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Synthesis, Characterization & Antibacterial Evaluation of Different Nanostructures of Zinc Oxide

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Abstract: In the present research work, synthesis of Zinc oxide nanostructure, their antibacterial evaluation and characterization was carried out. In this study, zinc nanoparticles were rapidly synthesized from Zinc Acetate [Zn $(O_2CCH_3)_2$] solution with different precursors. The synthesized ZnO nanostructures were characterized by different spectroscopic and analytical techniques such as Zeta-Sizer, XRD, TEM. The SEM. X-ray diffraction, Zeta-Sizer, SEM analysis studies confirmed the formation of well dispersed zinc oxide nanostructures with average particle size to be in the range of 35 to 60 nm. The antibacterial evaluation of the ZnO nanoparticles using different precursors (NaOH, KOH, LiOH) was done by Agar well diffusion method against *E. coli, P. aeruginosa, B. subtilis* and *S. aureus* bacteria and the zone of inhibition were measured.The antibacterial evaluation of the synthesized nanostructures shows that it has been the driving force for the development of new surfaces with antibacterial agents that might affect differently several microorganisms and hence helps in prevention of infection.

Keywords: *Zinc Oxide, Antimicrobial, Different Shaped Nanoparticles, XRD, SEM, TEM.*

I. INTRODUCTION

Zinc oxide (ZnO), a wide band gap (3.4 eV) *II-VI* compound semiconductor, has a stable quartzite structure with lattice spacing a = 0.325 nm and c = 0.521 nm. It has attracted intensive research effort for its unique properties and versatile applications in transparent electronics, ultraviolet (UV) light emitters, piezoelectric devices, chemical sensors and spin electronics [1,2]. Invisible thin film transistors (TFTs) using ZnO as an active channel have achieved much higher field effect mobility than amorphous silicon TFTs. These transistors can be widely used for display applications. ZnO has been proposed to be a more promising UV emitting phosphor than GaN because of its larger exciton binding energy (60 meV) [3,4,5]. This leads to a reduced UV lasing threshold and yields higher UV emitting efficiency at room temperature.Surface acoustic wave filters using ZnO films have already been used for video and radio frequency circuits. Piezoelectric ZnO thin film has been fabricated into ultrasonic transducer arrays operating at 100 MHz.15 Bulk and thin films of ZnO have demonstrated high sensitivity for toxic gases [6,7].

Based on these remarkable physical properties and the motivation of device miniaturization, large effort has been focused on the synthesis, characterization and device applications of ZnO nanomaterials. An assortment of ZnO nanostructures, such as nanowires, nanotubes, nanorings, and nano-tetrapods have been successfully grown via a variety of methods including chemical vapor deposition, thermal evaporation, and electrodeposition, *etc* [8,9]. These nanostructures have been subjected to electrical transport, UV emission, gas sensing, and ferromagnetic doping studies, and considerable progresses have been achieved[10,11].

II. MATERIALS AND METHODS

A. Chemicals Used

Zinc Acetate $Zn(CH_3COO)_2.2H_2O$, different precursors Like:- sodium hydroxide (NaOH), Potassium hydroxide (KOH), Lithum hydroxide monohydrate (LiOH), ethanol (C2H5OH) and methanol (CH3OH) ofanalytic grade were used without further purification. The bacterial strains gram +ve *Staphylococcus aureus (S. aureus*, 502) and fungal strain *Aspergillus nige (A. niger)* (456) were procured from National Collection of Industrial Microorganisms (NCIM); The bacterial strains of gram −ve*Escherichia coli* (*E. coli*, DH5_) and fungal strain *Botrytis cinerea (B. cinerea*, 359) were procured from Microbial Type Culture Collection, (MTCC) IMTECH, Chandigarh and three *Aspergillus* species (*sl*1, *sl*2 and *sl*3_ isolated from soil samples were procured from culture collection, Department of Bio and Nano Technology, GJUS and T Hisar.

