Synthesis, Characterization and antimicrobial activity of ZnO nanoparticles in the food packaging

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***Abstract*: This deals with synthesis and characterization of ZnO nanoparticles and their antimicrobial activities against pathogenic microorganisms. The synthesis process has been carried out by refluxing zinc acetate precursor and with complexing agent Polyethylene Glycol (PEG). All synthesized ZnO nanoparticles has been characterized by UV visible spectroscopy (UV), X-ray diffraction (XRD), FTIR (Fourier transform infrared spectroscopy). All Zinc oxide nanoparticles (ZnO) have been allowed for antimicrobial activity.**

Keywords: ZnO, Characterization, Antimicrobial activity, Complexing agents

1. INTRODUCTION

Nanotechnology is a research hot spot in modern materials science. This technology is capable of providing miscellaneous novel applications that range from innovative fabric compounds, food processing, and agricultural production to sophisticated medicinal techniques. It is considered as the synthesis, characterization, and exploration of materials in the nanometer region (1–100 nm) [2]. Foodborne diseases are a global public health issue. The CDC has estimated 47.8 million foodborne illnesses, 127,839 hospitalizations and 3,037 deaths for 2011 in the U.S. alone, (CDC, 2011), which would result in medical expenses and productivity losses, and affect global health, trade and the economy. As a result, the demand for new technologies to control foodborne pathogens has increased significantly in recent years. As such, food packaging plays an important role in providing safety and maintaining quality of food. Food packaging with new functions is known as active packaging, developed as a result of consumer demand for safety and more natural products with a longer shelf life, better cost benefits and convenience. The aim of this study was to synthesize and characterize the PEG complexing ZnO nanoparticles by an economical and easy method, and observes the comparative effects of complexing and without complexing ZnO nanoparticles on different biological activities taking ace in living system. Our basic purpose was to examine the role played by surfactants (PEG) in exhibiting any biological activity by ZnO nanoparticles. Antimicrobial agents are of great importance to numerous industries, such as environmental, food, packaging, medical, healthcare, decoration industries, and so forth.

Nano-sized ZnO exhibits varying morphologies and shows significant antimicrobial activity over a wide spectrum of bacterial species explored by a large body of researchers [3]. ZnO is currently being investigated as an antimicrobial agent in both microscale and nanoscale formulations. ZnO exhibits significant antimicrobial activities when particle size is reduced to the nanometer range, then nano-sized ZnO can interact with bacterial surface and/or with the bacterial core where it enters inside the cell, and subsequently exhibits distinct bactericidal mechanisms. The interactions between these unique materials and bacteria are mostly toxic, which have been exploited for antimicrobial applications such as in food industry [2].

A Reflux process can be defined as a process in a closed reaction vessel inducing decomposition or a chemical reaction(s) between precursor(s) in the presence of a solvent at a temperature higher than the boiling temperature of this solvent. The pressure can be autogeneous (in such a case the pressure value depends on the filling of the reaction vessel) or imposed (the pressure value being higher than 1 bar (105 Pa) at the starting point of the experiment through the compression of the reaction medium). Depending on the experimental conditions (pressure and temperature), the reflux system can be heterogeneous or homogeneous and in subcritical or supercritical conditions [4].

The Reflux method was employed to synthesize ZnO nanostructure. This technique is based on thermal decomposition of organometallic compound in organic solvent and has been successfully applied for the synthesis of various types of nanosized metal oxide with large surface area, high crystallinity and high thermal stability

