ISSN: 0975 -8542

Journal of Global Pharma Technology

Available Online at: www.jgpt.co.in

RESEARCH ARTICLE

Electrochemical Preparation and Characterization of Zinc Oxide Nanoparticles as a Model for Drug Delivery in Pharmaceutical Compounds

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Abstract

Zinc oxide nanoparticles (ZnO NPs) were prepared by electrochemical method in solution contains NaNO3 as electrolyte and Polyvinylpyrrolidone (PVP) as stabilizer. The prepared ZnO NPs were characterized by EDX, SEM, TEM, and AFM studies .EDX-Spectrum of ZnO nanoparticles exhibited signals of zinc and oxygen only, and that means the sample of ZnO NPs having high purity. SEM micrographs givesa homogeneous particles distribution and having sheets like structure. From TEM study the size of ZnO NPs was found to be about 60 nm and the shape of nanoparticles was a bipyramid like shape. The average size of synthesized ZnO NPs from AFM study was found to be 60.99 nm; this result was agreement with result that obtained from TEM.

Keywords: *Electrochemical Preparation, Zinc Oxide Nanoparticles, AFM; SEM; TEM.*

Introduction

Nanotechnology includes the study, control and processing of materials at the nanometer scales, typically they having less than 100 nanometers dimensions [(1-2)]. The properties of such materials are new and can be adjusted by controlling the dimensions of these structures and their synthesis by biological, chemical or physical methods. [(3)]Nanostructured semiconductor metal oxides are of large important for many technologic applications due to their various interesting electronic and optical properties.

The design and preparation of nanostructured semiconductor oxides with a adjusted physical and chemical property for advanced applications have drawn a large deal of interest in the field of catalysis [(4-7)]. Metal oxide nanoparticles semiconductors possess widely developed in the past decenniums. They have been vastly used in several applications like catalysts, sensors, capacitors, cosmetic, batteries, medical and engineering science [(8–13)]. Among them zinc oxide that have many unique characteristics such as high-specific surface area, chemical stability, non-toxicity, high electron communication features, and electrochemical activity [(14-18)].Zinc oxide can be termed a multifunctional material due to its unique physical and chemical properties [(19)] like high chemical stability, broad range of radiation absorption high electrochemical coupling coefficient and high photo-stability, is a multifunctional substance [(20,21)].

Properties of ZnO nanoparticles (ZnO NPs) depend on their preparation method. It's found to be various techniques have been used for preparation of ZnO NPs such as hydrothermal [(22),] laser ablation [(23)], radiolytic synthesis [(24)], sol-gel [(25),] and electrochemical methods [(26)]. The purpose of this paper was to preparation of ZnO NPs by electrochemical method in presence of Polyvinylpyrrolidone (PVP) as stabilizer and $NaNO_3$ as electrolyte and study the characteristics of prepared ZnO NPs such as SEM , TEM , EDX, and AFM.

Experimental Section

The electrochemical system for preparation of zinc oxide nanopowders (ZnO NPs) is shown in schematic Figure (1). A 250 ml-glass beaker, pure zinc plate as positive electrode (anode), graphite plate as negative electrode

(cathode), DC-regulated power supply (30 volt-as a maximum voltage and 5000 milli-ampere as a maximum current, UTP3345TD-China) was used for measuring and providing, stirrer plate and magnetic stirrer bar for stirring the electrolytic cell solution.