B. Synthesis of ZnO nanostructures (Sample A)

The ZnO nanostructures were synthesized by a simple hydrothermal route. The starting materials used for the synthesis were Zinc Acetate $Zn(CH_3COO)_2.2H_2O$ and sodium hydroxide (NaOH). All the chemicals were of analytic grade and were used without further purification. Experimental details are as follows 10.975 g $Zn(CH_3COO)_2.2H_2O$ and 6 g of NaOH were dissolved, each in 100 ml of distilled water, to form 0.5 M and 1.5 M solutions, respectively. The NaOH solution was slowly added to the $Zn(CH_3COO)_2.2H_2O$ solution under manual stirring. The mixture was ultrasonicatored for about 30 minute to ensure proper mixing of the two solutions. Further, the above solution was loaded into a teflon flasks. Finally the flask was sealed and maintained at 230˚ C for 6

h (hydrothermal treatment time, *t*H). It was then allowed to cool down to room temperature naturally. The precipitate was filtered off, washed with absolute ethanol and distilled water several times, and then dried in air at 80˚ C for 1 h. The white colored product so obtained was then used for various characterizations.

Synthesis of ZnO nanostructures (Sample B,C,D,E& F) the above procedure applied on all samples :- B (by using NaOH at 180° C for 10 hr.); sample C (using KOH at 200° C for 4 hr.); sample D (using KOH at 300° C for 3.5 hr.); sample E (using LiOH at 180° C for 8.5 hr.); sample F involve LiOH at 250° C for 3 hr.

C. Characterization of ZNO nanomaterials

The as synthesized ZnO nanostructures were characterized by different techniques.UV-vis absorption spectra were recorded using digital spectrophotometer (Shimadzu UV-1800 series). Photoluminescence (PL) spectra were obtained using (Perkin Elmer LS-55) Fluorescence Spectrometer (Range: 200–900 nm) having Xe Lamp as a source. Surface morphology and elemental analysis studies were performed using SEM: ZEISS EVO MA10 and EDS: OXFORD INCA 250 ISIS. High resolution transmission microscopy studies were performed using HRTEM: Tecnai G2 F30 S-Twin (FEI; Super Twin lens with Cs = 1_2 mm) operated at an accelerating voltage of 300 kV, having a point resolution of 0.2 nm and lattice resolution of 0.14 nm. Powder X-ray diffraction (XRD) investigations were performed using Advance powder X-ray Diffractometer (Bruker Axes D-8). The crystallite sizes were estimated using Scherrer's method. FTIR spectra were recorded using Perkin Elmer 2000 Optica model (spectral range of 10000 cm−1 to 370 cm−1) over the range 4000–400 cm−1.

