WATER TREATMENT IN INDUSTRIES BY USING METAL DOPED SEMICONDUCTOR NANOPARTICLES

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Abstract—*This paper portrays the preparation of Magnesium doped (3%,4%) zinc oxide NanoParticles by using SOL-GEL method. The structural characterization of the sample was investigated by X-ray diffraction, FE-SEM and FT-IR,UV. The XRD results shows the decrease in crystallite size of zinc oxide doping with Magnesium which is 23nm. The FT-IR showed the formation of functional groups like O-H stretching, O-H bending, CO adsorption on the surface of oxide, Zn–O bond and characteristic stretching mode of Mg-O. FE-SEM studies of MgZnO synthesized nanoparticles are homogeneous, uniformly distributed over the surface and good connectivity between the particles containing the mixer of spheroid-like and rod-like particles in which spheroid-like particles are dominant with grain size around 38-70 nm. UV-VIS studies are showing that the absorption of Mg doped ZnO is higher than the pure ZnO. Waste water from textile, paper and some other industrial processes are usually highly colored, toxic, carcinogenic or mutagenic. These coloured compounds are not only aesthetically displeasing but also inhibiting sunlight penetration into the stream and affecting aquatic ecosystem. In this paper we are going to discuss about the metal doped semi-conductors in industries to eradicate this problem to the better extent with the available chemicals that have robust chemical properties in an efficient way and less duration.*

***Index Terms*— Nanoparticles, SOL-GEL, FE-SEM, UV-VIS.**

1. Introduction:

In the present scenario, enhancing properties of the materials were observed for different applications when nanoparticles were doped. Low cost semiconductor photocatalysts with high photocatalytic efficiency offer great potential for environmental purification and converting photon energy into chemical energy. As a wide band gap(3.37eVat300K)semiconductor with large exciton binding energy(60meV),ZnO is a promising versatile material, which has been intensively studied in the fields of blue–violet light emitting diodes (LEDs), ultraviolet detector, solar cells, field-effect transistors (FETs), sensors, photo catalysts. Though ZnO is photo- catalytically active, its band gap is not wide enough to utilize the high-energy solar radiation. The band gap of ZnO can be controlled via divalent substitution on the cation site. Substituting Mg on Zn site widens the band gap of ZnO, and it is possible to obtain wide band gap Mg-doped ZnO alloys with different ratios of Mg doping, which could be in favor of photocatalytic activities of ZnO under ultraviolet(UV)light irradiation.

1.1 Zinc Oxide Nanoparticles (ZnO NPs):

Zinc oxide is non-toxic material and is listed as 'generally recognized as safe' (GRAS) by the United States Food and Drug Administration. Due to the antimicrobial properties ZnO has been used as linings and coating in food containers. ZnO nanoparticles, owing to their small size and larger specific surface area exhibit enhanced antimicrobial activities. Several mechanisms of antimicrobial activity have been proposed by several authors [4]

1.2 Crystal structure of Zinc Oxide:

Zinc Oxide occupies a special place among wide band gap semiconductors (GaN, ZnS), which have been actively studied because of an increased need for solid state light sources and detectors in the blue and UV spectral ranges [4, 5]. On the basis of GaN and alloys thereof, light emitting and laser diodes in the visible spectral range (460 nm) were developed. However, ZnO is considered to be more favorable for creating UV light emitting diodes and laser diodes, since the binding energy of excitons in it is considerably higher (60 meV) than in GaN (25 meV) [5]. Zinc oxide possesses a high radiation, chemical, and thermal resistance, it is widely used in creation of various instruments in particular, to form transparent contacts of solar cells. Due to its unique optical, acoustic, and electric properties, Zinc Oxide finds use in gas sensors, varistors, and generators of surface acoustic waves [4].ZnO single crystals are also used as substrates for obtaining gallium nitride thin films, since both crystals (ZnO and GaN) belong to the wurtzite structural type and the incommensurability parameter of their lattices along the c axis is 1.8% [1]. Recently, powders, films, and ceramics of zinc oxide have been finding use in the scintillation technique [7]. First and foremost, zinc oxide is known as an efficient phosphor [4, 7]. However, during many decades of research, there no consensus has been achieved on the emission mechanism of the crystal. Moreover, there are a number of models of luminescence that contradict each other (see below). The objective of this work was to examine and systematize basic experimental data on the optical and luminescence properties of ZnO and models that describe them. We also considered possibilities of controlling the characteristics of ZnO and prospects for applications of zinc oxide.