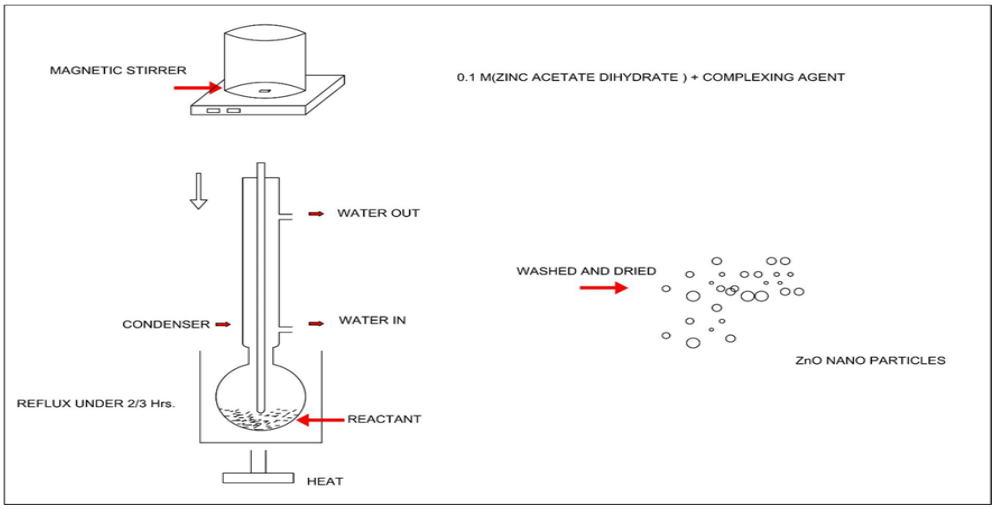

Fig.1 Reflux operation

2. MATERIALS AND METHOD

2.1 Materials

All chemicals used here were of Analytical Reagent (A.R) grade. Distilled water used in the all process. Methanol was used to wash the samples. Complexing agent Polyethylene glycol (PEG) was used in experiment. The microorganisms, Gram- positive (Staphylococcus Aureus) and Gram- negative (Escherichia Coli) were used for antimicrobial activity.

2.2 Synthesis of ZnO nanoparticles

Total 3 batches have been taken at normal temperature, without complexing agent and with complexing agent.

Zinc oxide (ZnO) nanoparticles were prepared by zinc acetate dehydrate (0.1N) and NaOH (0.1N). The solution kept on magnetic Stirring at room temperature for 3.5 hr. NaOH added dropwise and maintained pH of the solution. The samples were centrifuged at 10000 rpm and 10000 rpm for 10 min and washed with methanol for three times further, it was dried at 60ºc for one night and placed in furnace at 300ºc for 2 hr.

Zinc oxide (ZnO) nanoparticles were prepared by refluxing operation. Without complexing agent batch was taken at 85ºc for 2 hr. NaOH added dropwise and maintained pH of the solution. The samples were centrifuged at 10000 rpm and 10000 rpm for 10 min and washed with methanol for three times further, it was dried at 60ºc for one night and placed in furnace at 300ºc for 2 hr.

Before reflux operation with complexing agent, NaOH and PEG (0.6 ml) was stirred for 30 min. With complexing agent batch was taken at 85ºc for 2 hr. NaOH and PEG solution added dropwise and maintained pH of the solution. The samples were centrifuged at 10000 rpm and 10000 rpm for 10 min and washed with methanol for three times further, it was dried at 60ºc for one night and placed in furnace at 300ºc for 2 hr.

ANTIMICROBIAL ACTIVITY

0.1 ml distilled water added in two plates. ZnO nanoparticles added in two plates. E coli 10 mg/ml added in one plate and 6 mg/ml S.aureus in other plate. ZnO nanoparticles add in the plate. This process was carried out at the temperature of 37ºc and for 24 hr. then results analysed by microscopy.

3. RESULT AND DISCUSSION

3.1) Characterization:

Batch 1: At room temperature

UV Visible spectroscopy

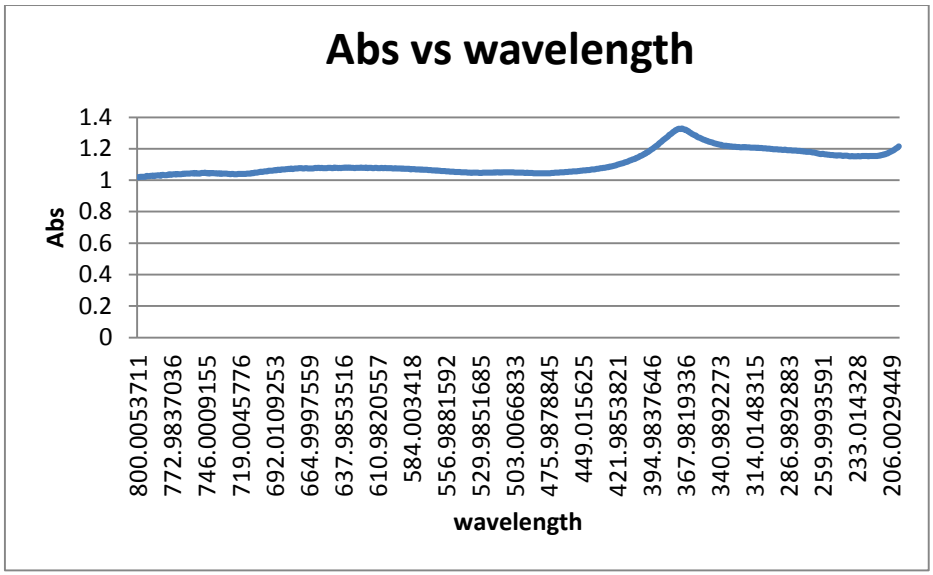

Fig.2 UV Visible spectroscopy

From Fig.2 for analytical study of the prepared sample, the amount of absorption within wave length of 360–380 nm was observed by uv-vis spectroscopy. Graph 6.1 shows the UV-Vis spectra of ZnO nanoparticles recorded between 200 and 800 nm. Absence of any other peak in the spectrum confirms that the synthesized products are zinc oxide only. As illustrated maximum peak value at 370 nm confirms the formation of ZnO nanoparticles in the solution.

XRD (X-ray diffraction)

Fig.3 represents the Xray diffraction pattern of ZnO nanopowder. A definite line broadening of the XRD peaks indicates that the prepared material consist of particles in nanoscale range. From this XRD patterns analysis, we determined peak intensity, position and width, full-width at half-maximum (FWHM) data. The diffraction peaks located at 31.84º, 34.52º, 36.33º, 47.63º, 56.71º, 62.96º, 68.13º, and 69.18º have been keenly indexed as hexagonal wurtzite phase of ZnO with lattice constants a = b = 0.324nm and c = 0.521nm (JPCDS card number: 36-1451) [5], and further it also confirms the synthesized nanopowder was free of impurities as it does not contain any characteristics XRD peaks other than ZnO peaks. The synthesized ZnO nanoparticle diameter was calculated using Debye-Scherrer formula

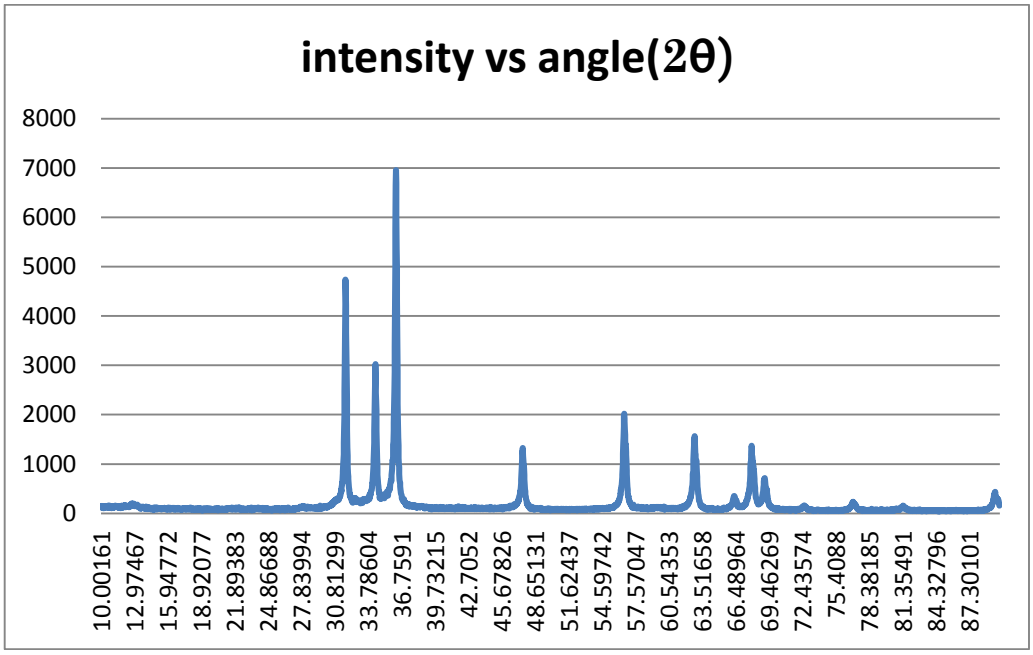

Fig 3 XRD (X-ray diffraction)

