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# FABRICATION OF ZINC OXIDE NANOPARTICLES BY CHEMICAL AND BIOLOGICAL METHODS

**Ragab Asmaa M.E.[1]; Eisa Nawal A.[2]; Mohamed F.G.[2]; Abu-Zeid N.M; Farroh K.Y.[3] and Hassan Eman, O.[2]**

* Plant Pathology Research Institute, Agricultural Research Center, Giza, Egypt; **Plant Pathology Department, Faculty of Agriculture, Moshtohor, Benha University, ***Nanotechnology and advanced Materials Central Lab. Agricultural Research Center, Giza, Egypt.
Corresponding author: Ragab Asmaa M.E. (am1980161@gmail.com)

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## ABSTRACT

In the current study, nanoparticles (NPs) of zinc oxide (ZnO) were prepared followed by determination of their characters among chemical and biological tools. In aqueous media from zinc acetate dihydrate and sodium hydroxide ZnONPs were fabricated by a precipitation method. To characterize the physicochemical properties of ZnO NPs transmission electron microscopy (TEM) and X-ray diffraction (XRD) were applied. The chemically fabricated ZnONPs (cZnO NPs) characterized with by crystalline structure with hexagonal structure of the wurtzite. The morphology of cZnO NPs was spherical with a primary size of 5-30 nm. Aqueous leaves extract of *Ocimum sanctum* was used for biological preparation, which may be more ecofriendly and economical compared to other commonly used methods. Results of XRD patterns confirmed the crystalline nature of biologically fabricated ZnONPs (bZnO NPs). Electron micrographs revealed that the resultant bZnO NPs characterized by a spherical shape and a particle size ranged from 8.92 to 37.9nm.The results revealed the crystalline nature of biologically produced ZnO NPs (bZnO NPs). Electron micrographs of the fabricated ZnO NPs also were identified with spherical shape and a particle size distribution of 8.92-37.9 nm.

**Key words**: Fabrication, Zinc oxide (ZnO), Nanoparticles (NPs), Characterization, *Ocimum sanctum*, Biological preparation.

## INTRODUCTION

It has been recently proven that nanotechnology (NT) uses several scientific fields through which materials and devices at nanometer scale are operated by these technologies **(Vaseem *et al*., 2010).** With a size of up to 100 nm, NT can be used to manufacture and apply some materials of this size. **Adams and Baughman (2005)** showed that Nanoparticles (NPs) have revolutionized all important industrial areas, including drug delivery to the food and agricultural industry.

Inorganic NPs composites have important properties at the scientific level represented in their antagonistic activity against fungi and bacteria at a very low concentration, due to their small size, increased surface-to-volume ratio, and outstanding physical and chemical properties **(Rai *et al*., 2009)**.NPs also characterized by its stability at pressure and high temperature **(Sawai, 2003)**.

**Kumar and Rani (2013)** reported that ZnO NP oxide is one of the most frequently applied nanomaterials in numerous commercial products for its ability to absorb ultraviolet rays, its antimicrobial activity, its catalytic, semiconducting and magnetic properties. Accordingly, ZnO NP has been widely used in many products such as sunscreen products, personal care products, paints, electronic materials, food additives, rubber industry and pharmaceuticals. Because of the unusual electrical, magnetic, chemical, optical, and mechanical characteristics of nanoparticles, they differ greatly from bulk ZnO.

As the size of a particle decreases with ZnO, its transparency to visible light increases and the chemical reactivity increases, while the small size ZnO is characterized by its opacity and low reactivity. That is why ZnO NPs have been widely used in the production of various materials that require high transparency or reactivity. It is being utilized as a food additive after being recently classified as recognized as Safe (GRAS) by the Food and Drug Administration Agency **(Espitia *et al*., 2012)**.The human body contains approximately two to three grams of zinc, while the body requires 10 to 15 mg daily **(Patnaik, 2003** and **Auer *et al*., 2005)**. Regarding the negative effects of these compounds in humans, such as the possibility of developing cancer, reproductive and genetic toxicities in humans, there are no scientific reports has demonstrated **(Auer *et al*., 2005** and **Araujo-Lima *et al*., 2017)**.

