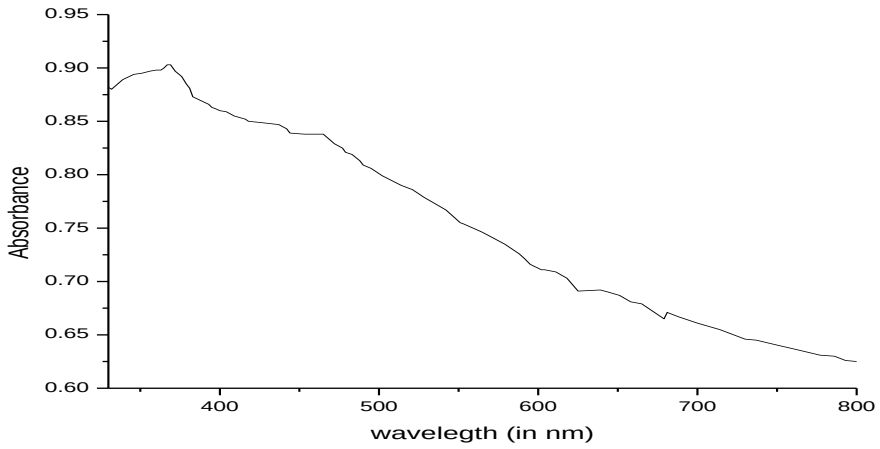
1.4.UV-Vis spectrum of synthesized zinc oxide nanoparticles.of 98% ZnO nanoparticles



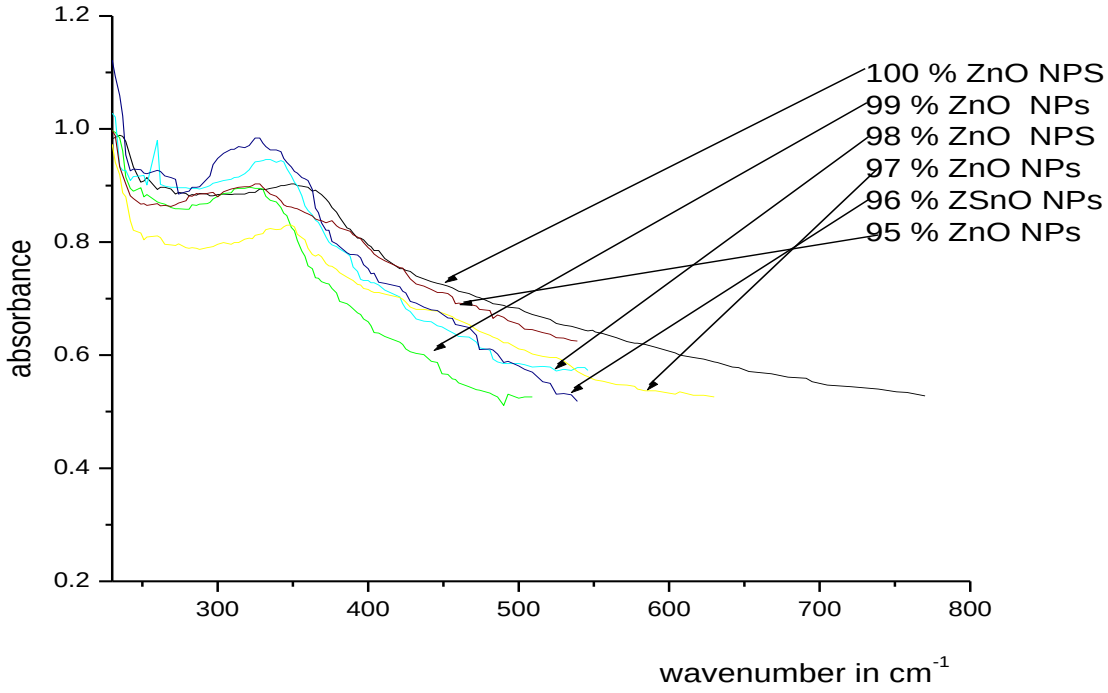
1.5.UV-Vis spectrum of synthesized zinc oxide nanoparticles.of 97 % ZnO nanoparticles



1.6UV-Vis spectrum of synthesized zinc oxide nanoparticles.of 96 % ZnO nanoparticles

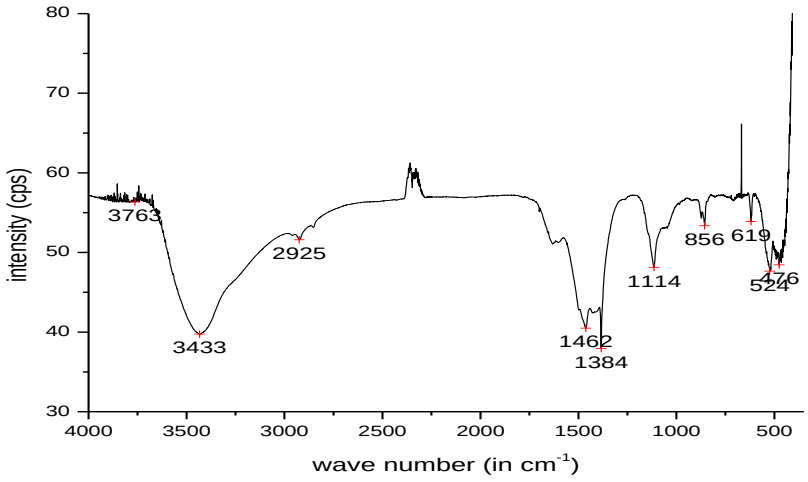


1.7.UV-Vis spectrum of synthesized 95% ZnO nanoparticles



1.8 Uv-visible spectrum of doped and undoped ZnO nanoparticles

APPENDIX 2:



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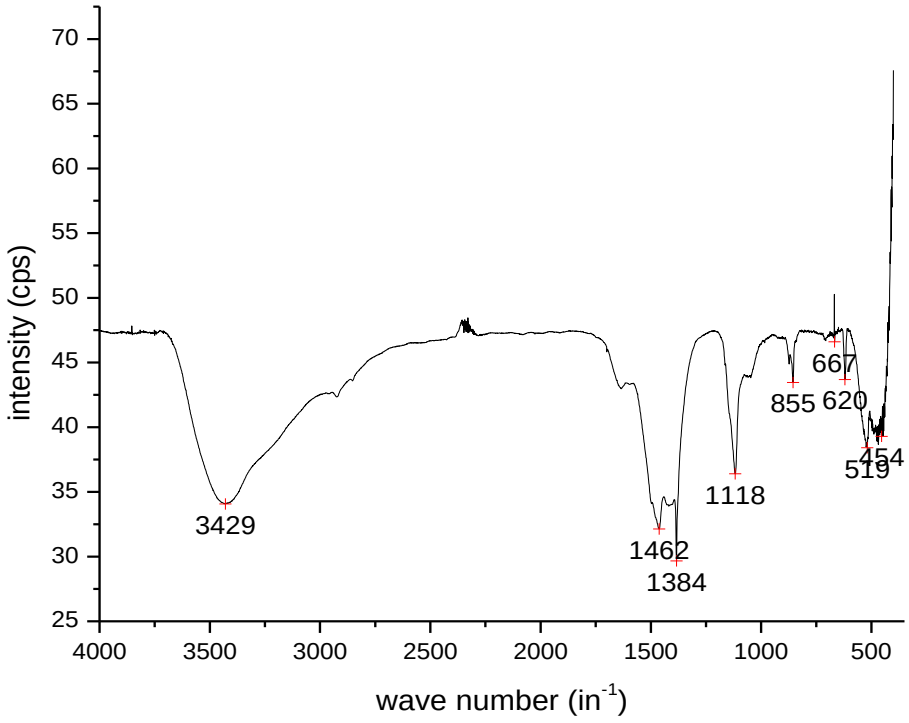
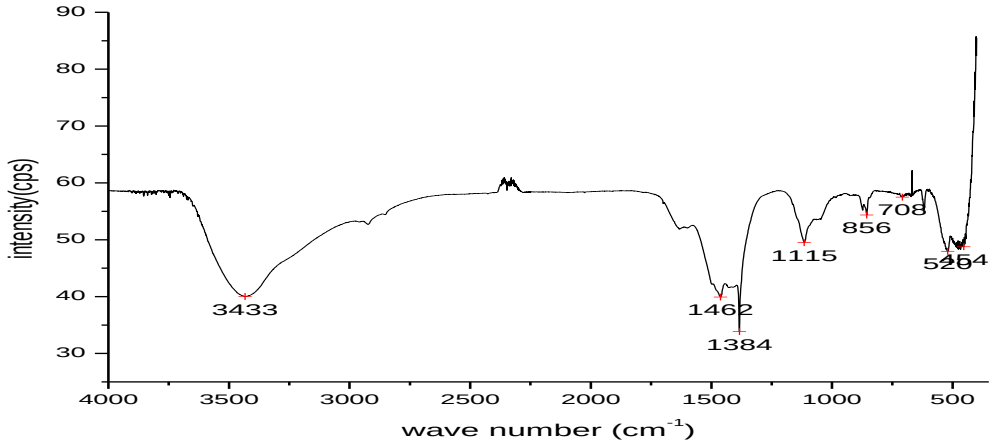
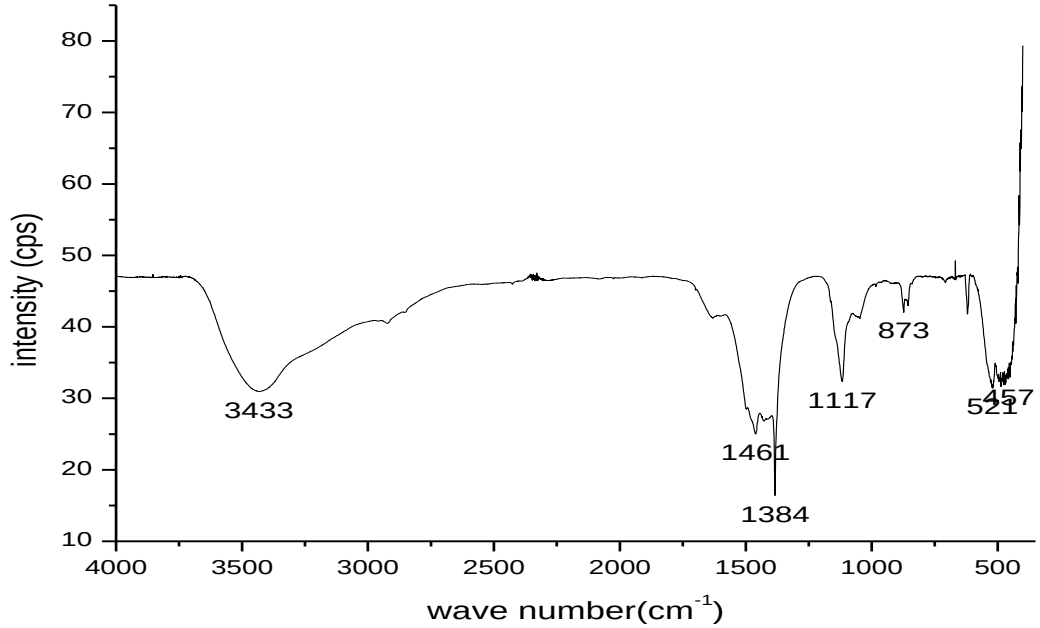
2.1. FT-IR patterns of 0 %Ni doped ZnO nanostructure