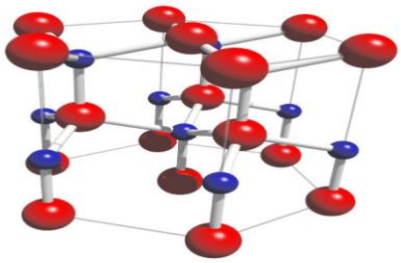


Figure 1.1.Crystal structure of Zinc Oxide (ZnO) (wurtzite structure)

1.3 Necessity of Doping To Zinc Oxide Nanoparticles:

Nanostructures ZnO have many potential applications in photocatalysis, solar cell, gas sensors, fuel cells, photovoltaics, antibacterial action and so on. Due to its inexpensiveness, nontoxic and environmentally safe, it has attracted more attention over last few years. From an environmental point of view, organic dyes and their effluents have become some of the main sources of water pollution. These organic dyes escape from traditional waste water treatment plants and remain in the water because of their high stability against light, temperature, chemicals and microbial attack[15]. Therefore, it costs a lot of time and money to remove such a stable pollutant from water in water treatment process. The photo catalytic technique is a cheap and effective technique to solve this problem.

Significant advances have recently been made in the area of semiconductor nanostructures for photocatalytic applications. In a photocatalytic system, a photo-induced molecular transformation or reaction takes place on the surface of the catalyst. The basic mechanism of a photocatalytic reaction is as follows: when the photocatalyst is illuminated with light of energy greater than its band gap, electron-hole pairs are generated. These pairs will then diffuse to the surface of the photo catalyst where they participate in chemical reactions with electron donors and acceptors. Amongst the large number of different nanostructures as photo catalyst materials, ZnO nanostructures are the most extensively studied as a photo catalyst material due to several advantages. A high redox potential, a direct band gap of 3.37 eV at room temperature, a high exaction binding energy of 60 meV, superior physical and chemical stability, inexpensiveness, easy synthesis and environmentally friendly material were identified as the main reasons for the wide acceptability of ZnO materials compared to other semiconductor photocatalysts[6]. Therefore, ZnO can be used as a photo catalyst material to remove organic dyes from wastewater with a high efficiency. In order to utilize solar radiation more effectively, In fact, the room temperature photoluminescence (PL) of ZnO nanostructures typically exhibits a near band edge emission in the UV region and a broad emission band in the visible region of the electromagnetic spectrum. The visible emission is due to defects such as oxygen vacancies and impurities. Therefore, to increase photocatalyst efficiency under visible irradiation, the band gap of ZnO nanostructures should be decreased, while, the defects density in ZnO nanostructure should be increased. In fact, choosing a cation ionic radius bigger than Zn2+ as a doping material result in the creation of significant lattice defects because of the charge compensation and the ionic radius mismatch between the cat ion and Zn2+, which could affect photo-catalytic properties of ZnO due to the increase in the visible region emission.

