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Research Article

SYNTHESIS OF ZnO NANOGEL FOR THE TREATMENT OF SUPERFICIAL SKIN MICROBIAL INFECTIONS

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ABSTRACT

Zinc oxide (ZnO) nanogel is an antimicrobial gel comprising of ZnO nanoparticles. Since, in nanoparticles, the surface to volume ratio is very large, therefore, ZnO nanogel is more effective for the treatment of superficial skin microbial infections as compared to other gels comprising ZnO and that has processed through traditional methods. Generally, infected skins are affected by bacterial strains which are attached to the surface of the skin by forming biofilms. The cause of attachment of these biofilms with the skin may be due to the electrostatic, vander walls, dipole-dipole and H-bonding hydrophobic types of interactions. ZnO nanogel when applied to the microbial infected skin reduces the microbial adhesion and biofilm formation. This screening characteristic of gel is due to the presence of ZnO nanoparticles of pore diameter 12.4 nm in the gel. The nanoparticles of ZnO is synthesized using sol-gel method and characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDS) and high resolution transmission scanning electron microscopy (HRTEM). The minimum inhibitory concentration (MIC) data tells that ZnO nanoparticles of such a small pore diameter inhibit the growth of microbes present on the skin surface.

Keywords: ZnO-nanoparticles; X-ray diffraction; Dipole-Dipole interaction; Antibacterial activity.

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1. INTRODUCTION

The synthesis, characterization and application of various nanoparticles like Ag, Ag₂O, Au, CaO, CuO, MgO, TiO₂, and SiO₂ etc have been discussed in details¹⁻⁹ in literature but here in this paper we first time report about the application of ZnO nanoparticles in the form of gel which have found tremendous application for the treatment of superficial skin microbial infections. The action of the gel is also superb, the nanoparticles present in the gel kill the biofilms present the infected skin which are attached to the skin by various interactions like dipole-dipole interaction, Vander walls interaction,

H-bond interaction and hydrophobic interactions. The pore diameters of the ZnO nanoparticles present in the gel are 12.4 nm which also supports the anti-microbial action of the gel.

ZnO is an n-type semiconductor. For a wideband bulk (pure) ZnO, energy band gap is 3.37 eV, binding energy is 60 meV¹⁰⁻¹². When the size of ZnO is made small in nm range, then due to quantum confinement of electron in the conduction band and holes in the valance band, the optical transition energy increases and absorption peak shifts to lower wavelength side¹³⁻¹⁶. For a size of 366 nm to 362 nm, the values of E increase from 3.43 to

3.47 eV. Absorption peak shifts to Ultra Violet (UV) region¹⁷. Gel of ZnO nanoparticles based on this method will show more effectiveness when applied on the skin.

ZnO exists in 3 different structures- hexagonal wurtzite, zinc blende and cubic rock salt¹⁸. Due to its unique characteristics like nontoxic and low cost, ZnO is used as a gas sensor, energy harvesting device like solar cells, veristors, pigments and cosmetics¹⁹. Various methods have been discussed for the synthesis of ZnO in literature but for our experiment, we have chosen sol-gel process because synthesis of ZnO nanoparticles are very easy by using this process and one can obtain very fine crystalline quality by using this process.

2. METHOD OF PREPARATION OF ZnO NANOGEL

An iron piece coated with polymer was refluxed in 100 ml of distilled water. The upper surface of the flask is attached with the coolant and was mounted on a magnetic stirrer. After 15 minutes of stirring, 5 ml of N-Cetyl-N, N-trimethyl ammonium bromide, a surfactant (CTAB) was added, followed by 10 ml of 0.1 M $Zn(CH_3COO)_2$ with continuous stirring. After 30 minutes 5 ml of 0.01 M of NaOH was added in the solution and the resulting mixture was further stirred for 30 min. The solution was kept at 35 °C for 2 hours. A 20 ml aliquot of this solution was taken in a tube and centrifuged at 2000 rpm. The supernatant was taken on indium tin oxide coated glass; a gel like structure is formed on the glass after 10 minutes.

The structure of the as-synthesized ZnO nanogel was analyzed by X-ray powder diffractometer (Shimatzou, XRD-6000), with CuK_{α} radiation of wavelength 0.15147 nm, working at 30 mA current and 20 kV voltage. The morphology and the composition of the synthesized ZnO nanogel were characterized by field emission scanning electron microscope (FSEM) equipped with an energy dispersive x-ray spectrometer (EDS). The particle size and orientation of synthesized ZnO nanogel was analyzed by the high-resolution transmission electron microscopy (HRTEM) and selected area electron diffraction (SAED) (JEOL EM-2100F) operated at a potential of 200 kV. The growth of bacteria was regulated at the Biology Department of University of Tabuk, KSA.