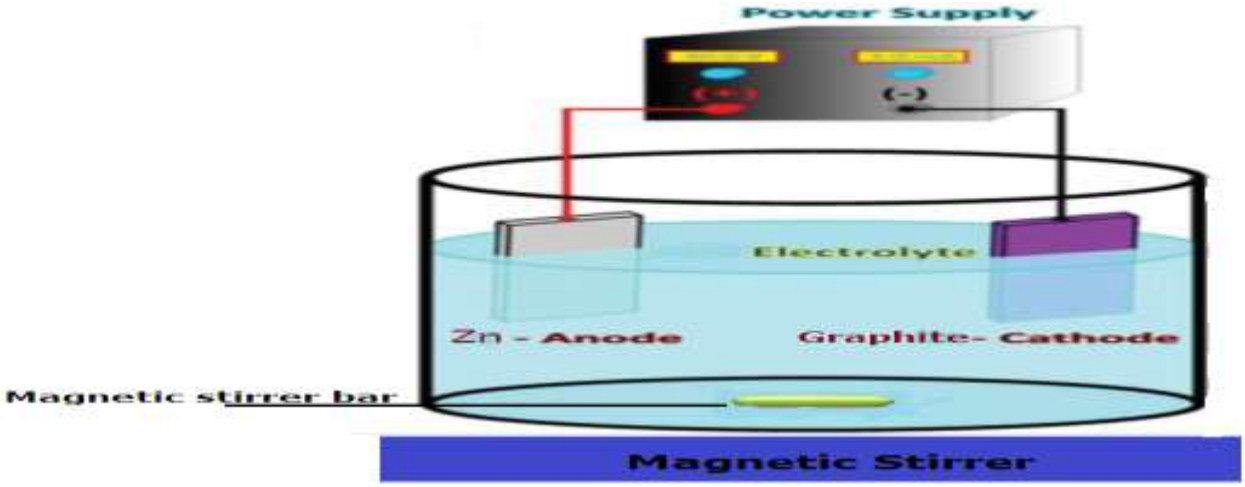

Fig.1; the electrochemical system for preparation of ZnO nanoparticles in PVP and $NaNO_3$

Prior of electrolysis, both cathode and anode were rinsed by acetone and then deionized water to removing any organic materials from the surfaces of the two electrodes. In typical electrochemical method; the electrochemical cell was be bored with 200 ml solution contain of (4 ml of 10 g/100 ml $NaNO_3$as electrolyte, 10 ml of 1 g/100 ml Polyvinylpyrrolidone (PVP) as stabilizer, and deionized water).

A pure zinc plate (1 cm x 4 cm) and inert graphite electrode (1.5cm x 5 cm) were as anode and cathode respectively, being vertically and placed face toward face in electrochemical cell solution with 4cm apart. The electrolysis reaction was carried out in an undivided electrolytic cell for one hour with stirrer at room temperature. The range of voltage (20-30) volt and current density (100 mA/cm^2). After about (10) minutes of beginning of electrolysis, milky suspended solution is obtained of ZnO nanoparticles.

The pH of solution rises during of electrolysis until reaches to a maximum value of 11.4 and remains constant to end of electrolysis. The zinc electrode is used once of each experiment. The produced white precipitates of ZnO nanoparticles were centrifuged, washing with deionized water and ethanol for several times and then dried at 60 ℃ for 60 minutes.

Result and Discussion

EDX- Spectroscopy

The purity and stoichiometry of electrochemical prepared ZnO NPs was examined by energy dispersive X-ray spectroscopy (EDX) as shown in figure (2) which is elucidates presence specific signals of zinc and oxygen only,suggesting that the high purity of ZnO NPs sample. The weight percentages of Zn and O were 73.92 and 26.08% respectively, and the atomic percentages were 40.91 % and 59.09 % of Zn and O respectively.

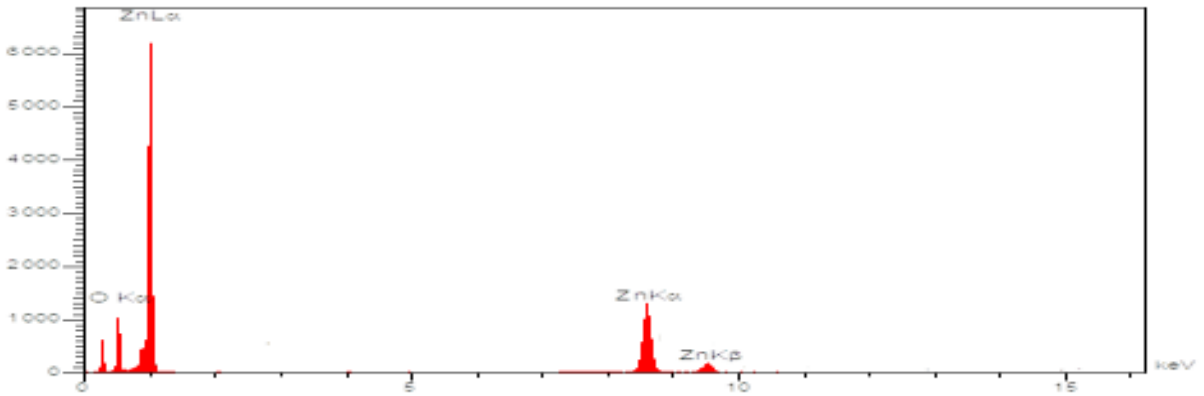

Figure 2: EDX-spectrum of electrochemical prepared ZnO- NPs in presence of PVP as stabilizer and $NaNO_3$ as electrolyte