1.4 Mg Doped Zinc Oxide Nanoparticles:

One of the major challenges in optimizing the optical properties of ZnO is the incorporation of doping ions into the ZnO lattice. Doping with selective elements offers effective method to enhance and control the structural, electrical and optical properties of ZnO nanoparticles. ZnO doped with proper elements, such as Al, Mn, Cd, Mg, Ni and Cr, has been studied [14]. By doping with a wider band-gap material , the band gap of ZnO particles can be tuned for manufacturing light-emitting devices operating in a wider wavelength region. The doping of ZnO with a metal (Al, Cu, Fe, Ni, Co) can change its properties. ZnO is an ideal material for UV emission; but the intensity of UV emission is weak. However, doping with Group II elements (Cd, Mg) may modulate the value of the band gap and increase the intensity of UV emission . Mg-doped ZnO nanostructures are very interesting owing to their unique optical and electrical properties and more work is in progress to control the physical properties of doped ZnO particles systematically [12]. Therefore, a suitable method for preparing doped ZnO with less operating cost, less synthesis time, narrow size range and better properties is a challenge for scientists. Hence, in this work, we present a simple and low-cost chemical method for synthesizing monodisperse Mg-doped ZnO nanoparticles with semispherical shape, without using a surfactant. Also, we investigate the effect of magnesium doping and annealing on the structural and optical properties of ZnO nanoparticles. Doping of selective elements alter its electronic structure and then influence the structural, optical and catalytic properties. Recently, Mg-doped ZnO nanocrystals have been reported that it possessed an enhanced photo-catalytic activity.

2. Mechanism Of Photocatalysis:

When photocatalyst absorbs Ultraviolet (UV) radiation from sunlight or illuminated light source (fluorescent lamps), it will produce pairs of electrons and holes. The electron of the valence band of metal oxide becomes excited when illuminated by light. The excess energy of this excited electron promoted the electron to the conduction band of ZnMgO therefore creating the negative-electron (e-) and positive-hole (h+) pair as shown in the figure 2.1.1. This stage is referred as the semiconductor's 'photo-excitation' state. The energy difference between the valence band and the conduction band is known as the 'Band Gap'. Wavelength of the light necessary for photo-excitation is: 1240 (Planck's constant, h) / 3.2 ev (band gap energy) = 388 nm

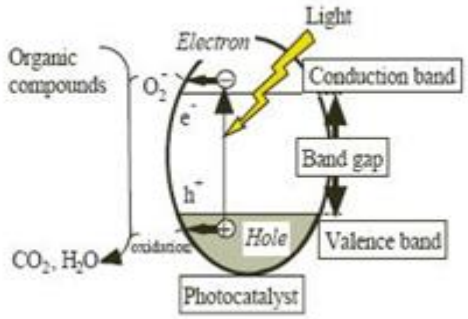


Figure 2.1 Mechanism of Photo catalysis.

The positive-hole of titanium dioxide breaks apart the water molecule to form hydrogen gas and hydroxyl radical. The negative-electron reacts with oxygen molecule to form super oxide anion. This cycle continues when light is available.

2.1 Decontamination Of Water With Photocatalysis:

Waste water from textile, paper and some other industrial processes are usually highly colored, toxic, carcinogenic or mutagenic. These colored compounds are not only aesthetically displeasing but also inhibiting sunlight penetration into the stream and affecting aquatic ecosystem. Dyes usually have complex aromatic molecular structures which make them more stable and difficult to biodegrade. Some dyes are reported to cause allergy, dermatitis, skin irritation, cancer and mutations in humans. Among many organic pollutants, methylene blue (MB) is one of pollutant color for environment undesirable which effects on aesthetic of environment. Thus, environmental contamination by these toxic chemicals has emerged as a serious global problem. On the contrary, bleached dye after degradation of solution is relatively less toxic and almost harmless. Secondly, dye containing colored water is almost no practical use, but if this colored solution is bleached to give colorless water, then it may be used for some useful purposes like washing, cooling, irrigation and cleaning. Recently, photo catalytic reactions induced by illumination of semiconductors in suspension have been shown to be one of the most promising processes for the wastewater treatment.