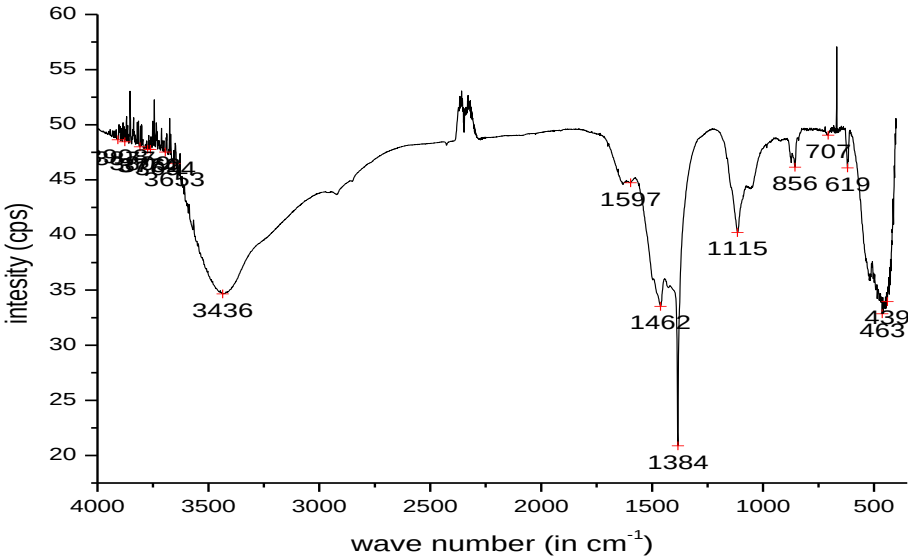
2.2.FT-IR patterns of 0 % Ni doped ZnO nanostructure



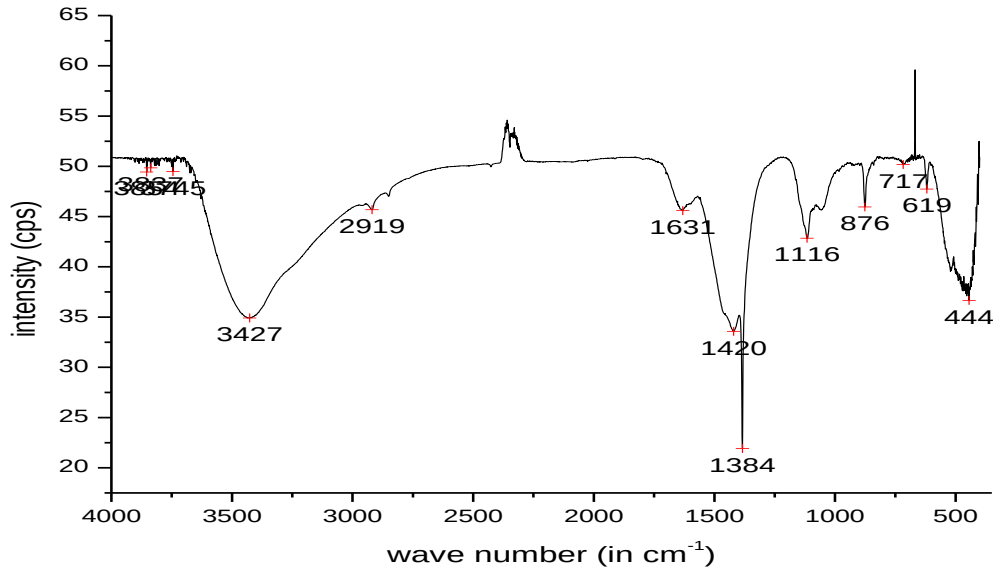
2.3.FT-IR patterns of 2 % Ni doped ZnO nanostructure



2.4. FT-IR patterns of 3 %Ni doped ZnO nanostructure



2.5.FT-IR patterns of 4 % Ni doped ZnO nanostructure

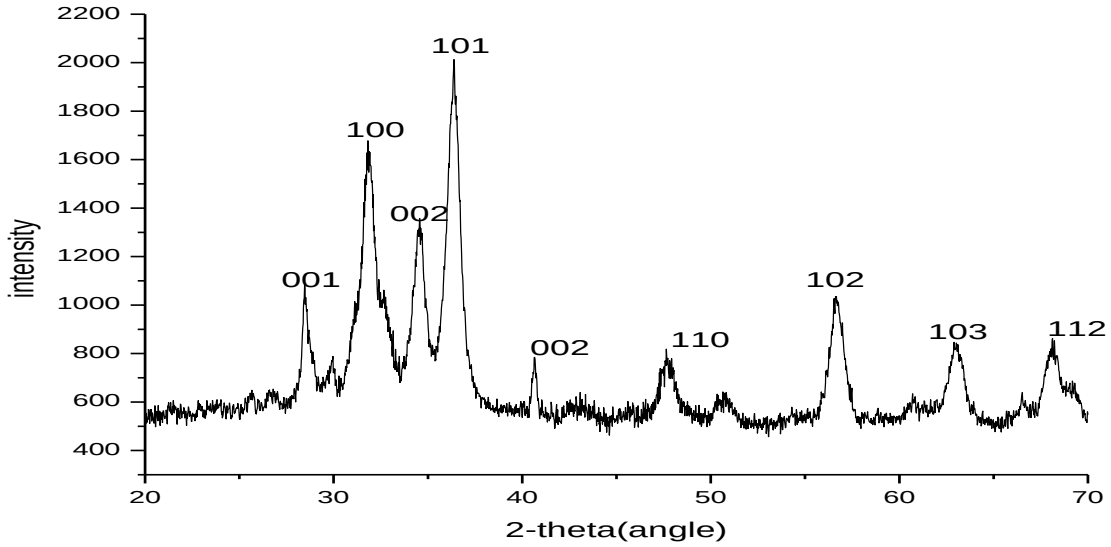


2.6. FT-IR patterns of 5 % Ni doped ZnO nanostructure

APPENDIX3:

3.1.1. XRD spectrum of 0 %Ni doped % ZnO nanostructure and with their unit cell parameters

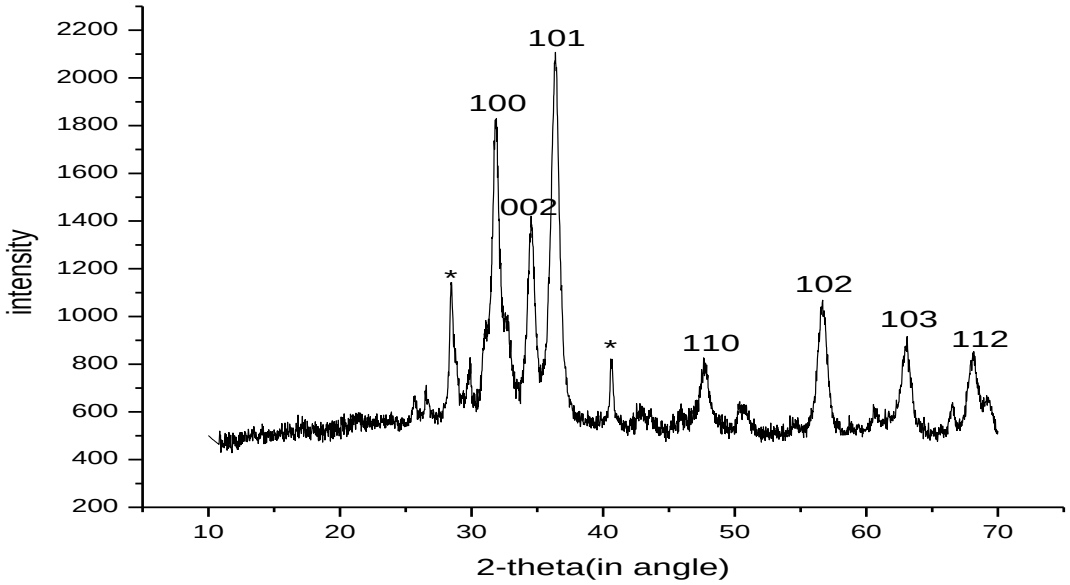
Peaks#	2Θ(°)	d	a	c	h	k	l
1	31.83	2.7927	3.245	5.62	0	0	1
2	34.50	2.5939	2.994	5.186	0	0	2
3	36.37	2.4698	2.849	4.935	1	0	1
4	47.96	1.9025	2.202	3.813	1	1	0
5	56.66	1.3383	1.874	3.246	1	0	2
6	62.96	1.4755	1.705	2.953	1	0	3
7	67.97	1.3383	1.598	2.751	1	1	2



3.1.2. XRD spectrum of 0%Ni doped % ZnO nanostructure and with their unit cell parameters

3.2.1.XRD spectrum of 1% Ni doped ZnO nanostructureand with their unit cell parameters

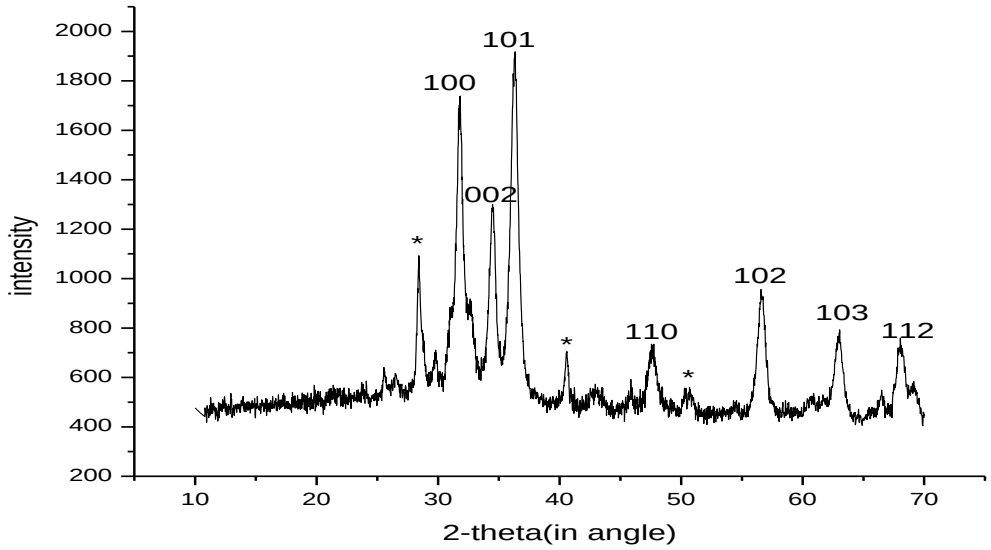
Peaks#	2Θ(°)	d(Å)	a_0	c	h	k	l
1	32.00	2.7950	3.24	3.61	0	0	1
2	34.72	2.5725	2.998	3.192	0	0	2
3	36.52	2.4554	2.849	4.935	1	0	1
4	47.82	1.9047	2.202	3.815	1	1	0
5	56.82	1.3414	1.874	3.245	1	0	2
6	63.13	1.4735	1.704	2.953	1	0	3
7	68.09	1.3763	1.586	2.748	1	1	2



3.2.2. XRD spectrum of 1 %Ni doped % ZnO nanostructure and with their unit cell parameters

3.3.1.XRD spectrum of 2%Ni doped ZnO nanostructureand with their unit cell parameters