Scanning Electron spectroscopy

The morphology of electrochemical synthesized ZnO nanoparticles in presence of ($NaNO_3$and PVP) was investigated by scanning electron spectroscope (SEM) as shown in figure (3) ; the SEM images reveals in almost a homogeneous particles distribution and having sheets like shape.

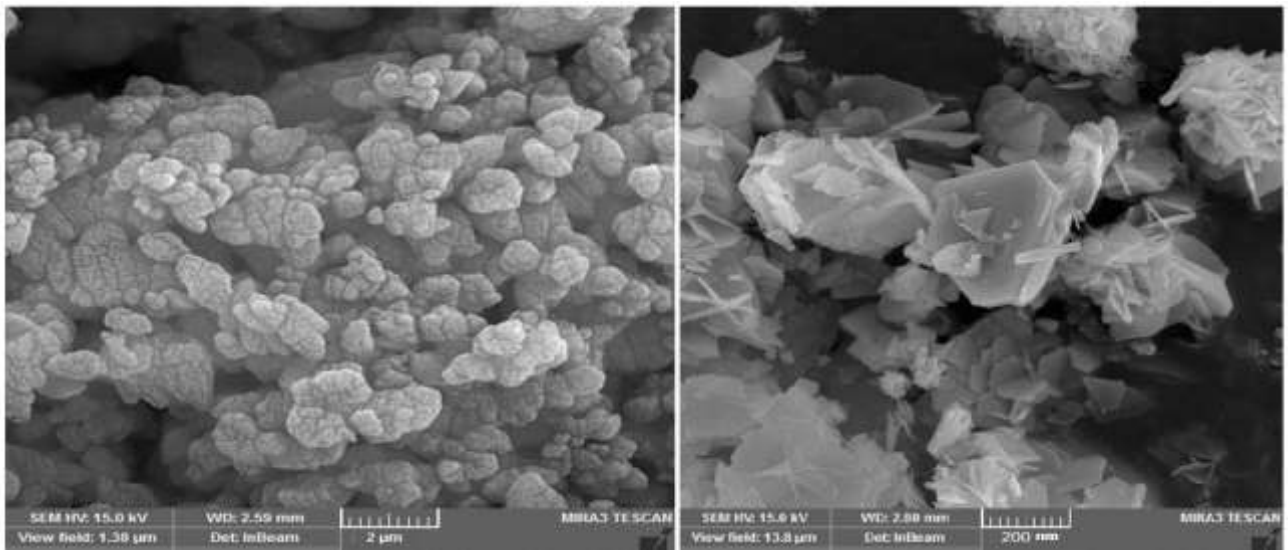

Figure 3: SEM micrograph of electrochemical prepared ZnO- NPs in presence ofPVPas stabilizer and $NaNO_3$as electrolyte

Transmission electron spectroscopy

The morphology and size of ZnO NPs which is electrochemically synthesized in (15 ml of 10 g/100 ml $NaNO_3$, 10 ml of 1 g/100 mlPVP) was assayed by transmission electron spectroscopy (TEM) micrograph of ZnO NPs as shown in Figure (4); which elucidates the TEM micrograph of ZnO NPs .The nanoparticles exhibited a bipyramid like shape, having a sizes in the range of (55-65) nm.