$d = 0.89\lambda/\beta\cos\theta$

Where the 0.89 is Scherrer's constant λ is the wavelength of X- rays, θ is the Bragg diffraction angle, and β is the full width at half maximum (FWHM) of the diffraction peak corresponding to the plane. The average particle size of the sample was found to be 16.21 nm which derived from the FWHM of more intense peak corresponding to the plane located at 36.33º using Scherrer formula [7].

Batch 2: without complexing agent

UV Visible spectroscopy

For analytical study of the prepared sample, the amount of absorption within wave length of 360–380 nm was observed by uv-vis spectroscopy. Fig.4 shows the UV-Vis spectra of ZnO nanoparticles recorded between 200 and 800 nm. Absence of any other peak in the spectrum confirms that the synthesized products are zinc oxide only. As illustrated maximum peak value at 368 nm confirms the formation of ZnO nanoparticles in the solution [1].

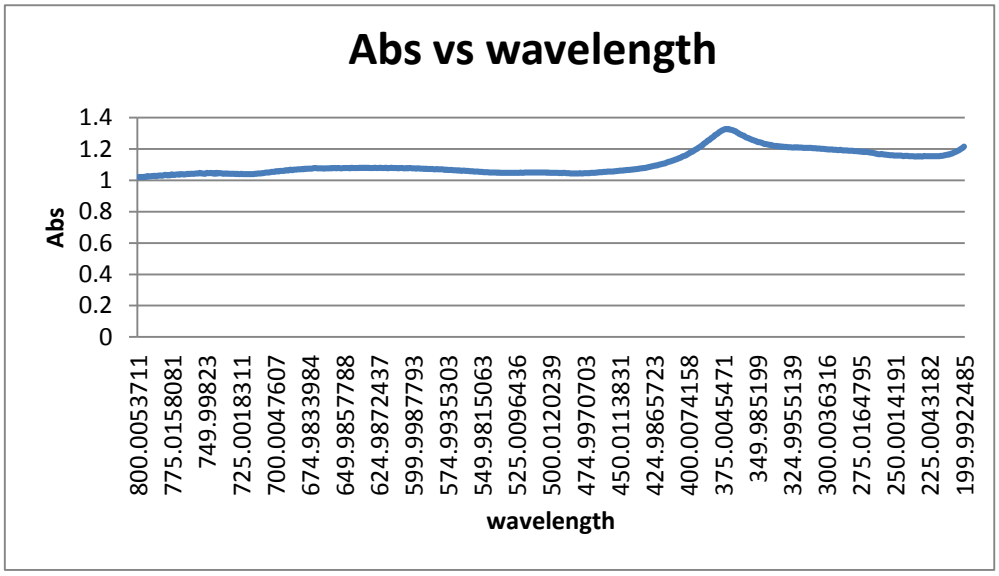

Fig.4 UV Visible spectroscopy

XRD (X-ray diffraction)