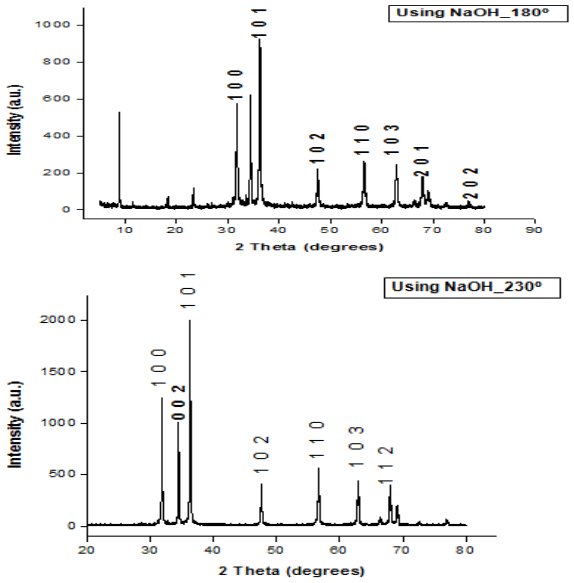
III. RESULTS AND DISCUSSION

A. The XRD spectra of ZnO Sample A (Using NaOH at 230˚C)

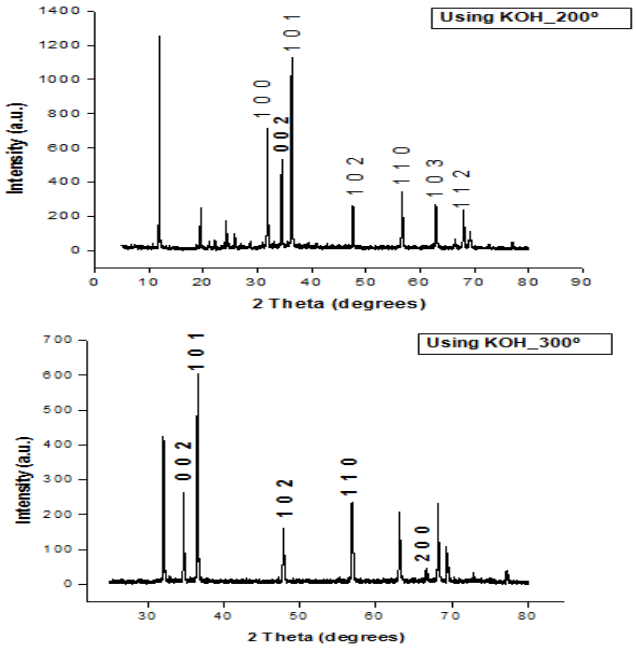
The diffraction peaks in the spectra were in good agreement with those of the standard data (JCPDS 83-0944). Almost all the diffraction peaks with similar relative intensity as reported in the standard data were observed in the experimental XRD pattern and were significantly sharp, hence confirming the purity and high crystallinity of the synthesized product. The sharp diffraction peaks of the powder imply their good crystalinity. All diffraction peaks in the XRD pattern of as prepared ZnO nanoparticles can be assigned to the hexagonal structure phase (JCPDS card No. 01-079-0207) The sharp diffraction peak at around 31.68˚(which matches 31.69˚in JCPDS data) corresponds to the (1 0 0) plane, other peaks which corresponds to the hexagonal phase were at 34.40˚ [0 0 2], 36.22 [1 0 1], 47.50˚ [1 0 2], 56.50˚ [1 1 0] and 62.76˚ [1 0 3]. All the peaks correspond to the JCPDS data. XRD pattern of samples indicate that phase pure hexagonal structure of ZnO has been successfully synthesized. Well crystallized diffraction peaks and no characteristic peaks of impurities of sample were observed, suggesting that the prepared ZnO samples is well crystallized and with high purity.

B. XRD spectra of ZnO Sample B (Using NaOH at 180˚C) Sample B

The sharp diffraction peak at around 31.61˚(which matches 31.69˚in JCPDS data) corresponds to the (1 0 0) plane, other peaks which corresponds to the hexagonal phase were at 34.31˚ [0 0 2], 36.28˚ [1 0 1], 47.60˚ [1 0 2], 56.71˚ [1 1 0], 62.86˚ [1 0 3] and 68.30˚[2 0 1]. All the peaks correspond to the JCPDS data.



C. XRD spectra of ZnO (Using KOH_200) Sample C



All diffraction peaks in the XRD pattern of as synthesized ZnO nanostructures can be assigned to the hexagonal structure phase ZnO (JCPDS card No. JCPDS 01-076-

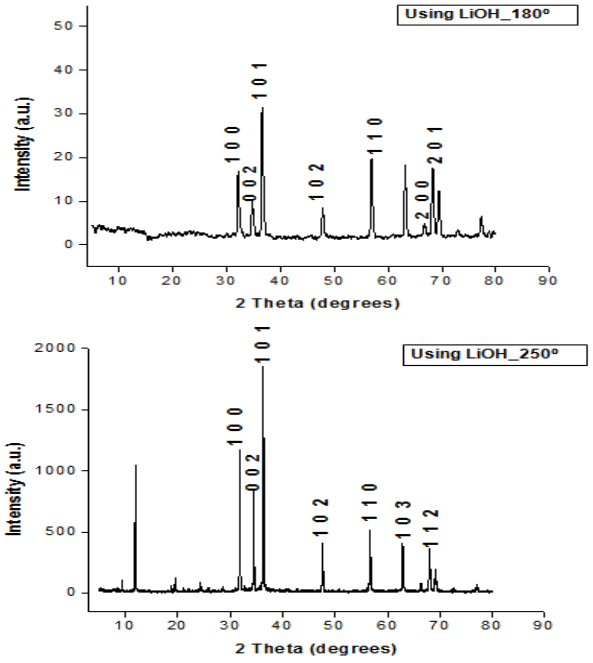
0704). The sharp diffraction peaks at around 31.78˚(which matches 31.73˚in JCPDS data) corresponds to the (1 0 0) plane, other peaks which observed which corresponds to the hexagonal phase, 34.30˚ [0 0 2] (which matches 34.37˚in JCPDS data), 36.32˚ [1 0 1],47..65˚ [1 0 2], 56.45˚ [1 1 0], 62.75˚ [1 0 3] and 67.78˚ [1 1 2].

