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# Synthesis and Antibacterial Activity of ZnO Nanoparticles Using the Precipitate and Irradiation Methods

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**Abstract:** In this study, ZnO NPS were synthesized in two methods: the precipitate and irradiation methods. X-Ray powder Diffraction, Transmission and Scanning Electron Microscopy were used to study the crystal structure and surface morphology of the synthesized NPS, which showed that the average particle size of ZnO NPS synthesized by irradiation method was better than which synthesized by precipitate method. The antibacterial activity against *staphylococcus epidermidis* and *E.coli* bacteria were studied and showed that ZnO NPs prepared by Irradiation method have higher antibacterial activity against *S.epidermidis* and *E.coli* than ZnO NPs prepared by precipitate method because of ZnO NPs prepared by Irradiation method smaller than ZnO NPs prepared by precipitate method. In current study, we concluded that irradiation method is better than precipitate method in the preparation of ZnO NPS and in the inhibition of the work of types of bacteria such as (*staphylococcus epidermidis* and *E.coli*).

**Keywords**: ZnO NPS, X-ray, TEM, SEM and antibacterial activity.

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## Introduction:

Nanoparticles can be generally defined as a particle that has a structure in which at least one of its phases has one or more dimensions in the nanometer size range (1). Nanoparticles have attracted a great attention in recent years because of their unique physical and chemical properties such as high strength with good thermal conductivity, higher damping property and mechanical stability (2,3) and because of the ease with which they can be synthesized and modified chemically(4). Metal oxides play a very important role in many areas of chemistry, physics and materials science. The metal elements are able to form a large diversity of oxide compounds. These can adopt a vast number of structural geometries with an electronic structure that can exhibit metallic, semiconductor or insulator character (5). In recent years, metal oxide nanoparticles have a great attention because of the modification of properties perceived due to size effects, distribution and morphology due to present a higher surface-to-volume ratio with decreasing size of nanoparticles, thus modifying the catalytic, electronic, and optical properties of the metal NPs (6,7). Among various metal oxides, zinc oxide (ZnO) nanoparticles is the most promising candidate because of its unique physical and chemical properties such as high chemical and mechanical

stability (8). Their size-dependent electronic and optical properties (9), a good transparency, high electron mobility, wide band gap and strong room temperature luminescence. ZnO nano particles have many applications, such as Nano generators and gas sensors (10). Solar cells, biosensors, (11). Varistors, photo detectors and photo catalysts (12). There are many methods to prepare Nanoparticles such as: gas-condensation, Vacuum Deposition and Vaporization, Chemical Vapor Deposition (CVD) and Chemical Vapor Condensation (CVC), Mechanical Attrition, Chemical Precipitation, Sol-Gel Techniques, Electrodeposition and irradiation methods (13). In this work the chemical precipitation and irradiation methods were carried out.

## Experimental:

### Preparation of ZnO NPS by precipitation method:

50 ml of $Zn(NO_3)$. $6H_2O$ (1M) was mixed with urea under stirring for 10 minutes at room temperature, 50ml of NaOH (1M) was added drop a wise to the solution under vigorous stirring and amount of precipitate was formed, this precipitate was filtered and washed with distilled water for many times and dried in oven at 80$^o$C for 1 hour, finally the powder obtained was calcined at 350$^o$C for 3 hours to obtain ZnO NPS (14).

### Preparation ZnO NPS by irradiation method:

The irradiation system in (Figure 1) was used to prepare ZnO NPS.

**Figure (1): The Irradiation System**

The irradiation system consist of mercury lamp with power 125W placed inside quartz tube, which placed inside Pyrex tube used as reactor. 50ml of $Zn(NO_3).6H_2O$ (1M) was mixed with 0.5g of urea under stirring for 10 minutes at room temperature, 50ml of $NaHCO_3$ (1M) was added drop a wise to solution under stirring. The mixture was placed inside the Pyrex tube and then irradiated for 30 minutes using ice bath and amount of precipitate was formed, this precipitate was filtered and washed with distilled water for many times and dried in oven at 80$^o$C for 1 hour, finally the powder obtained was calcined at 350$^o$C for 3 hours to obtain ZnO NPS.

### Preparation of bacterial culture solution:

The antibacterial activity of ZnO NPS, which prepared by both

precipitate and irradiation methods against the positive and negative bacteria was determined by well diffusion test. Positive and negative bacteria cultured in nutrient broth for 24 hours at 37°C. Inhibition ability of prepared NPS by both irradiation and precipitate synthesis was tasted against bacteria gram positive (*staphylococcus epidermidis*) and bacteria gram negative (*E.coli*). In a petri dished containing nutrient agar as a culture media with two holes (5mm each one). These holes filled with nanoparticles solution ZnO and incubated for 24h at 37°C.