3. Methylene Blue Dye:

Methylene blue is a compound consisting of dark green crystals or crystalline powder, having a bronze-like luster. Solutions in water or alcohol have a deep blue color. Methylene blue is used as a bacteriologic stain and as an indicator. Methylene blue (or MB) is a basic aniline dye with the molecular formula C16H18N3SC. At room temperature, it appears as a solid, odorless, dark green powder that yields a blue solution when dissolved in water. It has many uses in a number of different fields. For instance, chemists use it to detect oxidizing agents and biologists use it to stain tissue samples and detect nucleic acids. In medicine, it is used as a treatment for various illnesses and disorders, including methemoglobinemia, schizophrenia, kidney stones, and herpes infections. In aquaculture, it is used to prevent freshwater fish eggs from being infected by bacteria and fungi. In addition to its needed effects, some unwanted effects may be caused by methylene blue. In the event that any of these side effects do occur, they may require medical attention.

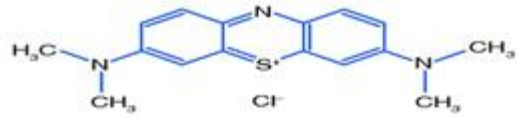


Figure 3.1.1 Physical appearance of Methylene blue

3.1. Methylene Blue Dye Degradation Application:

Photocatalytic activities of the as-synthesized powder were evaluated by decolourization of Methylene Blue dye in aqueous solution. The experiments were carried out in the presence of visible light irradiation with catalyst in dark and in the presence of Mg Doped ZnO nanoparticles as photo catalyst and distilled water. The photocatalytic reactor consists of a Pyrex glass beaker with a magnetic stirrer in it and a metal halide lamp in a wooden setup. Reaction was set up by adding0.5 gm of the as-synthesized powder into 95 ml of Methylene blue (1PPM) solution and 5mL of distilled water in the Pyrex glass beaker of 200 ml volume and the suspension was magnetically stirred in dark for 30 min as shown in the fig 3.1.3 To obtain adsorption/ desorption equilibrium before irradiating the light in the beaker.

Figure 3.1.2 Mythelyne blue with photo catalyst

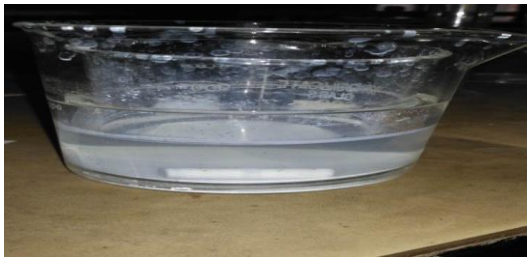


Figure 3.1.3 Dye degradation in the presence of light

During the reaction, the solution was maintained at room temperature and the distance of the lamp from the solution was 30 cm. Then the light source was activated, 10 ml of the sample was withdrawn at 10 min time interval over irradiation time for 40 min. The suspension was centrifuged at 1000 rpm for 10 min and filtered to remove the catalyst particles before measuring absorbance. The absorbance of the clear solution was measured at a λ max of 667 nm for quantitative analysis.

The incandescent bulb was used as visible light source with a definite power of 400 W, 220 V and 60 Hz frequency.

3.2. Methylene Blue Degradation Studies:

The photocatalytic activity of as synthesized nanomaterial was evaluated by the degradation of Methylene Blue (MB) dye in aqueous solution. The decolourisation of the Methylene blue dye as shown in the fig 3.2.1 was examined visible light irradiation with catalyst is nano sized.

Figure 3.2.1. Degradation of Methylene Blue Dye after photocatalysis

4. Degradation Studies Of Mg With ZnO NP's:

The corresponding plots of percentage degradation as a function of time under visible light irradiation with catalyst shown in Fig 4.1. The experimental results show that when the dye solution is exposed to visible light irradiation for 40 min the percentage of degradation was found to be 100% .