D. The XRD spectra of ZnO (Using KOH_300) Sample D

The sharp diffraction peaks at around 34.55˚(which matches 34.37˚in JCPDS data) corresponds to the (0 0 2) plane, other peaks observed which corresponds to the hexagonal phase, 36.31˚ [1 0 1] (which matches 36.21˚in JCPDS data), 47.77˚ [1 0 2], 56.76˚ [1 1 0], 66.64˚ [2 0 0] and 69.45˚ [2 0 1].

E. The XRD spectra of ZnO (Using LiOH at 180 ^{0} C) Sample E

The sharp diffraction peak at 31.96˚(which matches 31.73˚in JCPDS data) corresponds to the (1 0 0) plane, other peaks which correspond to the hexagonal phase, 34.62˚ [0 0 2], 36.53˚ which is almost equal to 36.20˚ [0 0 2], similarly the sharp peak was observed at 47.61˚ [1 0 2], 56.74˚ [1 1 0], 66.61˚ [2 0 0], 68.29˚ [2 0 1] showing their respective planes.



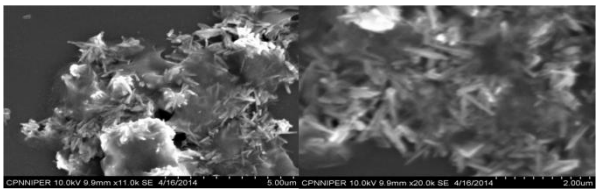
F. The XRD spectra of ZnO (Using LiOH at 180 ^{0} C) Sample F

The sharp diffraction peak at 31.79˚(which matches 31.73˚in JCPDS data) corresponds to the (1 0 0) plane, other peaks which corresponds to the hexagonal phase, 34.46˚ [0 0 2], 36.41˚[1 0 1], 47.60˚ [1 0 2], 56.60˚ [1 1 0], 62.92˚ [1 0 3] and 67.79˚ [1 1 2] having their respective planes.

IV. THE SEM ANALYSIS

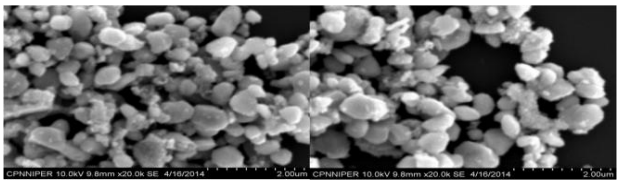
A. The SEM Analysis of Zno Nanostructures

(i) ZnO (Sample A)



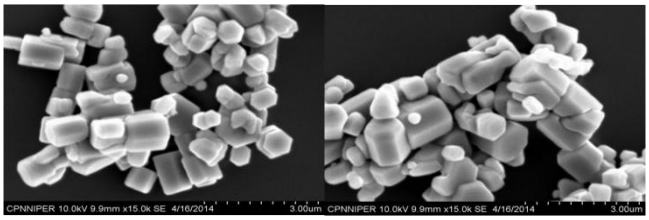
Above figure depicts that the prickly structure (1-4µm) of ZnO were formed, which were the result of agglomeration of the nanostructures of ZnO (5-30 nm). The structure thus formed shows prickly flower like structures.

(ii) ZnO (Sample B)



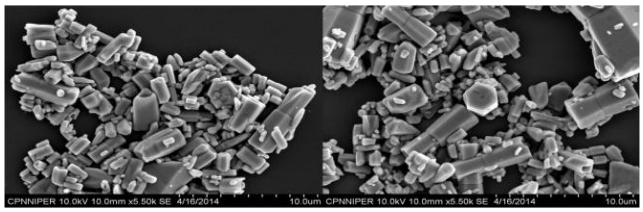
The SEM analysis of the sample B showed that the stone like balls or sweet snacks like structures of the size 200 nm- 0.8 µm were formed due to the agglomeration of the ZnO nanostructures (20-50nm).

(iii) ZnO (Sample D)



The SEM analysis of the sample D showed that the hexagonal structures of the size 300 nm-1.2µm were formed due to the agglomeration of the ZnO nanoparticles (20-50nm).