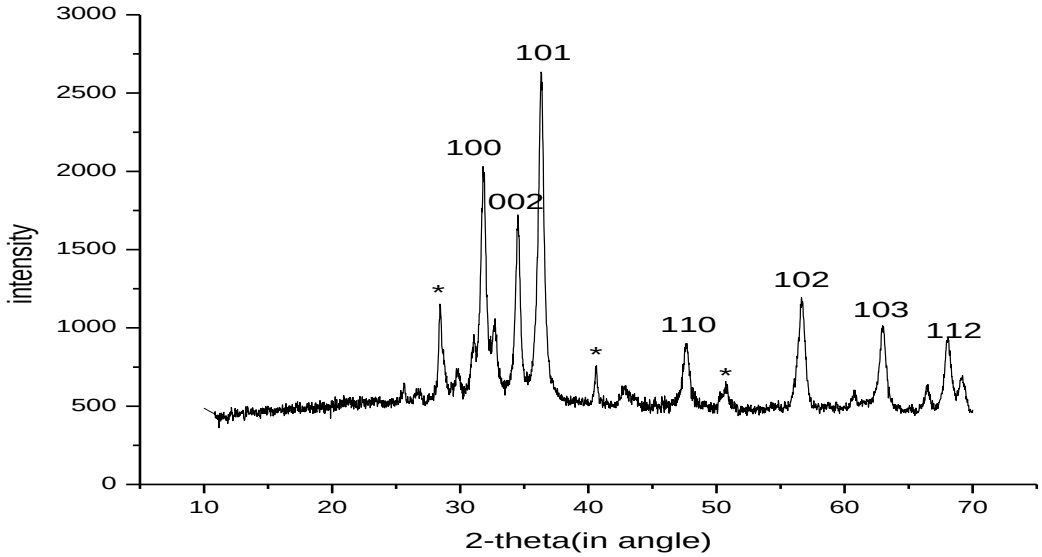
Peaks#	2Θ(°)	d(Å)	a (Å)	c (Å)	h	k	l
1	31.66	2.81	3.247	5.63	0	0	1
2	34.39	2.598	3.009	5.198	0	0	2
3	36.21	2.47	2.852	4.940	1	0	1
4	47.81	1.912	2.206	3.821	1	1	0
5	56.54	1.626	1.8771	3.251	1	0	2
6	62.93	1.539	1.778	3.079	1	0	3
7	67.79	1.377	1.589	2.754	1	1	2



3.3.2. XRD spectrum of 2%Ni doped % ZnO nanostructure and with their unit cell parameters

3.4.1.XRD spectrum of 3% Ni doped ZnO nanostructureand with their unit cell parameters

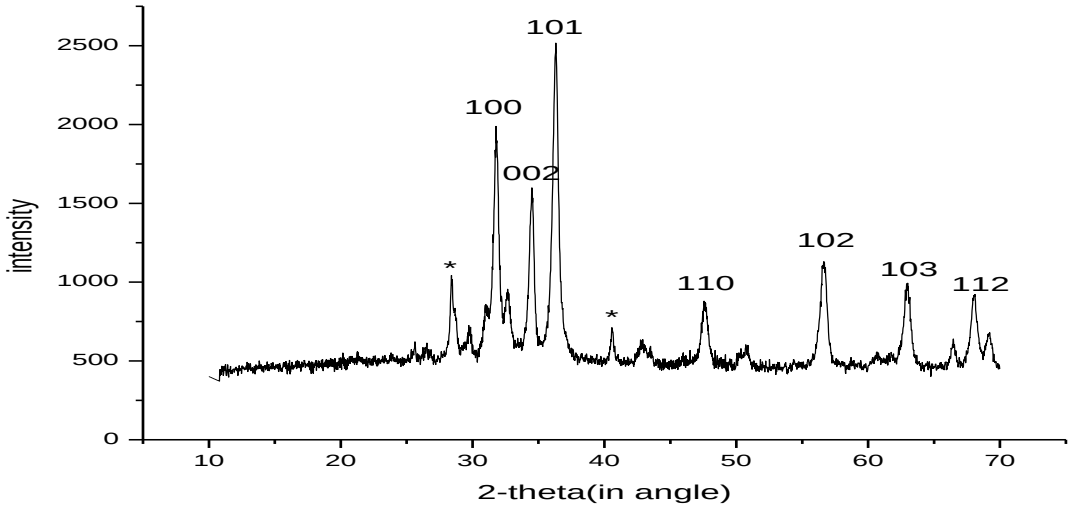
Peaks#	2Θ(°)	a (Å)	c(Å)	d(Å)	h	k	l
1	31.84	3.15	5.46	2.8097	0	0	1
2	34.51	3.249	5.63	2.6035	0	0	2
3	36.34	2.999	5.195	2.4685	1	0	1
4	47.66	2.432	4.211	1.9104	1	1	0
5	56.66	1.875	3.248	1.6258	1	0	2
6	62.96	1.703	2.903	1.4766	1	0	3
7	68.09	1.587	2.699	1.3776	1	1	2



3.4.2. XRD spectrum of 3 %Ni doped % ZnO nanostructure and with their unit cell parameters

3.5.1.XRD spectrum of 4% Ni doped ZnO nanostructureand with their unit cell parameters