**Parashar *et al.* (2009)** showed that nanomaterials can be synthesized in several ways which are chemical, physical or green synthesis methods. The chemical methods proved to be the most popular method due to its low cost and reliability compared to the other method. The size and morphology of the NPs are strictly controlled by this approach, however it is possible that some poisonous compounds could end up present as a result of chemical adsorption on the surface, which would make them detrimental in medical applications. Therefore, the use of green methods for the fabrication of zinc oxide is attributed to the small amount of pollutants that can be produced, as well as being energy-saving as well as cost-effective compared to the chemical methods used. Plants and their extracts, algae, bacteria, and viruses, are great moieties for the synthesis of ZnO NPs, since no need to use hazardous materials in addition to the laborious process of cultivation and downstream processing. Therefore, plant extracts are becoming more and more attractive due to the simplicity of the methodology and it is cost effective **(Mashwani *et al.*, 2015; Mashwani *et al.*, 2016** and **Jain *et al*., 2020)**.

One of the most explored sources for the synthesis of NPs is the biosurfactant that is based on plant extracts, because plants are characterized by their high ability to synthesize NPs. The mechanism of plant NPs fabrication is their ability to absorb minerals from water and soil and their excessive accumulation and further reduction to recoverable NPs. A lot of the primary and secondary metabolites that are found in plants are starches, tannins, saponins, terpenoids, phenols, flavonoids, and polypeptides, which act as distinct reducing agents in addition to capping agents. Light solvents such as water, ethanol, or methanol are employed to extract the plant metabolites, which are allowed to react with the zinc salt solution under different conditions to produce the highest yield from the production of zinc oxide NPs by plants such as *Mentha spicata* **(Abdelkhalek and Al-Askar, 2020)** *Melia azedarach* **(Lakshmeesha *et al.*, 2020)**), *Azadirachta indica* **(Ali *et al.*, 2021)**, *Nigella sativa* **(Awan *et al*., 2021)**.

## MATERIALS AND METHODS

**Preparation of zinc oxide NPs: Chemical Reagents:** Zinc acetate dihydrate was obtained from Lobachemie, India. Sodium hydroxide pellets were purchased from Emsure®, Germany. Chemicals of analytical reagent grade were employed in the experiment without additional purification. During the whole experiment, both distilled water and deionized water were used to make solutions and for washing.

**Preparation of zinc oxide NPs *via* chemical precipitation method:** In the current study, powder zinc oxide NPs were prepared using wet-chemical co-precipitation method according to the protocol of **Koutu *et al.* (2016).**To obtain the final zinc oxide product, the final precipitate was dried at 50°C and then annealed at 600˚C for three hours to get crystalline ZnO-NPs before being ground into NPs powder.

**Biological fabrication of zinc oxide NPs using aqueous *Ocimum sanctum* leaves extract:**

**Preparation of *Ocimum sanctum* leaf extract:** Leaves of *Ocimum sanctum* were collected from Tersa village (Tukh, Qalyubia). To remove dust the fresh leaves were rinsed several times with distilled water (d.$H_2O$). On completely drying, leaves were chopped into a fine pieces, an amount of 5 g of chopped leaves were boiled at 60°C for 1 hour with 50mL $H_2O$. The extract was filtered and centrifuged for 15 min at 7000 rpm. The supernatant was obtained in a new tube and kept at 4°C for further studies**.**

**Biological fabrication of zinc oxide NPs:** Biological fabrication of zinc oxide NPs was determined as described by the method of **Shende *et al.*, (2016).** The final pellets were dried at 50°C before annealing at 600°C for three hours to get crystalline NPs. The annealed samples were ground into a NPs powder and then subjected to characterization.

**Characterization of fabricated NPs:** Chemically and biologically fabricated zinc oxide NPs (cZno NPs and bZno NPs) were analyzed by X-ray diffraction (XRD). The morphological features of the synthesized NPs were visualized using a transmission electron microscopy (JEOL TEM 100CXII, Tokyo, Japan).

## RESULTS AND DISCUSSION

**Characterization of fabricated NPs:** Ps were synthesized with enhanced particle properties using chemical routes that help in controlling the surface energy. The functionalized particles were characterized.