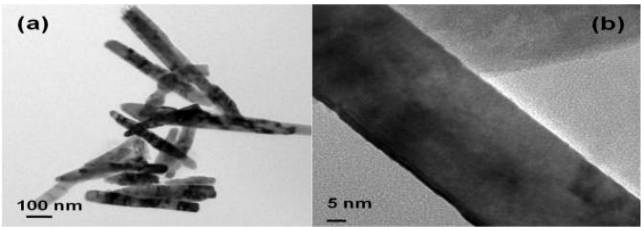
(iv) ZnO (Sample E)



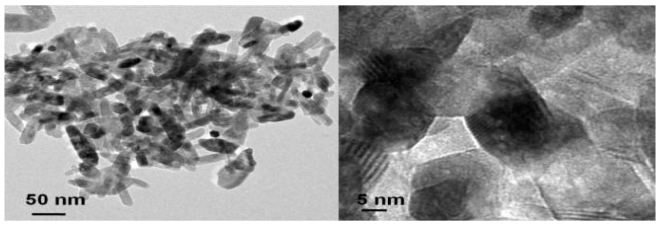
The SEM analysis of the sample E showed that the hexagonal dumb bell like structures of the size 0.5-8µm were formed due to the agglomeration of the ZnO nanostructure (20-50nm).

V. THE HRTEM ANALYSIS

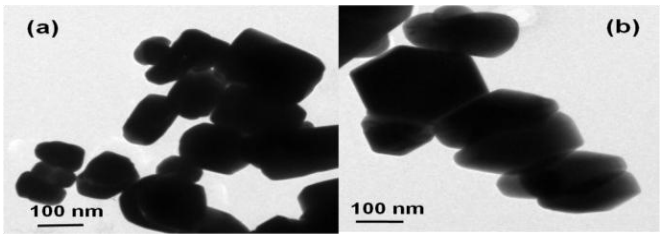
(i) Zinc Oxide (sample A): The HRTEM images of Zinc Oxide (sample A) depicted that the dried turmeric root like structures were observed and their size were measured in the ranges from 100-800 nm long and width was observed to be 40-90 nm.



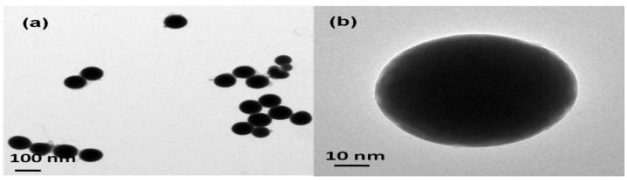
(ii) Zinc Oxide (sample B): Irregular ball like structures were observed in the size range 15-45 nm in HRTEM analysis.



(iii) Zinc Oxide (sample D): Hexagonal structures were observed in the size range 40-250 nm in HRTEM analysis of sample D.



(iv) Zinc Oxide (sample E): From HRTEM analysis it was observed that spherical ball like structures were observed in the size range 35-90 nm.

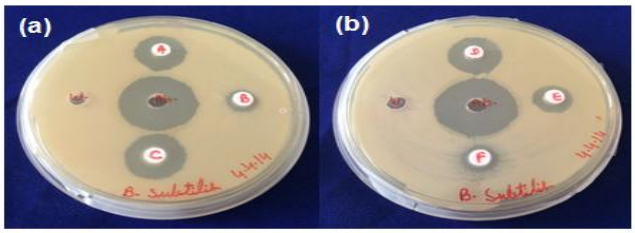


A. Antimicrobial Activity

The evaluation of antibacterial activity of ZnO nanoparticles synthesized using different precursors (at different-different conditions), like NaOH (Samples A and B), KOH (Samples C and D), LiOH (Samples E and F) were performed against *S. aureus* (Gram +ve), *B. subtilis* (Gram +ve), *P. aeruginosa* (Gram -ve), *E. coli* (Gram -ve) strains by Agar Well Diffusion method as described previously. The diameters of the zone of inhibitions were measured using scale and divider.

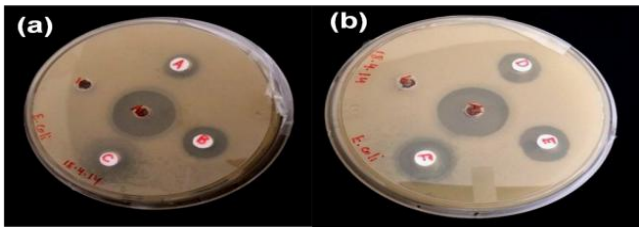
(i) Antibacterial activity against*B. subtilis*

The zones of inhibition of six samples ZnO (A), ZnO (B), ZnO (C), ZnO (D), ZnO (E) and ZnO (F) were measured against *B. subtilis* and were found that the ZOI of six samples were found to be 16 mm,10 mm, 16.5 mm, 16 mm, 12 mm and 10 mm respectively, While zone of inhibition (ZOI) of the antibacterial drug (Ciprofloxacin) was found to be 23 mm were as the negative control i.e. water (w) showed no zone of inhibition.