3. RESULT AND DISCUSSION

3.1 X-ray diffraction (XRD)

The prepared materials of ZnO were characterized using X-ray diffraction (XRD). When X-ray beams were incident on the crystals of ZnO, they get reflected from the crystal plane. The path difference between the rays are given by $2d\sin\theta=n\lambda$, where d is the crystal plane, λ is the wavelength of light and n is the order of diffraction pattern. Fig.1, tells that line broadening of XRD peaks of prepared material is in the nanometer range. Its intensity versus 2θ plot tells that peaks are located at definite angle of 2θ like 31, 34, 36, 47, 56 and 63 degrees respectively. This plot also tells that obtained

particles are partially crystalline in nature with hexagonal wurtzite phase without any impurity.

The average crystallite size of ZnO nanoparticles was determined by Scherer formula²⁰

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where D is the crystallite size, K the shape factor, λ is the wavelength, θ is the diffraction angle and β is the full width at half maximum (FWHM). Corresponding to the maximum intensity of diffraction plane (101), the average particle size of ZnO is found to be 12.4 nm.

3.2 Energy Dispersive X-ray Spectrometer (EDS)

The chemical composition mapping of synthesized ZnO nanogel was examined by using energy dispersive x-ray spectrometer (EDS) as shown in Fig. 2, which reveals that Oxygen peaks occur at energy 0.54 keV while peak of Zn occurs at energy 1.04 keV, 8.60 keV and 9.53 keV. confirming that the grown material is pure ZnO. No other peaks related to impurities were detected in the spectra within the detection limit of the EDS pattern²¹.

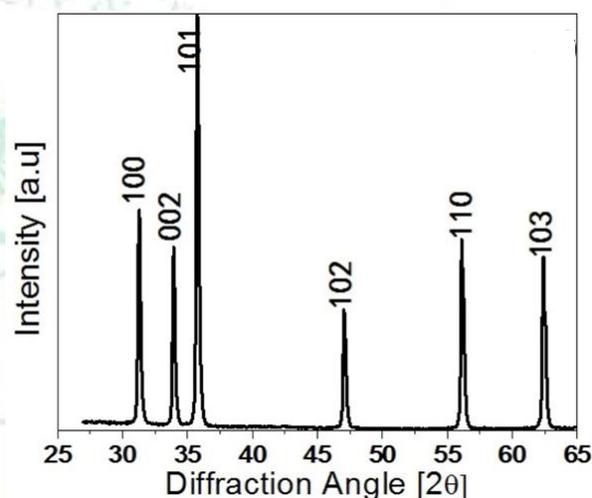


Figure 1: XRD of ZnO Nanoparticles

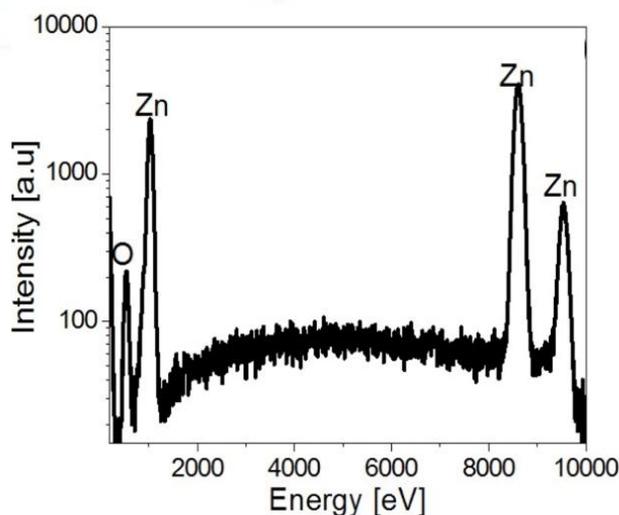


Figure 2: EDS spectra of ZnO nanoparticles

3.3 The selective area electron diffraction (SAED)

Furthermore, the morphological characterization of the prepared ZnO nanogel was analyzed by transmission electron microscopy equipped with the selected area electron diffraction (SAED). Fig. 3 presents a low magnification TEM micrograph of ZnO nanoparticles. It is clear that ZnO possesses uniform shaped nanoparticles with an average size of 12.4 nm with no glassy or amorphous interface along the grain boundaries. It can also be seen that the synthesized ZnO is composed of well defined crystals with a hexagonal wurtzite structure²². The results obtained from TEM images are inconsistent with the particle size estimated by the FWHM of the XRD.

The selective area electron diffraction (SAED) pattern in Fig. 3 further proves that the ZnO particles present in the nanogel has uniform structure and their crystalline structure consists of no amorphous phase. In addition, the diffraction images consist planes indexed as (100), (002), (101), (102), (110), (103), (200) and (201) reflections which are matching to the typical hexagonal phase of ZnO.

3.4 SEM Analysis

The structural characterization of ZnO was carried out using SEM. The morphology of the ZnO is presented in Fig 4. The SEM images of particle at higher magnifications show that the smallest particle size of ZnO was found to be 12.4 nm. The SEM result was in accordance with results obtained from XRD.

