



Microwave Assisted Synthesis of Zinc Oxide Nanoparticle and its Antimicrobial Activity

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Abstract : Zinc oxide nanoparticles were synthesized via chemical reduction method using Zinc sulfate heptahydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) as a precursor and urea was used as reducing agent, Sodium hydroxide was used as a stabilizing agent and deionized water were used as medium. ZnO nanoparticles were characterized by using UV-Vis, FESEM, and EDAX. Zinc nanoparticles were confirmed by UV-Vis studies which shows sharp peak at 355nm. The SEM images showed that ZnO nanoparticles were rod in shape. The EDAX pattern shows the 74.76% of Zinc present in synthesized sample. The advantage of nanotechnology has led the development of materials with new properties for used as an antimicrobial agent. Thus, ZnO nanoparticle shows very good antimicrobial properties against *Staphylococcus aureus* and *Candida albicans*.

Keywords : Zinc nanoparticles, Chemical reduction, UV-Vis, SEM, EDAX, Antimicrobial, and Urea.

Introduction:

Nanomaterials are synthesized from the novel ecofriendly and sustainable biological and engineering process¹. Nanomaterials contain particles in the size of 1nm-100nm and are by definition, a material having a size below 100nm in at least one dimension. In 21st century, the nanotechnology has emerged as an interdisciplinary field with the biosynthesis of metal nanoparticles. Nanoscience and nanotechnology are growing research areas. The activities have increased exponentially in these fields during the past decade, finding applications in everyday life such as sunscreens, antibacterial agents, bio-medical products and surface protection just to name a few examples²⁻⁵.

In recent years, application of nanoparticles within size range has received significant attention due to their novel properties. Among several of nanoparticles, ZnO nanoparticle have received more attention. ZnO nanoparticle has a wide band gap (3.37eV), semiconductor having large excitation binding energy of 60 meV at room temperature. It has been considerably for its catalytic, electrical, optoelectronic, and photochemical properties⁶⁻⁹. ZnO nanostructures have a great advantage to apply to a reaction process due to their large surface area and high catalytic activity¹⁰.

Many methods have been described in the literature for the production of ZnO nanostructure such as laser ablation¹¹, hydrothermal methods¹², electrochemical depositions¹³, sol-gel method¹⁴, chemical vapour deposition¹⁵, thermal decomposition¹⁶, and combustion method^{17,18}. Now-a-days, ZnO nanoparticles were prepared by ultrasound¹⁹, microwave-assisted combustion method²⁰, two-step mechanochemical-thermal synthesis²¹, anodization²², co-precipitation²³, and electrophoretic deposition²⁴ methods. In this paper, we report an effective precipitation method for the preparation of ZnO nanoparticles by using microwave irradiation. The

microwave irradiation method considered herein fast, mild, energy-efficient, and environment-friendly.

Materials & Methods:

Zinc sulfate heptahydrate, Sodium hydroxide and urea were used in experiment. All chemicals were of analytical grade and used without further purification obtained from Eswar scientific. And deionized water is used for the preparation of solution.

Synthesis of zinc oxide nanoparticles:

1mm of aqueous solution of Zinc sulfate heptahydrate was prepared. From that Solution different concentrations such as 50µl, 100µl, 150µl etc was taken in a glass vial, a drop of urea and NaOH was added to the solution. The solution was kept under microwave oven for 5 mins. After 5 mins the colour changes from transparent to white colour solution which indicates the formation of zinc oxide nanoparticle, the white colour solution was centrifuged in cooling centrifuge machine for 5 mins at 10,000 rpm. The settled ZnO nanoparticles were collected and dried in a hot air oven at 60°C. The dried sample was used for further characterization.

Characterization of zinc oxide nanoparticles:

The synthesized ZnO nanoparticles were characterized using UV-Vis, SEM, and EDAX. The reaction of zinc sulfate heptahydrate solution with urea and sodium hydroxide as the reducing and stabilizing agent was optically measured using shimadzu UV-Visible spectrophotometer. Shape and size of the synthesized zinc oxide nanoparticles were studied by using scanning electron microscope. The elemental composition of the synthesized nanoparticle was determined using energy dispersive X-ray spectroscopy.