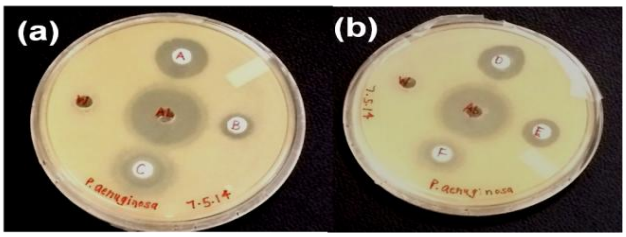
(ii) Antibacterial activity against *E. coli*

In case of *E. coli* also, the zone of inhibition were measured and it was observed that the sample ZnO (A) produced 12 mm inhibition zone, ZnO (B) produced 14 mm inhibition zone and ZOI produced by ZnO (C), (D), (E) and (F) were found to be 12.5 mm, 13.5 mm, 15 mm and 15.5 mm respectively, no ZOI was produced by water (w) i.e. the negative control. While that of antibacterial drug (Ciprofloxacin) was found to be 21.5 mm.



(iii) Antibacterial activity against *P. aeruginosa:*

The zone of inhibition against *P. aeruginosa* were measured and it was found that the ZnO sample (A) produced 16.5 mm zone of inhibition, ZnO (B) produced 15mm inhibition zone and ZnO (C) showed 19.5 mm zone while sample (D), (E) and (F) showed 15 mm, 16 mm, and 19 mm respectively. The zone of inhibition of Ab i.e. antibacterial drug (Ciprofloxacin) was found to be 26 mm. and no ZOI case of distal Water.



(iv) Antibacterial activity against *S. aureus*:

The zone of inhibition against *S. aureus* were measured and it was found that the sample ZnO (A) produced 18.5

mm inhibition zone, ZnO (B) produced 18 mm inhibition zone and ZOI produced by ZnO (C), (D), (E) and (F) were found to be 13 mm, 13 mm, 12 mm and 15 mm respectively, while zone of inhibition produced by (Ab) i.e. antibacterial drug (Ciprofloxacin) was found to be 23.5 mm and no ZOI was produced by (-ev) control i.e. Distell water.

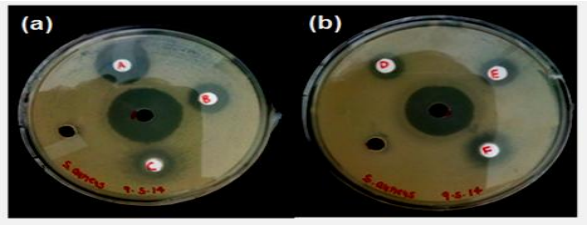


Table 1: Zone of Inhibition of Antibacterial activity of ZnO nanostructures

Bioactive Agent/Samples		**Zone of Inhibition (mm)**			
		E. coli ***(-ve)***	***S. aureus*** ***(+ve)***	***P. aeruginosa*** ***(-ve)***	***B .subtilis*** ***(+ve)***
ZnO Nanoparticles synthesized using different precursors like NaOH, KOH & LiOH at different experimental conditions	***ZnO (A)***	12	18.5	16.5	16
	ZnO (B)	14	18	15	10
	ZnO (C)	12.5	13	19.5	16.5
	ZnO (D)	13.5	13	15	16
	ZnO (E)	15	12	16	12
	ZnO (F)	15.5	15	19	10
	Antibiotic(Ab)	21.5	23.5	22.5	23
	Water (W) negative control	0	0	0	0

CONCLUSION

In this work, Zinc Oxide nanostructure having different structure/shapes were synthesized by hydrothermal method using different precursors like Sodium Hydroxide (NaOH), Potassium Hydroxide (KOH), Lithium Hydroxide (LiOH) at different experimental conditions. Characterization of the as synthesized ZnO nanostructures were carried out using TEM, SEM, XRD, UV-VIS spectroscopy, Zeta-sizer. Also evaluation of ZnO nanostructure showed good antibacterial activity against *E. coli* and *P. aeruginosa* (Gram-ve) as well as *S. aureus* and *B. subtilis* (Gram+ve) bacteria used in the present study. Hence, this research work intends to develop innovative and more appropriate nanosized ZnO nanostructures having antibacterial properties that could be easily used for antibacterial coating in food packaging and in other areas.

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