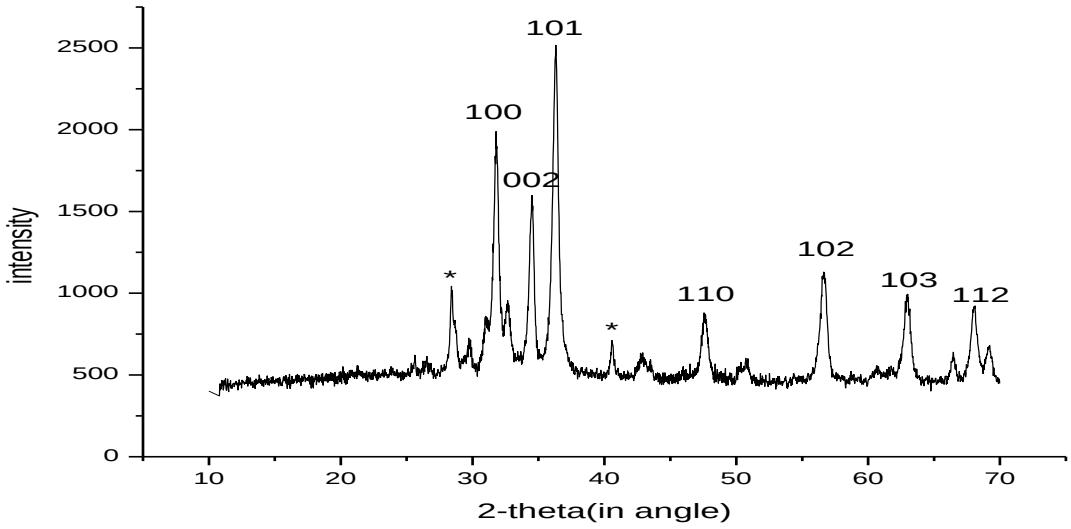
Peaks#	2Θ (°)	a(Å)	c(Å)	d(Å)	h	k	l
1	31.86	3.248	5.62	2.8097	0	0	1
2	34.54	2.996	5.189	2.6035	0	0	2
3	36.36	2.852	4.94	2.4685	1	0	1
4	47.68	2.199	3.193	1.9104	1	1	0
5	56.70	1.873	3.245	1.6258	1	0	2
6	62.96	1.703	2.949	1.4766	1	0	3
7	68.05	1.589	2.752	1.3805	1	1	2



3.5.2. XRD spectrum of 04%Ni doped % ZnO nanostructure and with their unit cell parameters

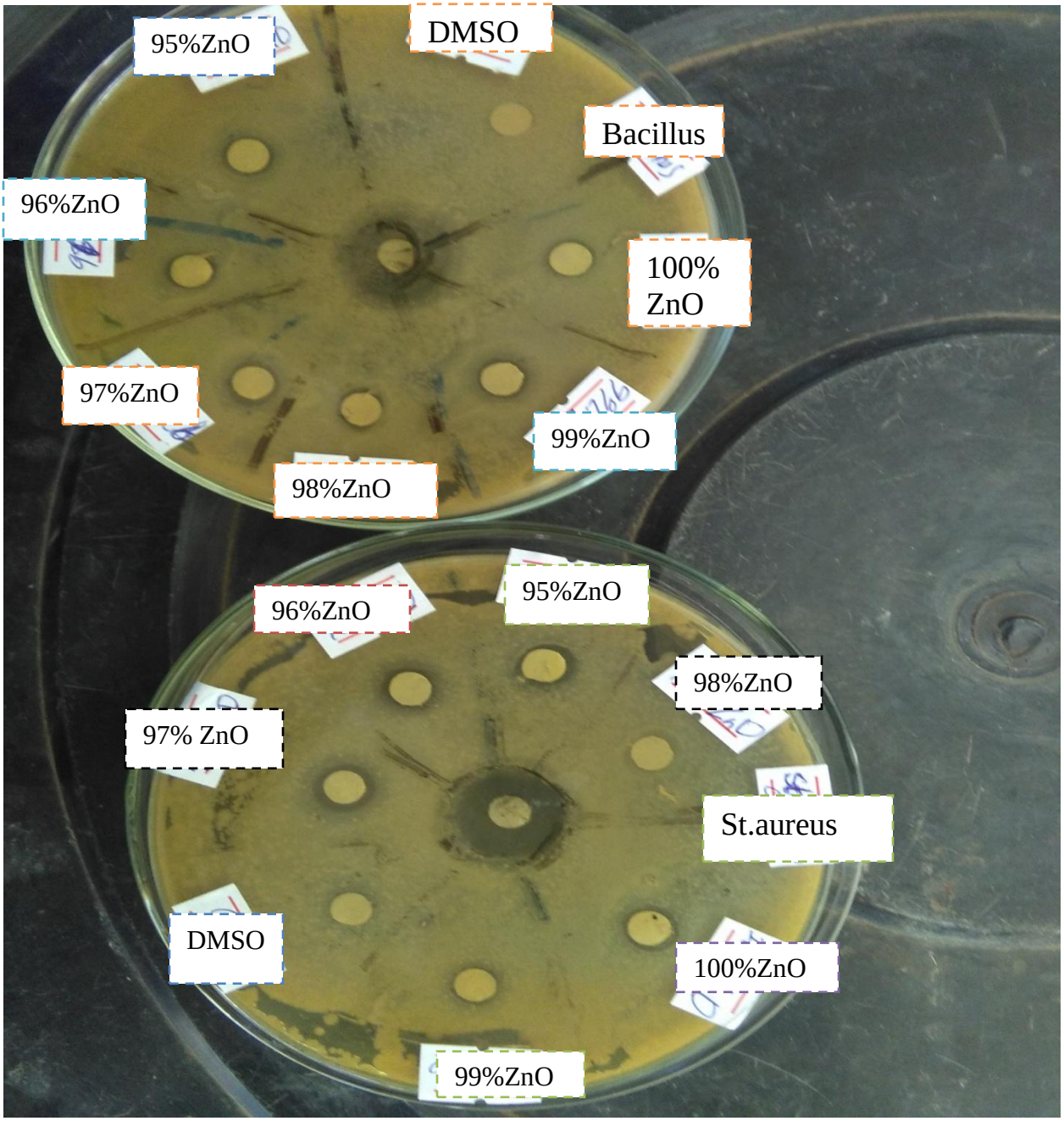
3.6.1.XRD spectrum of 95 % ZnO nanostructureand with their unit cell parameters

Peaks#	2Θ(°)	a(Å)	c(Å)	d(Å)	h	K	l
1	31.82	3.248	5.627	2.8089	0	0	1
2	34.53	2.999	5.1952	2.5969	0	0	2
3	36.33	2.854	4.943	2.4732	1	0	1
4	47.61	2.206	3.821	1.9010	1	1	0
5	56.36	1.875	3.248	1.6255	1	0	2
6	63.00	1.722	2.983	1.4764	1	0	3
7	68.08	1.589	2.752	1.3745	1	1	2