Antimicrobial activity:

The antimicrobial activity was performed by disc diffusion method using Gram – **positive** bacteria: *Staphylococcus aureus* and Gram – **negative** bacteria: *Escherichia coli*, *Candida albicans*. Nutrient Agar and Potato Dextrose Agar media was used, sterilized and solidified. Then two bacterial strains *Staphylococcus aureus* and *Candida albicans* were swabbed on the plates. Sterile disc were dipped in zinc oxide nanoparticle solution (10mg/10ml) and placed in the nutrient and potato dextrose media and kept for incubation at 37°C for 24 hours and for fungal which was incubated at 25°C for 48 hours. Zone of incubation for control and ZnO were measured and the mean values of zone diameter were presented. The antimicrobial potential of test compounds was determined on the basis of mean diameter of zone of inhibition around the disc in millimeters. The zones of inhibition of the tested microorganisms were measured.

Results and discussion:

Visual observation:

The formation of zinc Nanoparticles was initially confirmed by visual observation. The change in colour of the reaction mixture confirms the formation of Zinc oxide nanoparticles. According to this study the addition of sodium hydroxide to the aqueous solution of zinc sulfate heptahydrate and urea, there will be a colour change from colourless to white with the deposition of white precipitate²⁶ it is due to the formation of Zinc oxide nanoparticles as shown in fig(1).



Figure.1: (a) zinc sulfate heptahydrate and urea mixture solution, (b) After addition of sodium hydroxide, formation of ZnO nanoparticle.

UV-Visible spectroscopy:

UV-Vis absorption spectroscopy is widely being used technique to analyses the optical properties of nano sized particles²⁷. The UV-Vis absorption for ZnO nanoparticles ranges between 300-550nm²⁸. Fig (2) shows the UV-Vis spectra of synthesized ZnO nanoparticles shows sharp peak at 355 nm due to SPR band which confirms the formation of ZnO nanoparticles in the solution.

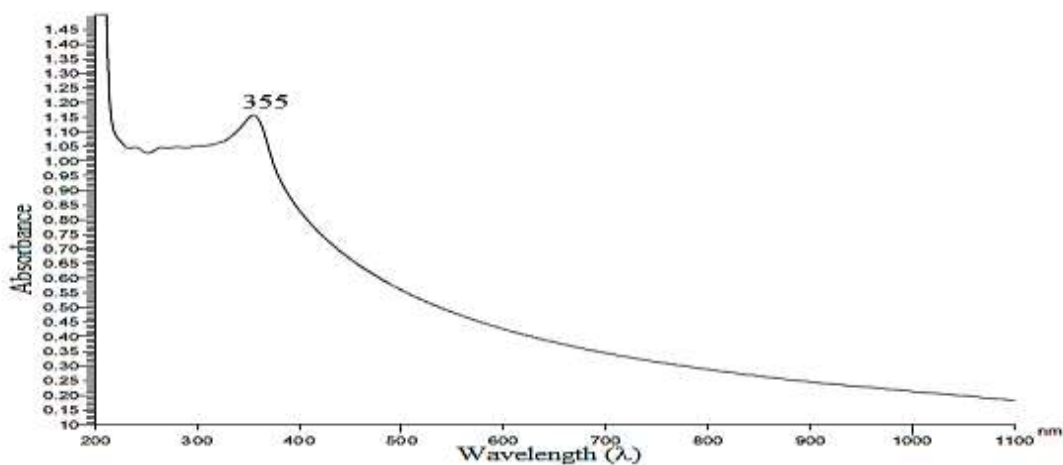


Figure.2 UV-Vis spectra for ZnO nanoparticle.

Scanning Electron Microscope:

Size and shape of the materials were determined by Scanning Electron Microscope. Figure 3 shows the SEM image of synthesized ZnO nanoparticle. In this study SEM image revealed clear shape of nanoparticle which we synthesis. The image shows that the particle are in nanorange and the most of them seems to be in rod shape.