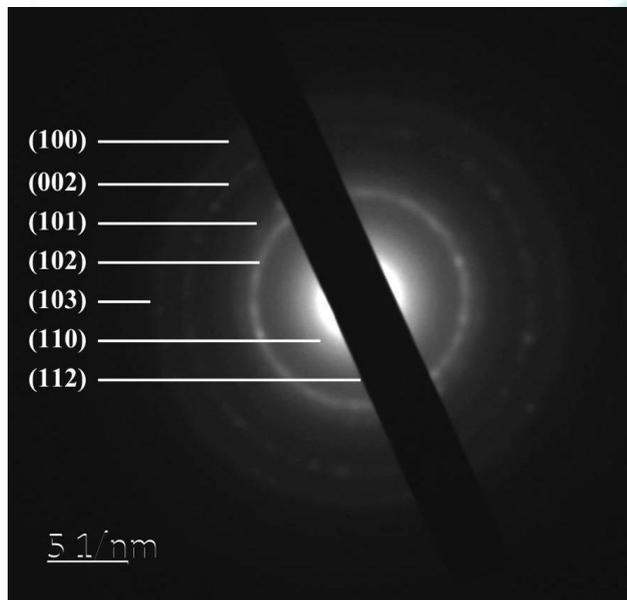


Figure 3: TEM of ZnO nanogel consisting Nanoparticles

3.5 Antibacterial Activity

Antibacterial screening of the ZnO nanogel was examined against different microorganisms. The relationship between the bacterial growth of *E. coli* and *Bacillus* sp. with different concentrations of ZnO nanogel is depicted in Fig. 5. It can be seen that the bacterial growth decreases linearly with increasing concentrations of ZnO nanogel up to 600 $\mu\text{g/ml}$ showing

a constant decrease in bacterial growth, suggesting that ZnO nanogel has a dose-dependent antibacterial activity as reported earlier^{10, 14}. At higher dose in the range of 600 $\mu\text{g/ml}$ to 1000 $\mu\text{g/ml}$, antibacterial activity is independent. Thus, dose consideration is the important issue for the preparation of gel.

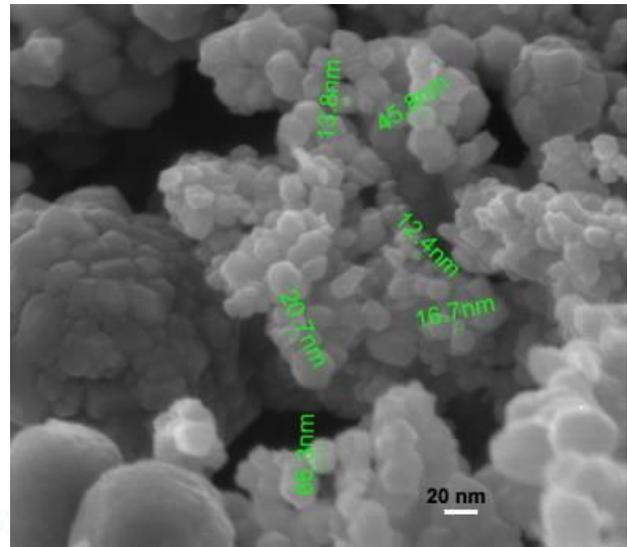


Figure 4: SEM of ZnO Nanoparticles

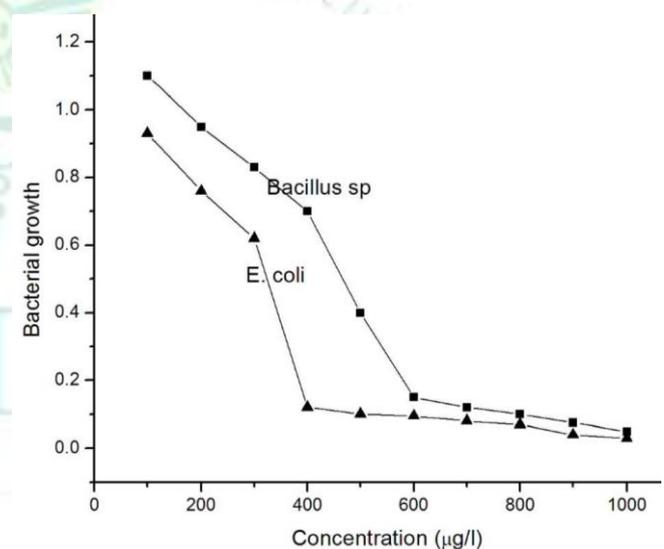


Figure 5: Plot of bacterial growth versus concentration

CONCLUSIONS

Synthesis of ZnO nanoparticles in the form of gel is the new approach to study the antibacterial action against infected skin. We observe choice of the particle in nm range and dose consideration plays an important role in the effectiveness of the gel. The presence of nanoparticles increases UV activity necessary for the killing of biofilms present on the infected skins.

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