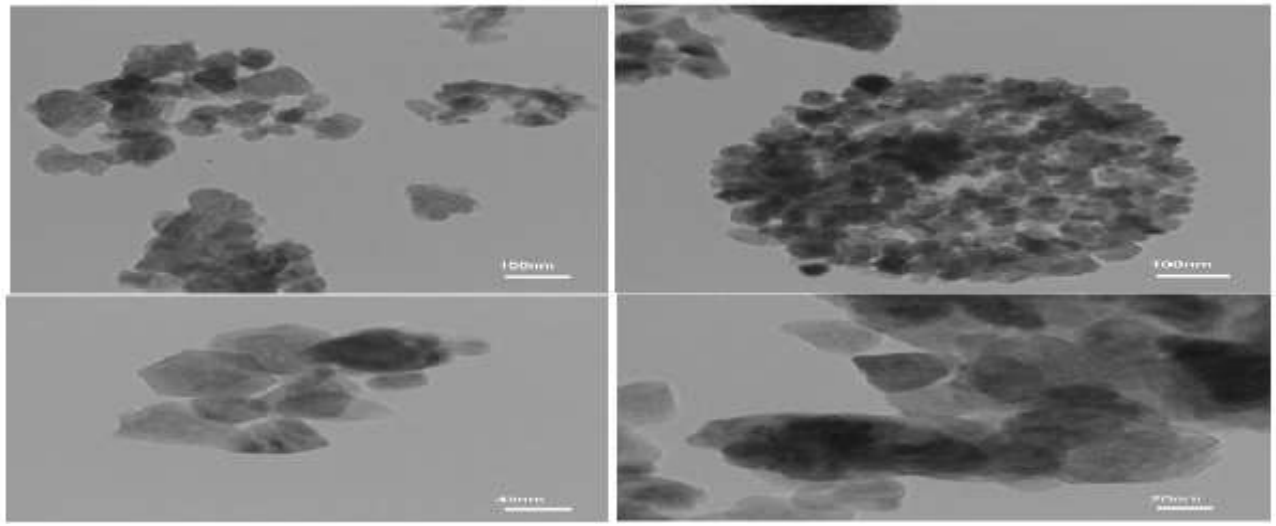

Figure 4: TEM micrograph of electrochemical prepared ZnO- NPs in presence of PVP(1g/100ml) as stabilizer and $NaNO_3$(10 g/100 ml) as electrolyte

Atomic force microscopy analysis

Atomic force microscope (AFM) topography imaging is a useful tool to getting information about morphology, topography, and textile of several surfaces. In the AFM image of ZnO NPs (Fig (5)); the opaque colors are the faint structures while the bright colors are the soaring structures due to various directions of ZnO grains. The 3-D AFM image (Fig (5 a)) illustrates the formation of homogeneously distribution of ZnO NPs and no agglomerated was seen. The obtained information from granularity cumulation distribution (GCD) about the distribution of particles sizein Figure (6) which shows the range of particles diameters of ZnO NPs between (45-115) nm , and the average diameter is found to be (60.99) nm.

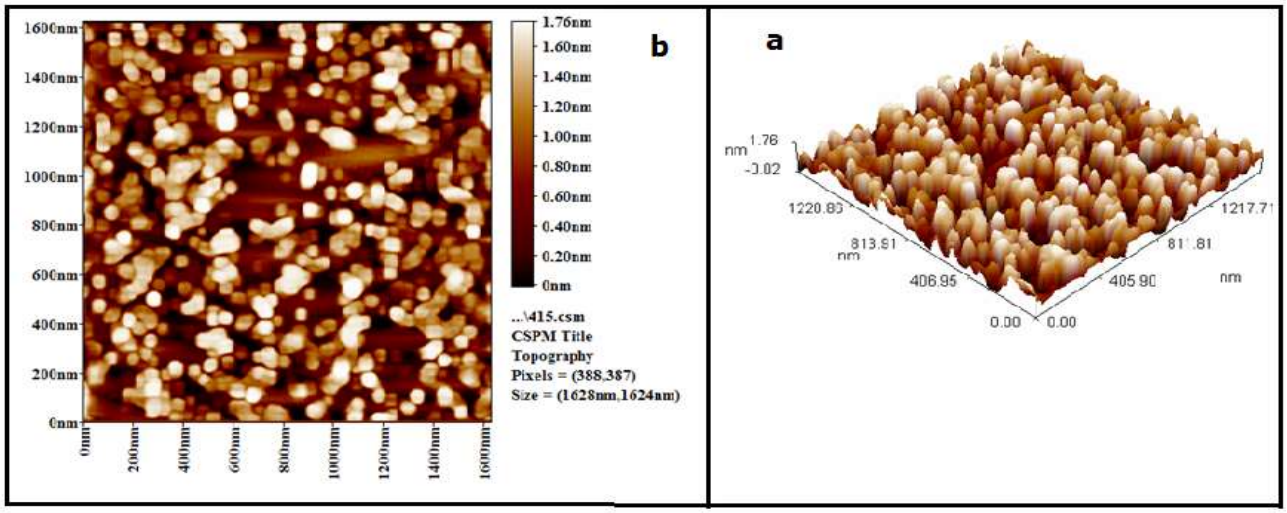

Figure 5: AFM images of electrochemical prepared ZnO- NPs in presence of PVPas stabilizer and $NaNO_3$as electrolyte