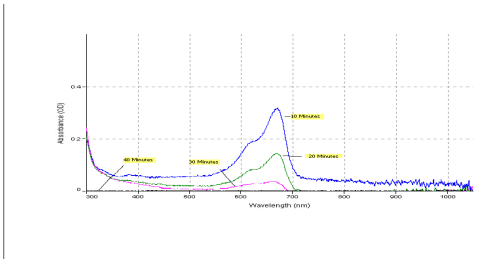
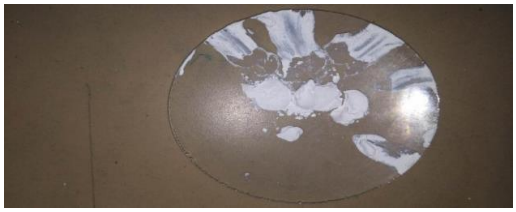


Figure 4.1. UV-VIS Result for Photo degradation of Mg Doped ZnO nano particles

4.1. Synthesis Of Mg Doped ZnO nano particles By Sol-Gel Method:

Nano powders were prepared by Sol-Gel method. 1M of $ZnsO_4.7H_2O$ and 0.01M ,0.02M $Mg(NO_3)_2.6H_2O$ are separately dissolved completely in distilled water. These two solutions are mixed in a beaker placed under continuous stirring on the magnetic stirrer. Citric acid was dropped in to the solution slowly until its pH reaches to 1.5. Then 20ml of ethylene glycol is added to this solution and continuously stirred until white precipitate was formed. White precipitate was placed in an oven at 100^0c until solvent was evaporated. Finally it is heat treated at 600^0c in an oxidizing furnace and after this it is thoroughly grinded. Final the nanoparticles of Mg doped ZnO are formed.



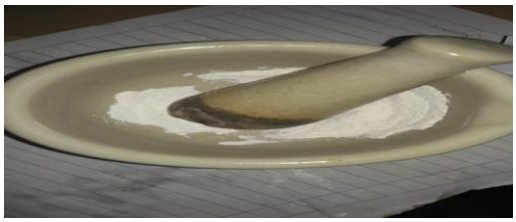


Figure:4.1.1 shows the material preparation of ZnMgO nanoparticles by sol-gel method.

5. RESULTS AND DISCUSSION

5.1. X-Ray Diffraction Analysis:

X-ray diffraction is an analytical technique generally used for phase identification of a crystalline material and can provide information on a unit cell dimensions as well. X-ray diffraction is now a common technique for studying crystal structures and atomic spacing. Although single crystal X-ray crystallographic investigation is the most precise source of information regarding the structure of a complex, the difficulty of obtaining crystalline complexes renders this method unsuitable for such a study. However, a variety of other spectroscopic techniques could be used with good effect for characterizing the metal complexes as X-ray powder diffraction. So, X-ray powder diffraction (XRD) measurements of three complexes are performed. The diffract gram obtained complexes has been given in figures and the observed diffraction data, with the help of the data obtained from the powder XRD, the particle size calculations are performed using Scherrer equation.

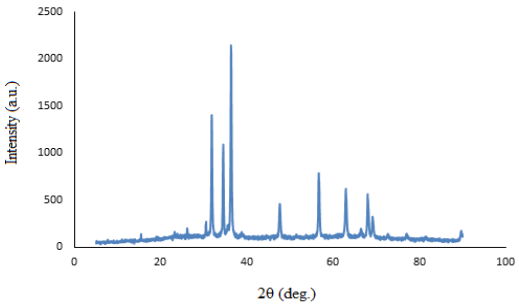


Figure 5.1.1 XRD Pattern for 3% Mg Doped Zinc Oxide NP

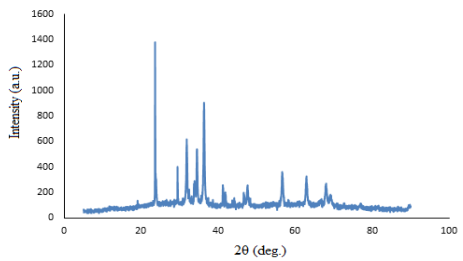


Figure 5.1.2 XRD Pattern for 4% Mg Doped Zinc Oxide NP

The diffraction pattern is recorded and radius 2θ value of 36.295,23.578 are observed, which corresponds to Bragg reflections of hexagonal wurtzite structure.