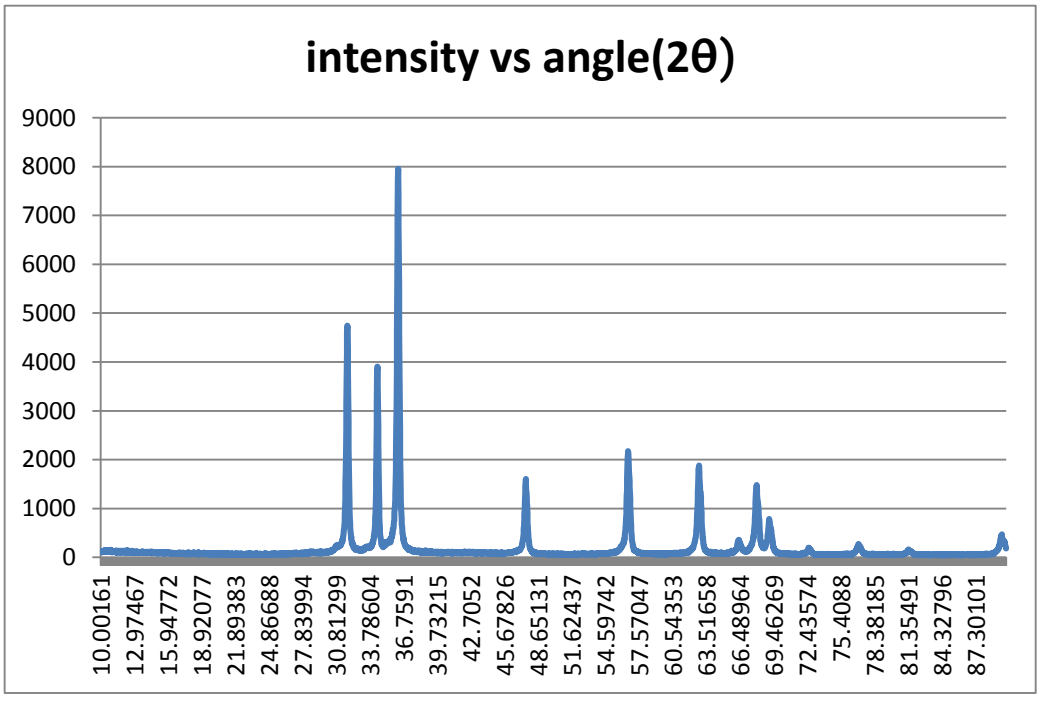

Fig.5 XRD (X-ray diffraction)

Fig.5 represents the Xray diffraction pattern of ZnO nanopowder. A definite line broadening of the XRD peaks indicates that the prepared material consist of particles in nanoscale range. From this XRD patterns analysis, we determined peak intensity, position and width, full-width at half-maximum (FWHM) data. The diffraction peaks located at 31.84º, 34.52º, 36.33º, 47.63º, 56.71º, 62.96º, 68.13º, and 69.18º have been keenly indexed as hexagonal crystal structures wurtzite phase of ZnO with lattice constants a = b= 0.324nm and c = 0.521nm (JCPDS Card #79-0208), and further it also confirms the synthesized nanopowder was free of impurities as it does not contain any characteristics XRD peaks other than ZnO peaks. The synthesized ZnO nanoparticle diameter was calculated using Debye-Scherrer formula

$$d = 0.89\lambda/\beta\cos\theta$$

Where the 0.89 is Scherrer's constant λ is the wavelength of X- rays, θ is the Bragg diffraction angle, and β is the full width at half maximum (FWHM) of the diffraction peak corresponding to the plane. The average particle size of the sample was found to be 47 nm which derived from the FWHM of more intense peak corresponding to the plane located at 36.18º using Scherrer formula [6].