**XRD of zinc oxide NPs:** Data in **Figure-1** show the XRD pattern of cZnO NPs **chemically fabricated**. The XRD peaks indicate that the prepared material is consisted of particles in nanoscale range. Therefore, XRD patterns analysis determined peak intensity, position and width, full-width at half-maximum data. The diffraction peaks was located at 31.75◦, 34.41◦, 36.24◦, 47.54◦, 56.59◦, 60.50, 62.85◦, 66.36◦, 67.94◦, 68.15◦, 69.09◦, 72.57◦and 76.93◦ and have been keenly indexed as hexagonal wurtzite phase of ZnO.

This results agrees with that found by **Zhou *et al*., (2007)** and **Khoshhesab *et al*., (2011)**. **JCPDS (1977)** confirmed that the fabricated nanopowder was free of impurities as it does not contain any characteristics XRD peaks other than ZnO peaks.

Diameter of the fabricated ZnO nanoparticle was calculated using Debye-Scherrer formula. The average particle size of the sample was found to be 17.45 nm which is derived from the FWHM of more intense peak corresponding to 101 plane located at 36.24° using Debye-Scherrer's formulad = 0.89λ /β cos θ (**Cullity 1967)**.

Results in **Figure-2** showed that the XRD pattern of the biological fabricated zinc oxide NPs (bZnO NPs) exhibited well-defined relatively broad peaks with high intensity which indicates the formation of nanocrystalline phase. These peaks are indexed within the hexagonal ZnO wurtzite-type structure, in agreement with that of Joint Committee on Powder Diffraction Standards. No additional peaks were detected within the resolution limit of the X-ray diffractometer. Crystallite size of prepared Zn ONPs was calculated around 14.54 nm; also, lattice parameters were found to be about 3.252 and 5.209, respectively.