Table 3 Crystal Size

Method	Crystal Size
3% Mg doped ZnO	23 nm
4% Mg doped ZnO	35 nm

Calculated by using Scherrer formula:

$$L = \frac{k\lambda}{\beta \cos\theta}$$

5.2. Field Emission-Scanning Electron Microscopy (FE-SEM):

The Field emission scanning electron microscope (FE-SEM) is the most widely used type of electron microscopic technique. It examines microscopic structure of the sample by scanning the surface/fractured surface of materials with higher resolution and much greater depth of field. The most important feature of an FE-SEM is the three-dimensional appearance of its images because of its large depth of field. FE-SEM enables us to obtain chemical information from the specimen by using various techniques, including the X-ray energy dispersive spectrometer (EDS). The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface, topography, composition and electrical conductivity from the image the average particle size is in the range of 37-70nm

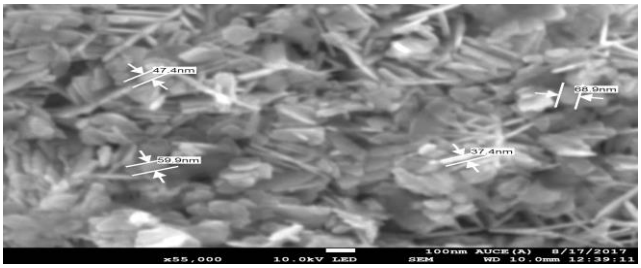


Figure 5.2.1 FE-SEM Morphology for 3% Mg Doped Zinc Oxide NP

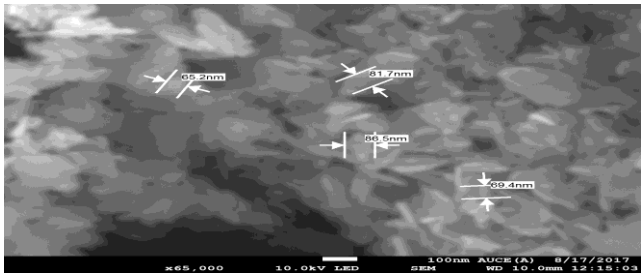


Figure 5.2.2 FE-SEM Morphology for 4% Mg Doped Zinc Oxide NP

5.3. Energy Dispersive X-Ray Spectroscopy (EDS):

Energy-dispersive X-ray spectroscopy (EDS, EDX, or XEDS) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum. To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron is an electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the X-rays is characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they are emitted, this allows the elemental composition of the specimen to be measured.

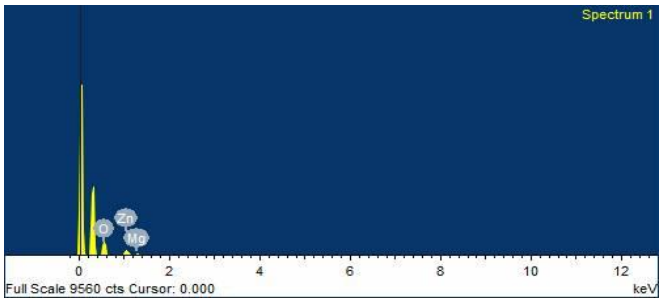


Figure 5.3.1.EDX of Mg Doped ZnO NP.

Table 4. Elemental Composition of 3% Mg Doped Zinc Oxide NP.

Element	Weight%	Atomic%
O K	60	82.95
Mg K	7.1	6.08
Zn K	32.9	10.97
Total	100	100.0

Table 5. Elemental Composition of4% Mg Doped Zinc Oxide NP.

Element	Weight%	Atomic%
O K	55.99	77
Mg K	10.01	9.01
Zn K	35	13.99
Total	100	100.0

The energy dispersive spectrum Figure12 revealed that the clear identification of elemental composition profiles of the synthesized nanoparticles, which suggest that the presence of magnesium as the ingredient element. Zinc nanoparticles typically shows optical absorption peak at 1 KeV to the surface Plasmon resonance. However other electrical signals along with Mg doped Zinc Oxide nanoparticles are also recorded, which are not observed the many other biosynthesis of nanoparticles. The elemental composition of Zinc and Oxygen, magnesium as shown in Table 4 and 5.