## Results and Discussion:

### X-ray diffraction (XRD):

The XRD pattern of the ZnO NPS, which prepared by both precipitate and irradiation methods are shown in (Figure 2 and 3) respectively. This pattern represent of the ZnO cubic phase depend on diffraction peaks at $2\theta$ values of 31.7°, 34.4°, 36.2°, 47.5°, 56.8°, 62.8°, 67.9°, 69.0°, 72.5° and 76.9° correspond to Miller Indices (*hkl*) (100), (002), (101), (102),(110), (103), (112), (201), (004) and (202) in two methods . The crystallite size was calculated from diffraction peaks using the Debye-Scherrer equation 1, which was found to be 18.55 nm in precipitate method and 12.24nm in irradiation method.

$$D = \frac{k\lambda}{B\cos\theta} \qquad \ldots\ldots(1)$$

Where k is Scherrer constant, B is the full width at half-maximum, λ is the X-ray wavelength, and θ is the Bragg diffraction angle.

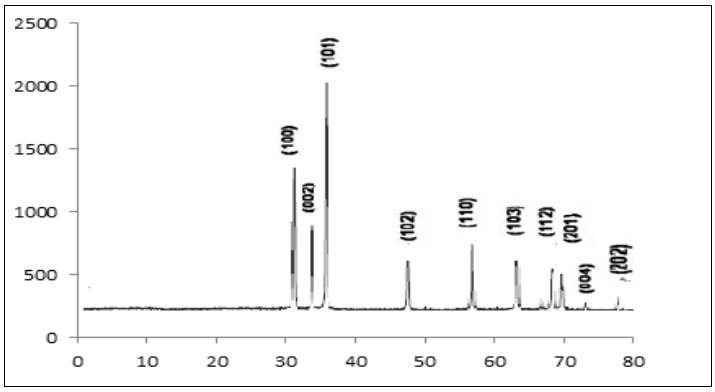


**Figure (2): X-ray diffraction (XRD) patterns of ZnO Nanoparticle prepared by precipitate method.**

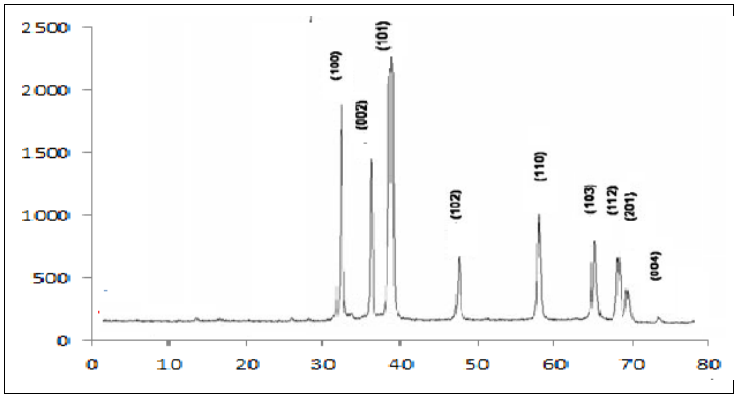


**Figure (3): X-ray diffraction (XRD) patterns of ZnO Nanoparticle prepared by irradiation method.**

## TEM and SEM microscopy:

The average particles size of ZnO nanoparticles prepared by precipitate and irradiation methods were determined on the TEM and SEM images. (Table 1, Figures 4 and 5) showed TEM of ZnO nanoparticles prepared by precipitate and irradiation methods respectively and (Figures 6 and 7) showed SEM of ZnO nanoparticles prepared in precipitate and irradiation methods respectively.