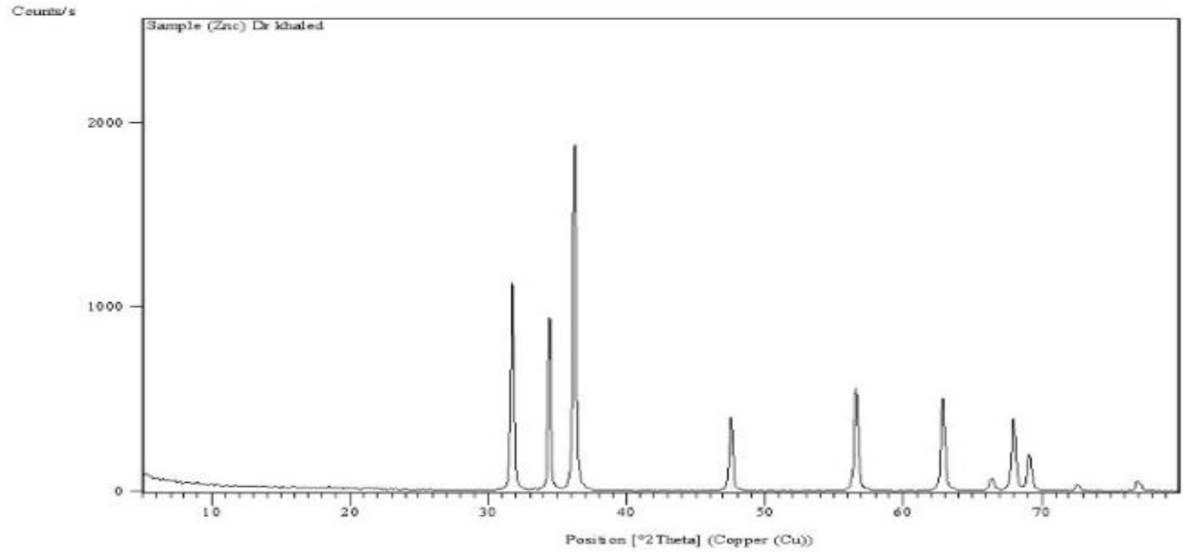


**Fig. 1: XRD patterns of cZnO NPs annealed at 600°C**.

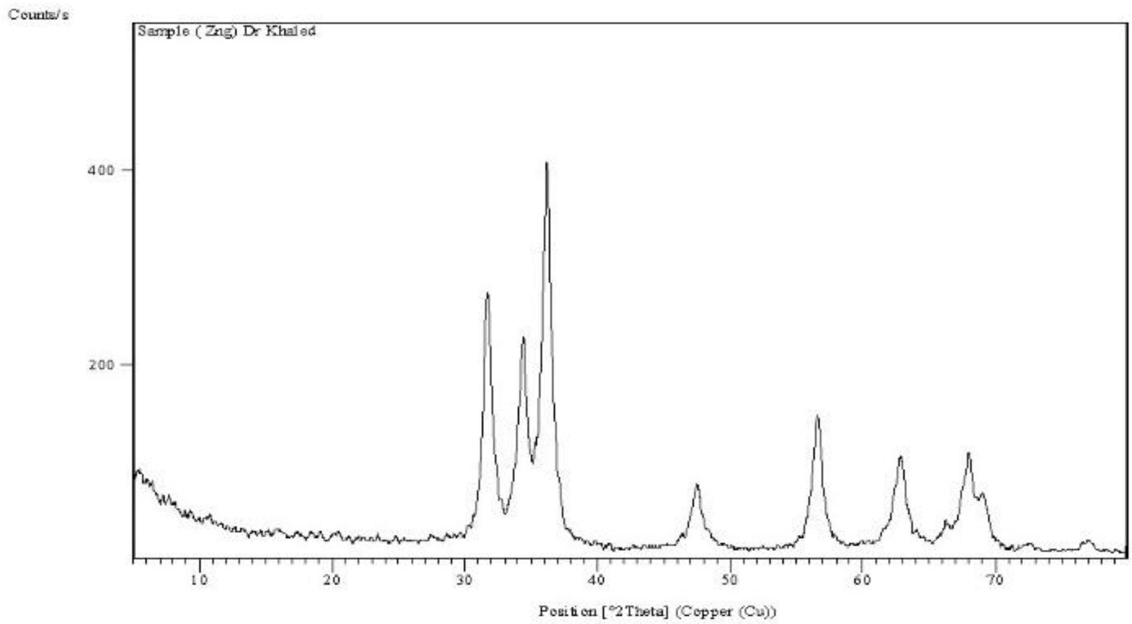


**Fig. 2. XRD patterns of bZnO NPs annealed at temperature 600˚C.**

**Transmission electron microscope analysis of the zinc oxide NPs:** Transmission electron microscopy is a useful tool for the determination of NPs morphology. It gives us information about the topography of the sample species. The shape and particle size of prepared NPs was determined by TEM. Most of ZnONPs shapes were predominantly spherical with size 5-30 nm and 8.92-37.9 nm forcZnO NPs and bZnO NPs, respectively. Also the average particle sizes cZnO NPs **(Figure-3)** and bZnO NPs **(Figure-4)** are 23.8 nm and 19.36 nm, respectively