5.4. Uv-Vis Spectroscopy:

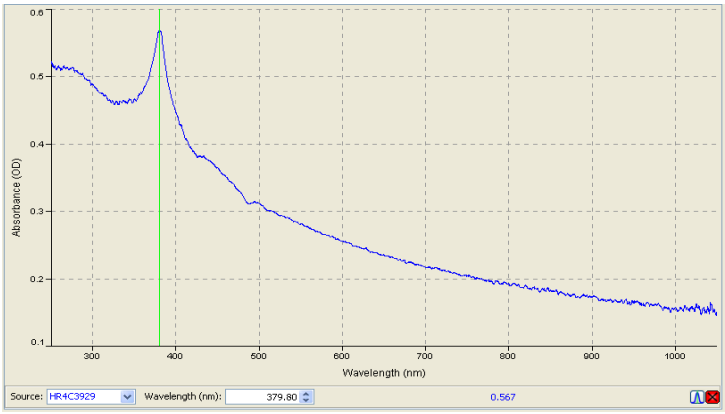


Figure 5.4.1 Absorption spectra of 3% Mg Doped ZnO nanoparticles

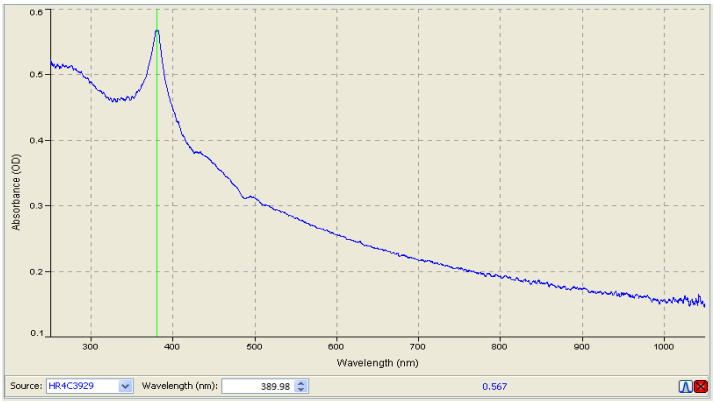


Figure 5.4.2Absorption spectra of 4% Mg Doped ZnO

The UV–Visible optical absorption spectrum of the Mg doped ZnO have been carried out at room temperature using UV Visible spectrometer from 250 to 500 nm .The absorption spectra show that the absorption of the samples was occurred at 379.80,389.98 nm .

The absorption is attributed to the doping of Mg to ZnO.

The band gaps (Eg) of Mg-ZnO were calculated by using the formula

$$E = \frac{hc}{\lambda}$$

Where h is plank's constant, c is the velocity of light and λ is the wavelength.

Table 6 . The bandgap of Mg-ZnOwere compared toband gap of ZnO is 3.36 Ev

SAMPLE	BAND GAP
3% Mg Doped ZnO	3.26 eV
4% Mg Doped ZnO	5.5. Ev

5.5. Fourier Transform Infrared Spectroscopy:

Two milligram of Mg Doped ZnO nanoparticales are prepared by mixing with 200 mg of spectroscopic grade KBr.