Batch 3: complexing agent

UV Visible spectroscopy

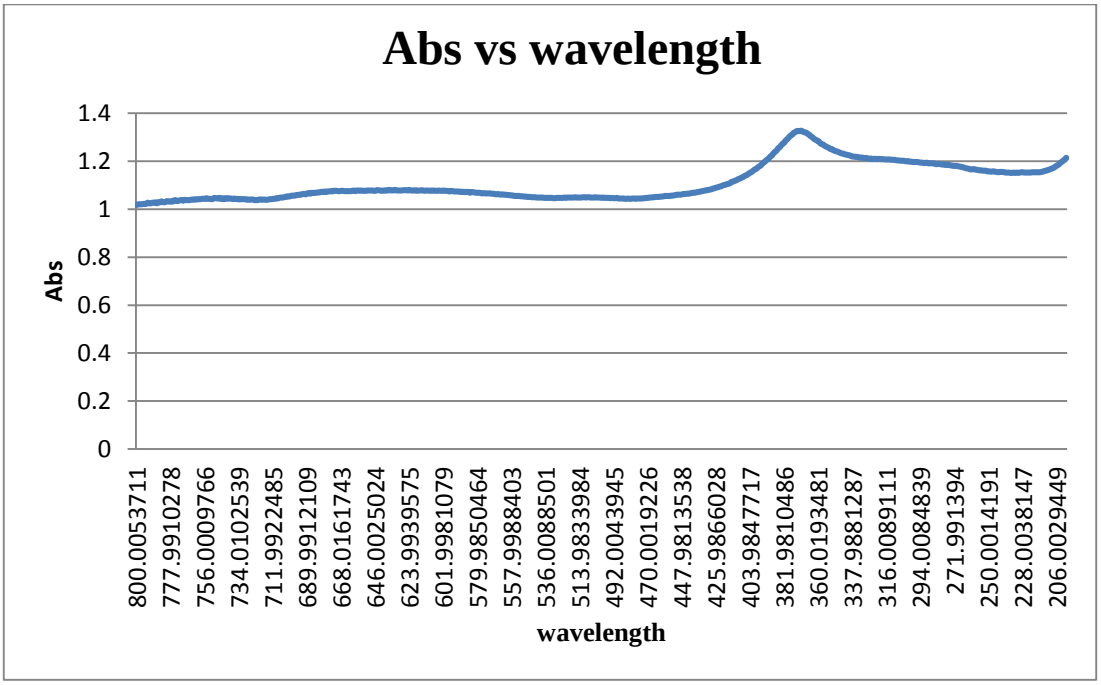

Fig.6 UV Visible spectroscopy

For analytical study of the prepared sample, the amount of absorption within wave length of 360–380 nm was observed by uv-vis spectroscopy. Fig.6 shows the UV-Vis spectra of ZnO nanoparticles recorded between 200 and 800 nm. Absence of any other peak in the spectrum confirms that the synthesized products are zinc oxide only. As illustrated maximum peak value it confirms the formation of ZnO nanoparticles in the solution [8].

XRD (X-ray diffraction)

Fig.7 represents the Xray diffraction pattern of ZnO nanopowder. A definite line broadening of the XRD peaks indicates that the prepared material consist of particles in nanoscale range. From this XRD patterns analysis, we determined peak intensity, position and width, full-width at half-maximum (FWHM) data. The diffraction peaks located at 31.84º, 34.52º, 36.47º, 46.13º, 56.71º, 63.96º, 68.13º, and 69.18º have been keenly indexed as wurtzite phase of ZnO with lattice constants a = b= 0.324nm and c = 0.521nm, and further it also confirms the synthesized nanopowder was free of impurities as it does not contain any characteristics XRD peaks other than ZnO peaks. The synthesized ZnO nanoparticle diameter was calculated using Debye-Scherrer formula