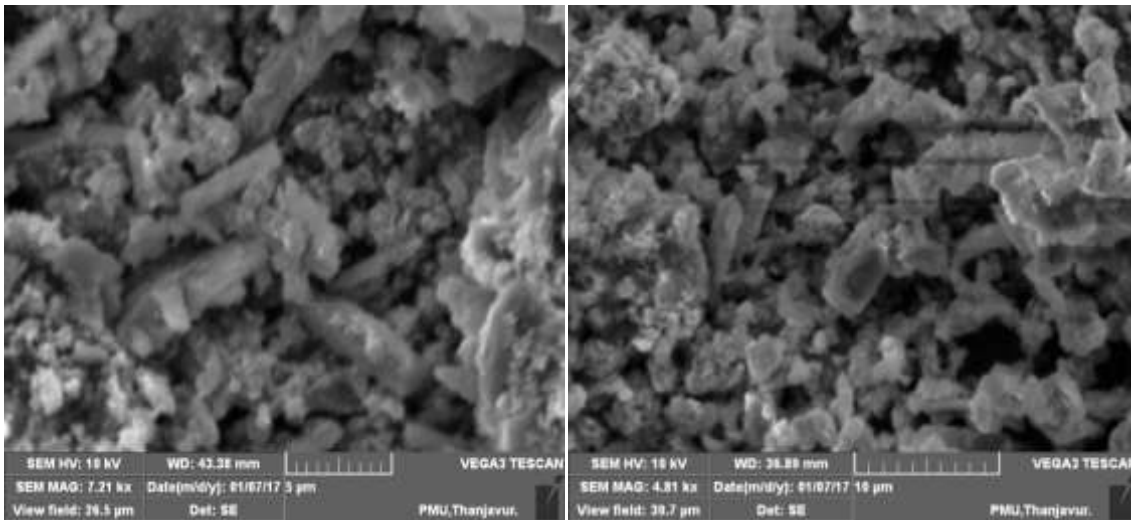


Figure.3. SEM images for ZnO Nanoparticle.

Energy Dispersion X-ray Analysis:

Energy Dispersion X-ray analysis is a technique used to determine the elemental composition of the particle. Using this technique the elemental composition of the synthesized ZnO nanoparticle were found out. Fig. 4 shows the image of zinc present in the synthesized ZnO nanoparticle. Table 1 confirmed that zinc present in larger amount than oxygen. The composition Zinc and Oxygen are 74.76% and 25.24% respectively from this data it was clearly understood that zinc shows high purity in the synthesized ZnO nanoparticles.

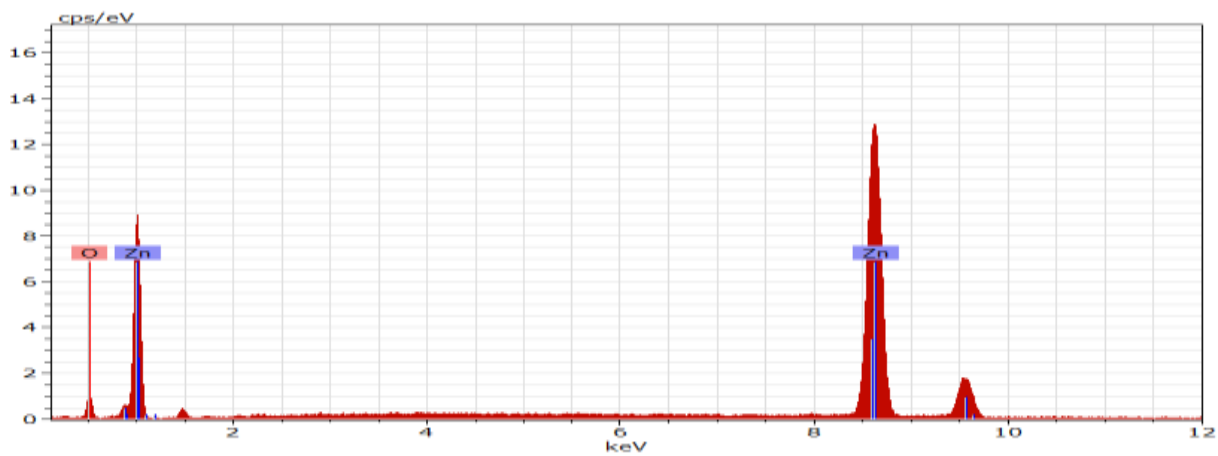


Figure.4: EDAX image of Synthesized ZnO nanoparticle

Spectrum: sample 16320

Table: 1: Elemental composition of ZnO nanoparticle

El AN	Series	unn.c[wt.%]	norm.c[wt.%]	Atom.c [at. %]	Error	(1 Sigma) [wt.%]
Zn 30	K-series	102.17	92.37	74.76	-	2.58
O 8	K-series	8.44	7.63	25.24	-	2.03
	Total	110.62	100	100	-	