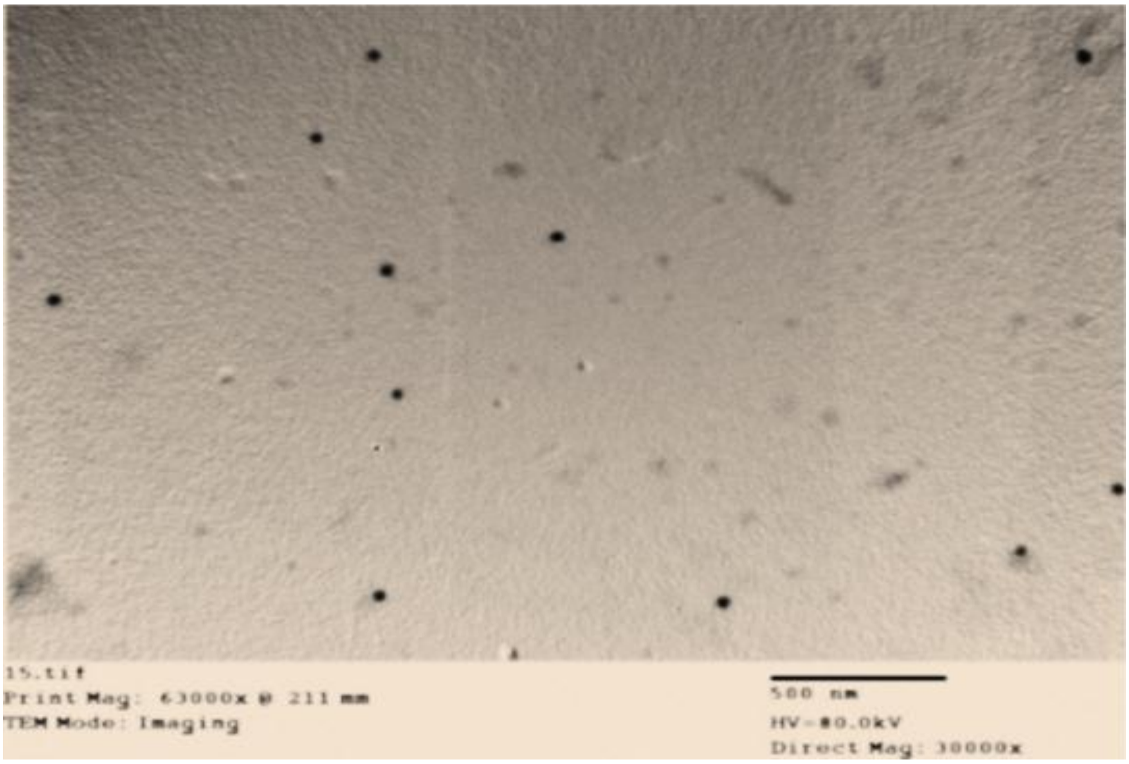


**Fig. 3. Transmission electron microscopy of cZnO NPs.**

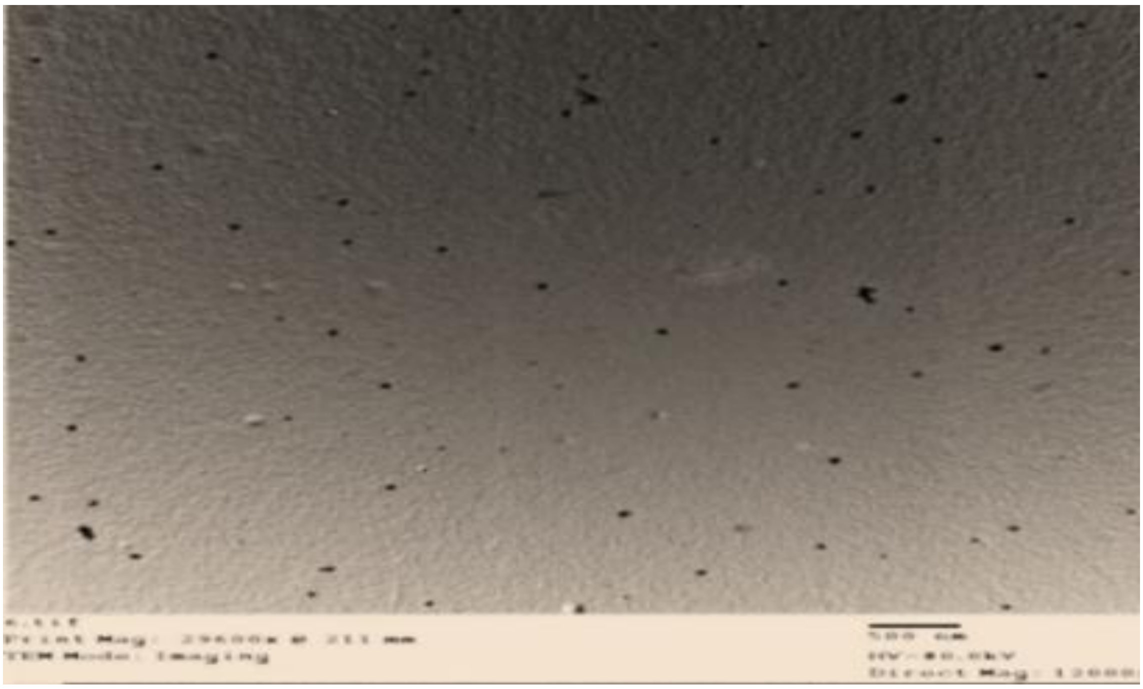

**Fig. 4. Transmission electron microscopy of bZnO NPs**.

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