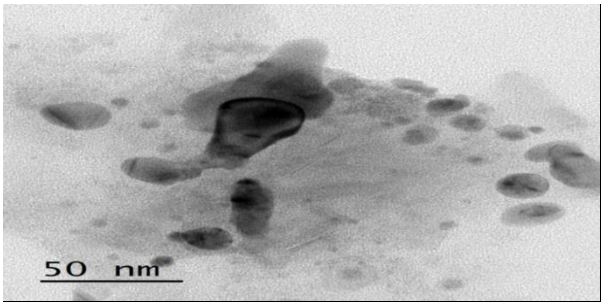


**Figure (4): TEM of ZnO nanoparticles prepared by precipitate method.**

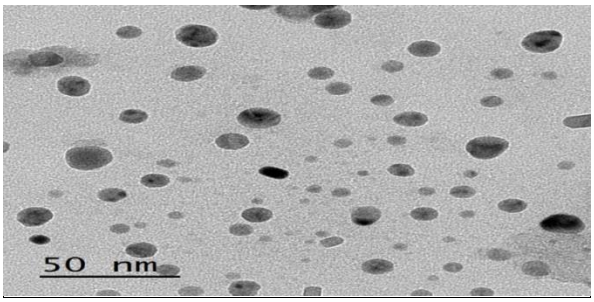


**Figure (5): TEM of ZnO nanoparticles prepared by Irradiation method**

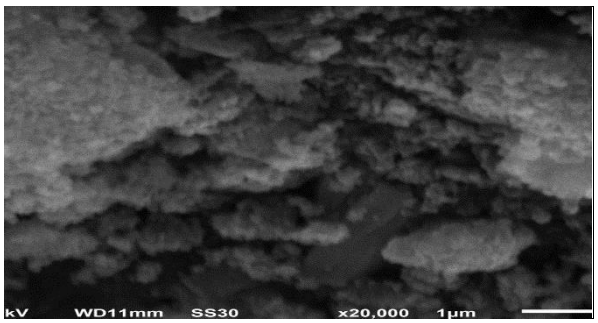


**Figure (6): SEM of ZnO nanoparticles prepared by precipitate method.**

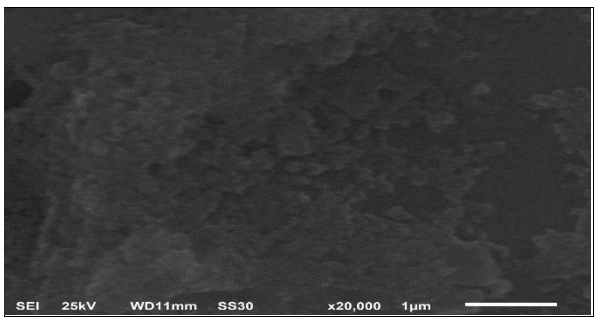


**Figure (7): SEM of ZnO nanoparticles prepared by Irradiation method.**

(Figure 6) shows the consist of aggregated and small crystals and (Figure 7) shows that the increase agglomeration occurs between particles with increasing the concentration of NaOH.

**Table (1): Average particles size of TEM and SEM of ZnO nanoparticles by precipitate and irradiation methods**

| | TEM | SEM |
|---|---|---|
| **Method** | **Average Particles size** | **Average Particles size** |
| **Irradiation** | 14 | 22 |
| **Precipitate** | 20 | 40 |

## Antibacterial activity of ZnO NPS:

ZnO NPS prepared by precipitate method gave inhibition zone 6mm with *staph. epidermidis* bacteria and 5mm in *E.coli* bacteria. Whereas ZnO NPS prepared by irradiation method gave inhibition zone with 13 mm for *staph. epidermidis* bacteria and 7 mm in *E.coli* bacteria. The concentration of 1 mg/ml of nanoparticle was used as shown in (Table 2).

**Table (2): Inhibition areas (mm) observed for ZnO Nanoparticle prepared by both precipitation and irradiation methods against gram positive (*Staphylococcus epidermidis*) and bacteria gram negative (*E.coli*).**

| Method | Conc. of ZnO NPs (mg/ml) | Inhibition zone (mm) | |
|---|---|---|---|
| | | ***Staphylococcus epidermidis*** | **E.coli** |
| **Precipitate Method** | 1 | 6 | 5 |
| **Irradiation method** | 1 | 13 | 7 |

ZnO NPs prepared by Irradiation method have higher antibacterial activity against *S.epidermidis* and *E.coli* than ZnO NPs prepared by precipitate because of ZnO NPs prepared by Irradiation method smaller than ZnO NPs prepared by precipitate method, as shown in (Figures 8 and 9). This agree with Siddiqi *et al* (15) they observed that ZnO NPs ability for bacterial inhibition was strongly dependent on particle size. The cell membrane of gram positive bacteria can be damaged more than gram-negative bacterium because of gram positive bacteria have no outer membrane in the cell wall and thick wall composed of multi layers of glycol peptide (16).

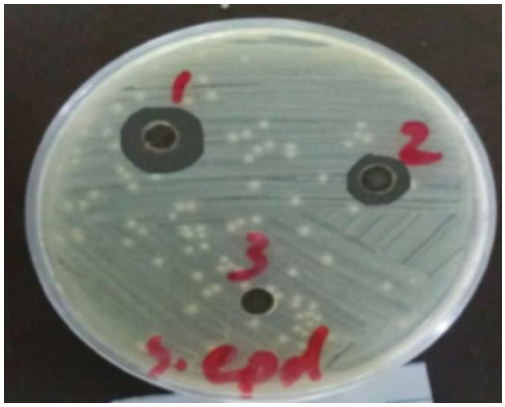


**Figure (8): Inhibition Zone of ZnO NPS prepared by (1) Irradiation method (2) precipitate method for *S. epidermidis* (3) solvent (D.W).**

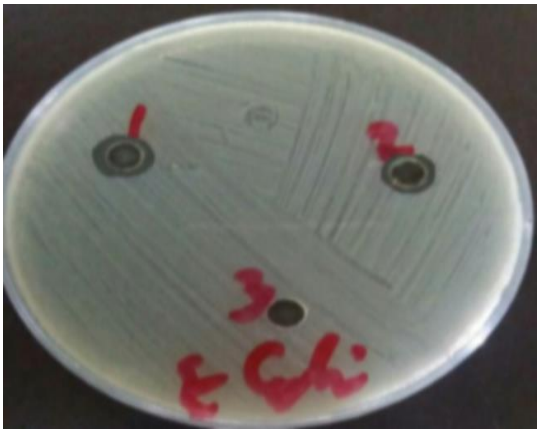


**Figure (9): Inhibition Zone of ZnO NPS prepared by (1) Irradiation method (2) precipitate method for *E.coli.* (3) solvent (D.W).**

## Conclusions:

In this work, we concluded that irradiation method is better than precipitate method in the preparation of ZnO NPS and in the inhibition of the work of types of bacteria such as (*staphylococcus epidermidis* and *E.coli*).

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