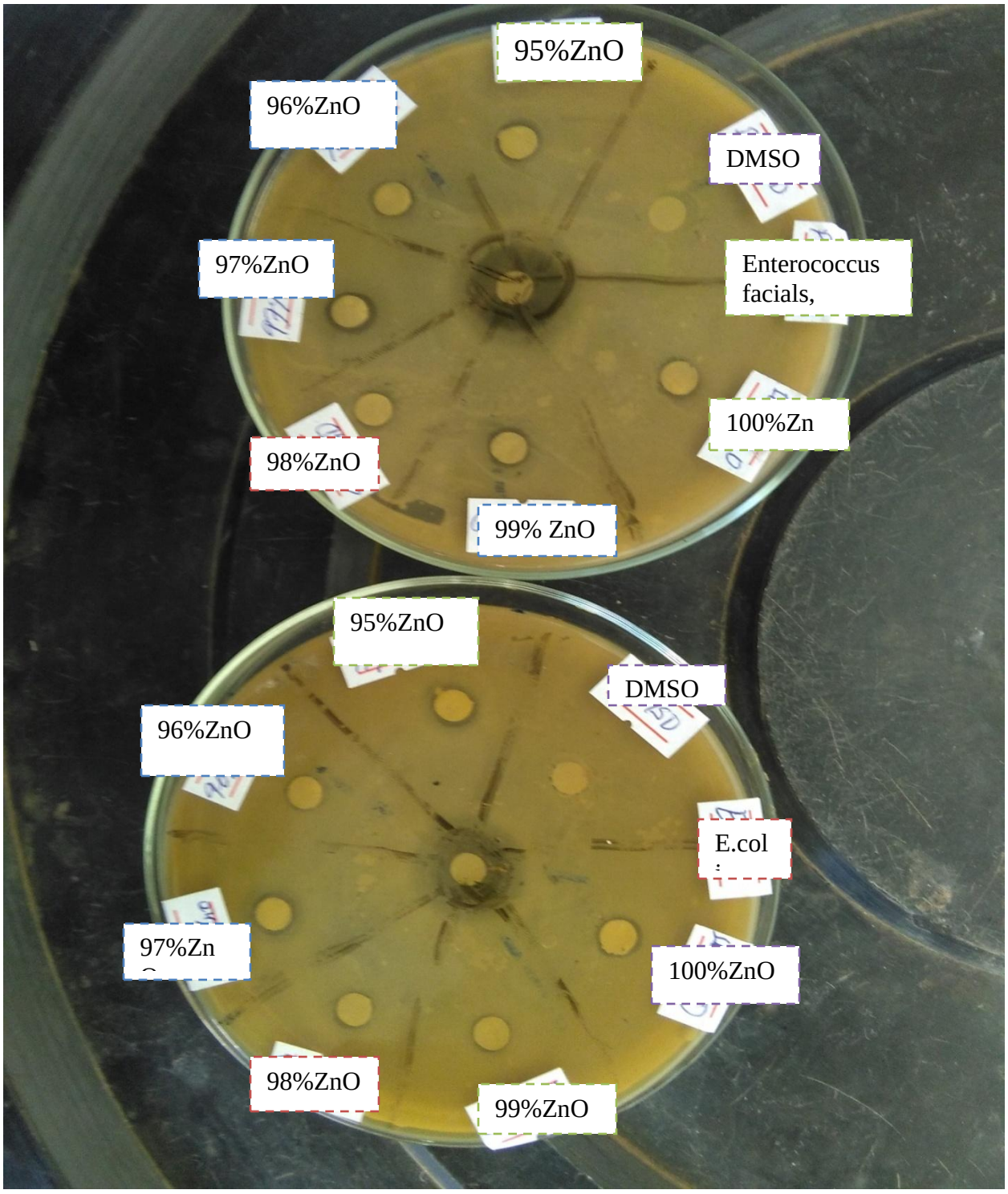


3.6.2. XRD spectrum of 5 %Ni doped % ZnO nanostructure and with their unit cell parameters

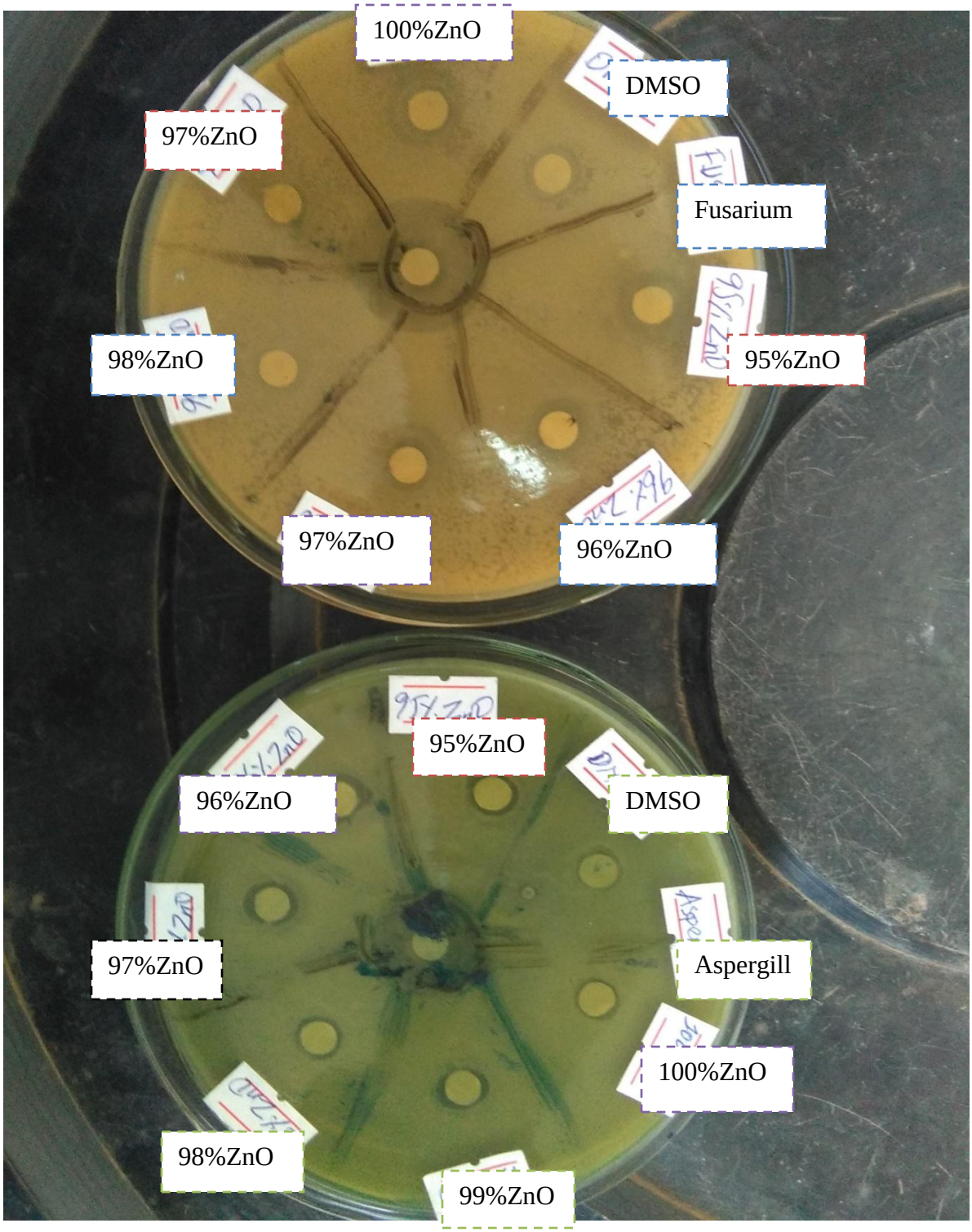
Appendix 4:



4.1Anti-bacterial activity of gram positive bacteria (*Bacillus and*St.aureus*)* for Ni doped and undoped ZnO Nps