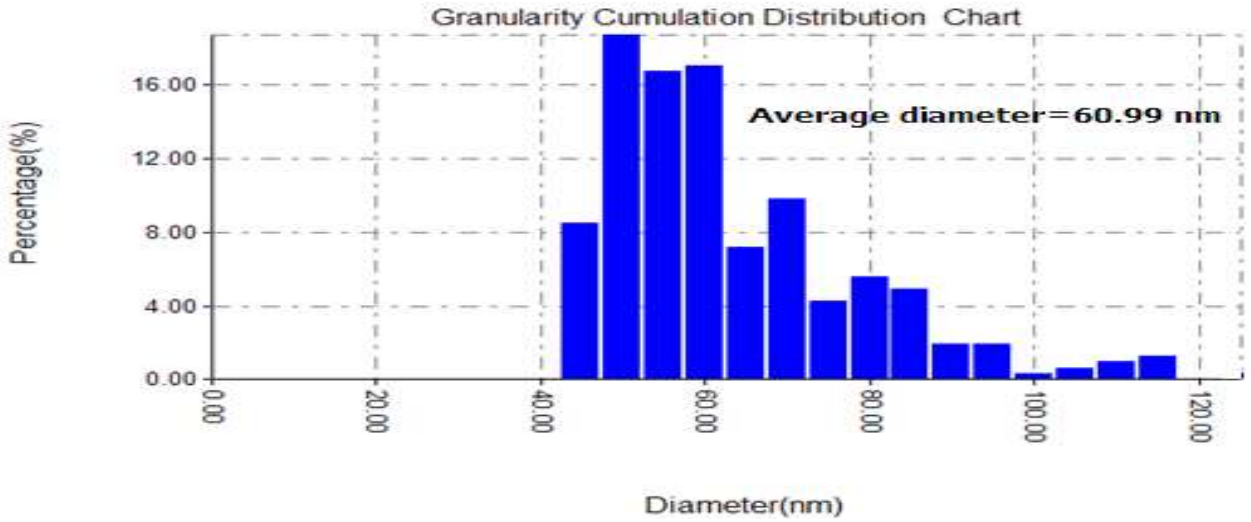

Figure 6: GCD-chart of electrochemical prepared ZnO- NPs in presence of PVPas stabilizer and $NaNO_3$as electrolyte

The other topographic information data were abridged in Table (1).

Table 1: the topographic information data which were produced from AFM

CSPM Imager surface roughness analysis	
Roughness average (Sa) nm	**0.445**
Root mean square (Sq) nm	**0.514**
Surface skewness (Ssk) nm	**0.000132**
Surface kurtosis (Sku)	**1.8**
Peak-peak (Sy) nm	**1.78**
Ten point height (Sz) nm	**1.77**
Mean summit curvature (Ssc) nm^{-1}	**-0.00248**
Root mean square slope(Sdq) nm^{-1}	**0.0393**
Surface area ratio (Sdr)	**0.0767**
Surface bearing index (Sbi)	**5.58**
Core fluid retention index (Sci)	**1.49**
Valley fluid retention index (Svi)	**0.0692**
Reduced submmit height (Spk) nm	**0.19**
Core roughness depth (Sk) nm	**1.55**
Reduced valley depth (Svk) nm	**0.0.0452**

Conclusion

It has been proposed successfully electrochemical preparation of ZnO- NPs including dissolution of zinc electrode in $NaNO_3$ as electrolyte and PVP as stabilizer of nanoparticles. This is an easy, economic, and shortened time method. The average size of ZnO- NPs was equalled to 60.99 nm which is obtained from GCD and about 60 nm which is produced from TEM micrographs. According to TEM micrographs the nanoparticles having a bipyramid like shape.

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