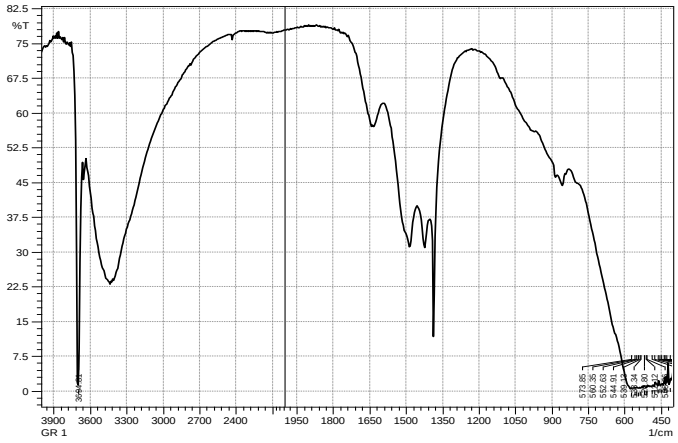


Figure 5.5.1 FTIR for 3% Mg Doped Zinc Oxide NP's

Table 7. Peak Assignments of 3% Mg Doped Zinc Oxide NP's:

S. No	Wave number (cm^{-1})	Peak Assignments
1	1400	O-C Stretching
2	1495	C=C Stretching
3	1645	$C \equiv C$ Stretching
4	3300-3600	O-H Bending
5	3700	C-H Bending

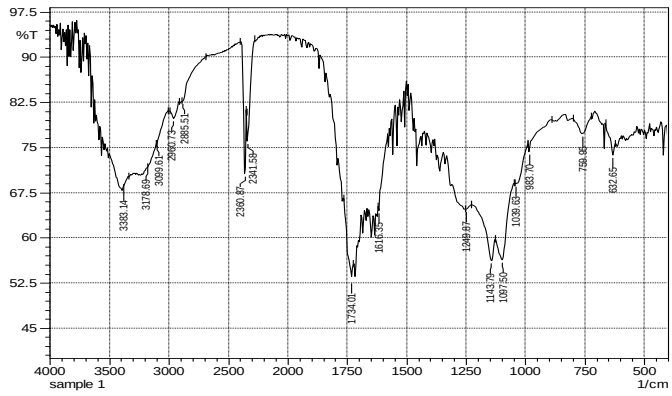


Figure 5.5.2 FTIR for 4% Mg Doped Zinc Oxide NP's

Table 8. Peak Assignments of4% Mg Doped Zinc Oxide NP's:

S. No	Wave number (cm^{-1})	Peak Assignments
1	1097.50	O - C Stretching
2	1143.79	C – C Stretching
3	1734	C = O Stretching
4	2341,2360.87	$C \equiv N$ Stretching
5	3300-3600	O- H Bending

Spectra are recorded using a Nicolet 520P spectrometer with detector at 450-3900 cm^{-1} resolution and 20 scans per sample.

FTIR Spectra of a Mg Doped Zinc oxide nanoparticles prepared from the SOL-GEL method was carried out to identify the possible bio-molecules responsible for capping and efficient stabilization of the metal nanoparticles.

The presence of bio-molecules is in the range observed from 450 cm^{-1} to 3700 cm^{-1}

6. CONCLUSION

In this paper Magnesium doped Zinc Oxide nanoparticles are synthesized. The prepared samples are characterized by using Spectroscopic, Microscopic, Conductivity studies such as XRD, FT-IR, FE-SEM, UV-Visible spectroscopy. From the XRD results, the crystalline size of magnesium doped ZnO is 23, 35 nm. FE-SEM studies of Mg Doped ZnO synthesized nanoparticles are homogeneous, uniformly distributed over the surface and good connectivity between the particles with AVERAGE size around 38-70 nm. From the FT-IR Result the presence of bio molecules is observed. UV-VIS studies of Mg doped ZnO are showing that the absorption peak at 379 nm, 389 nm which gives the energy band gap of 3.26 eV, 3.18 eV. To study their application we studied the Photo-Catalysis behavior of Mg Doped Zinc Oxide. These studies will help for an efficient degradation of dye pollutants being released from the industries and help improve the penetration of sun light into the waters.

7. Future Scope:

- At present work, only Mg dopant at one particular concentration i.e., 3% ,4% is used to dope ZnO.
- The scope of the work in future can be widened to include different types of dopants such as Al, Ni etc.,
- Properties should be compared for different concentrations of dopants to find out better properties.

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