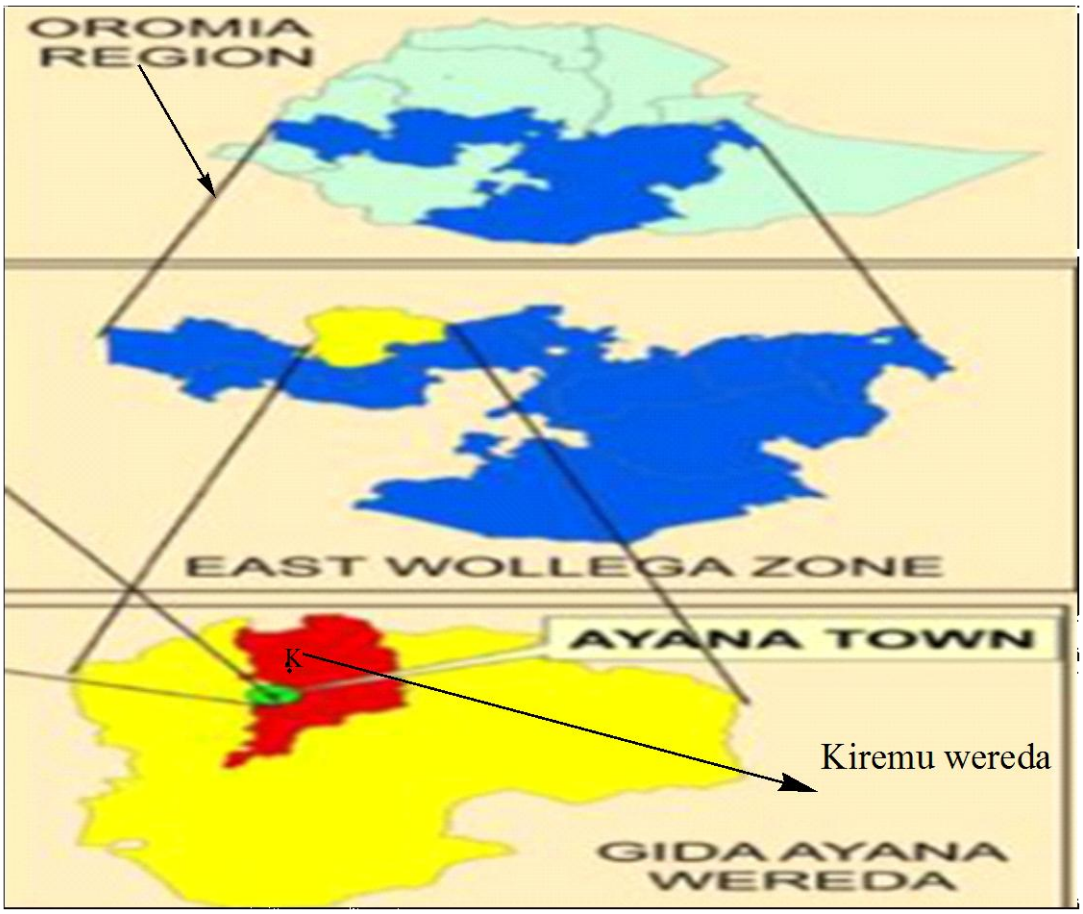


4.2 antibacterial activity of gram Negative bacteria (Enterococcus facials*and E.coli)* for Ni doped and undoped ZnO Nps



4.3. Anti-fungal activity (*Aspergillum and Fusarium) for* Ni doped and undoped ZnO Nps

Appendix5:



Adopted from www.com.