Antimicrobial studies:**Antimicrobial assay:**

Antibiogram was done by disc diffusion method using nanoparticle²⁹. Petri plates were prepared by pouring 30 ml of NA /PDA medium for bacteria/fungi. The test organism was inoculated on solidified agar plate with the help of micropipette and spread and allowed to dry for 10 mins. The surfaces of media were inoculated with bacteria/fungi from a broth culture. A sterile cotton swab is dipped into a standardized bacterial/fungi test suspension and used to evenly inoculate the entire surface of the Nutrient agar/PDA plate. Briefly, inoculums containing *Staphylococcus aureus* and *Escherichia coli* specie of bacteria were spread on Nutrient agar plates for bacteria and *Candida albicans* was spread on potato dextrose agar for fungus strains. Using sterile forceps, 6 mm diameter of filter papers containing the crude extracts of 50µl were laid down on the inoculated agar plate. The plates were incubated at 37°C for 24 h for the bacteria and at room temperature (30±1) for 24-48 hr. for yeasts strains. Each sample was tested in triplicate.

Measurement of zone of inhibition

The antimicrobial potential of synthesized compounds was determined on the basis of mean diameter of zone of inhibition around the disc in millimeters. The zones of inhibition of the tested microorganisms by the samples were measured using a millimeter scale.

Microorganisms	ZnNPs			Standard (30µl)
	(50µl)	(100µl)	(150µl)	
Bacteria				
<i>Staphylococcus aureus</i> (mm)	0.40±0.02	2.30±0.16	5.70±0.39	9.70±0.67
Fungus				
<i>Candida albicans</i> (mm)	0.50±0.03	2.10±0.14	5.50±0.38	9.50±0.66

Table: 2 Antimicrobial activities for synthesized ZnO nanoparticles.

Values were expressed as Mean ± SD for triplicates

Bacterial standard	Chloramphenicol
Fungal standard	Fluconazole
Control	Demonized water

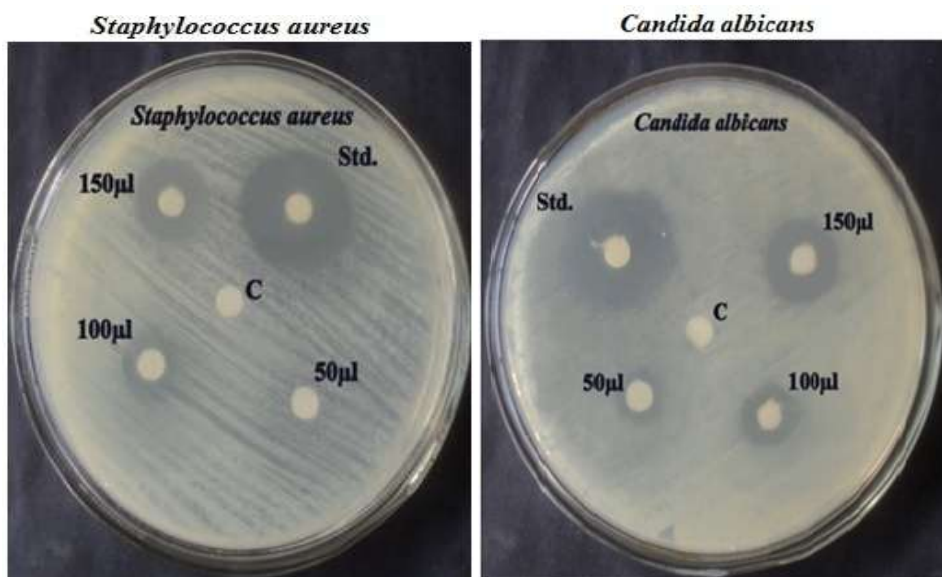


Figure:5:Antimicrobial activity for synthesized ZnO nanoparticles.

Conclusion:

The ZnO nanoparticle was successfully prepared via Chemical reduction method using zinc sulfate heptahydrate as a zinc source, urea as a reducing agent and sodium hydroxide as a stabilizing agent. A high production yield was obtained by using sodium hydroxide and urea. The method used is a simple and cheap method which does not require a solvent or calcinations after drying. The Synthesized ZnO nanoparticles were characterized using UV-Visible spectroscopy. The optical absorption peak intensity is found to be at 355nm. The SEM and TEM analysis image shows the clear image of rod like structure with high purity. The presence of zinc nanoparticle in major composition was confirmed by EDAX analysis which shows Zn-content of 74.76%. The crystalline structure of Zinc nanoparticles were confirmed by XRD shows that the synthesized ZnO nanoparticles were in hexagonal structure. Finally, it acts a very good antimicrobial activity against *Staphylococcus aureus* and *Candida albicans*. Further scope this study extends to analyze for antioxidant purpose, anti-cancer, medicinal, pharmaceutical, and toxic free cosmetics.

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