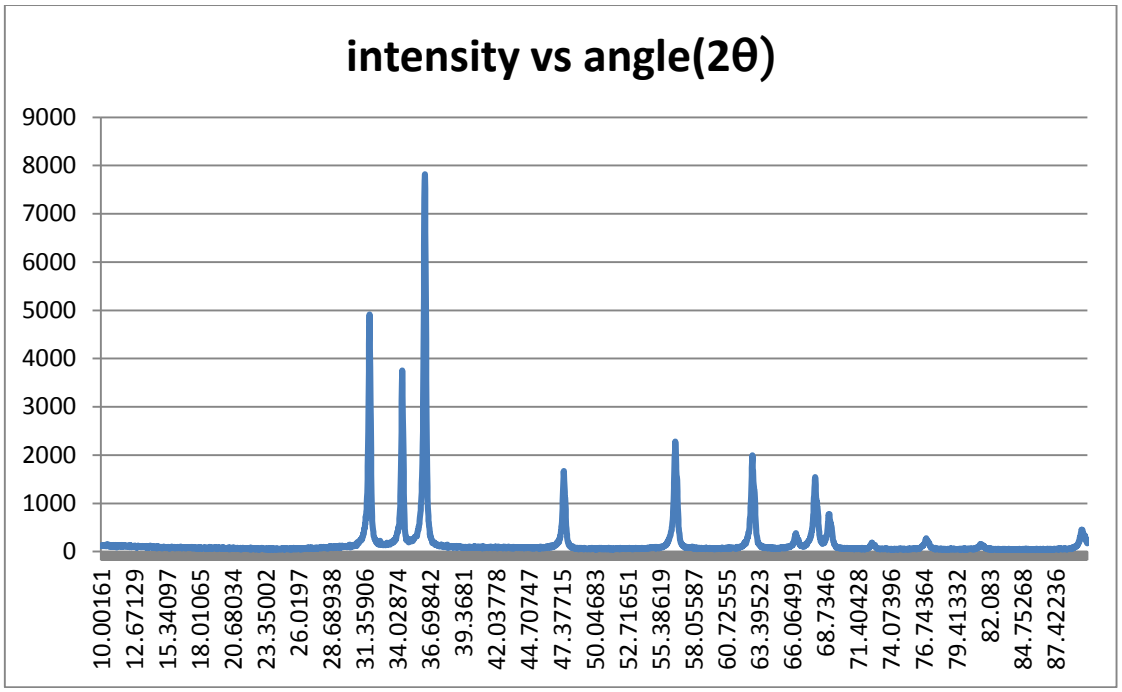

Fig.7 XRD (X-ray diffraction)

$$d = 0.89\lambda/\beta\cos\theta$$

Where the 0.89 is Scherrer's constant λ is the wavelength of X- rays, θ is the Bragg diffraction angle, and β is the full width at half maximum (FWHM) of the diffraction peak corresponding to the plane. The average particle size of the sample was found to be 40 to 50 nm which derived from the FWHM [8].

3.2 ANTIMICROBIAL ACTIVITY

All type of ZnO nanoparticles tested for their antimicrobial activity. Result given in table which that all ZnO and ZnO-PEG nanoparticles showed antimicrobial activity. It is evident that more activity was possessed by ZnO-PEG nanoparticles against gram- positive bacteria (Staphylococcus aureus) as compared to gram- negative (Escherichia coli) bacterial strains. A significant effect of complexing agent, i.e. PEG has been observed against resulting in enhancement of antimicrobial activity against all strains. The variation in activities between Gram negative and Gram positive bacteria is most probably due to difference in composition of cell wall, thickness of the wall and membrane, and presence of different groups on the surface [9]. It has been reported that ZnO-PEG nanoparticles bind and penetrate the cell wall of bacteria, more efficiently to Gram-negative bacteria (Escherichia coli) [10]. The resultant increased the antimicrobial activity by complexing nanoparticles might be due to combined activity of nanoparticles and complexing agent PEG. PEG are long chain molecules that interact with cell wall component changing the permeability of cell wall and cell membrane and also inside the cell these molecules interact with cellular molecules changing the functioning of the biocomponents.

Table 1 Antimicrobial activity of nanoparticles in terms of their zones of inhibition

Test sample	Zone of inhibition (mm) against bacterial strains	
	Staphylococcus Aureus	Escherichia coli
ZnO	10±0.71	7±0.73
ZnO-PEG	12±0.11	11±0.72

4. CONCLUSION

In summary, ZnO nanoparticles were synthesized and complexed with NaOH by using a simple chemical method and reflux method. X-ray diffraction patterns of ZnO and ZnO-PEG show that all samples exhibit wurtzite structure with no secondary phases. The characterizations of ZnO, ZnO-PEG nanoparticles revealed a decrease in size of ZnO nanoparticles after complexing. The band gap energy increases with the decrease in nanoparticles particle size. The antimicrobial activity of nanoparticles depicted a significant increase possessed by ZnO-PEG nanoparticles as compared to the without complexing ZnO

nanoparticles. All biological assays reveal highest activities in complexing ZnO nanoparticles as compared to the without complexing ZnO nanoparticles. Change in shape and size results in substantial changes. The ease of introduction of different types of reagents onto ZnO nanoparticles may broaden the molecular space in the search for new antimicrobial agents without complex chemical synthesis. In addition, the complexing ZnO can be further used for drug delivery applications.

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