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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF SELENIUM DOPED ZINC OXIDE (ZNO-SE) NANOPARTICLES

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ABSTRACT

In this study ZnO nanoparticles were synthesized by using different procedures. In the first procedure ZnO prepared by using sol-gel method by precipitating ZnO from a reaction of Zinc nitrate ($Zn(NO_3)_2$) and Sodium hydroxide (NaOH) different techniques are used sol-gel method with stirring and centrifugation to obtain ZnO nanoparticles. In the second procedure grinding of $Zn(CH_3COO)_2$ and $H_2C_2O_4.2H_2O$ in a mortar for 30 min is used, also by doping Selenium powder (5%) to the mixture and ZnO-doped Selenium was obtained. ZnO nanoparticles were characterized by X-ray (Rajendra *et al.,* 2009) diffraction (XRD) where diameter ranged between 16-33 nm by using Scherrer equation. d-space (d) was calculate theoretically by using Bragg's law and practically by the formula. Some lattice also were calculated and showed in the tables like: micro strain (ε), potential parameter (μ), bond length (L), unit cell volume (V) and stress (σ).

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INTRODUCTION

Nanotechnology deals with using objects in the nanometer scale (10^{-9}) size range to develop products with possible practical application. It is usually based on nanoscience insights. "Nanotechnology is concerned with materials and systems whose structures and components exhibit novel and significantly improved physical, chemical, and biological properties, phenomena, and processes due to their nanoscale size. The goal is to exploit these properties by gaining control of structures and devices at atomic, molecular and supra molecular levels and to learn to efficiently manufacture and use these devices (Filipponi *et al.,* 2010). Nanotechnology literally means any technology on a nanoscale that has applications in the real world (Bainbridge, 2004). Nanotechnology encompasses the production and application of physical, chemical, and biological systems at scales ranging from individual atoms or molecules to submicron dimensions, as well as the integration of the resulting nanostructures into larger systems.

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Nanotechnology is likely to have a profound impact on our economy and society in the early 21st century, comparable to that of semiconductor technology, information technology, or cellular and molecular biology. Science and technology research in nanotechnology promises breakthrough in areas such as materials and manufacturing, nanoelectronics, medicine and healthcare, energy, biotechnology, information technology, and national security. It is widely felt that nanotechnology will be the next Industrial Revolution (Bhushan, 2010) (Fang and Wu, 2013). Zinc Oxide (ZnO) nanostructure have been receiving remarkable attention because of their unique properties and vast application for nano devices and nano systems (Wang *et al.,* 2013, Ahsanulhaq *et al.,* 2007, Reddy *et al.,* 2007a, Reddy *et al.,* 2007b, Law *et al.,* 2005, Könenkamp *et al.,* 2005, Kim and Osterloh, 2005). Various approaches have been employed to grow verity of ZnO nanostructures (Ahsanulhaq *et al.,* 2008, Umar *et al.,* 2006b, Umar *et al.,* 2006a, Umar *et al.,* 2005c, Umar *et al.,* 2005b, Sekar *et al.,* 2005, Umar *et al.,* 2005a, Umar and Hahn, 2006, Cheng and Samulski, 2004). ZnO nano structures with flower-like morphology have been synthesized by solution methods such as hydrothermal, and solvothermal methods at relatively higher temperature of 160_200°C (Zhang *et al.,* 2004, Zhang *et al.,* 2005a, Zhang *et al.,* 2005b, Feng *et*

al., 2005). Zhang *et al*. reported flower-like ZnO nanostructure using hydrothermal method at high temperature of 180^{o}C (Zhang *et al.,* 2002). Recently Jiang *et al*. also reported morphologically controlled ZnO flower-like structure by hydrothermal methods at 180^{o}C (Jiang *et al.,* 2007). However, hydrothermal and solvothermal methods required a rather high-growth temperature, long reaction time, surfactants and additives for tailoring the shape and size of ZnO nanostructures. A wide variety of organic pollutants especially pesticides are introduced into the water system from various sources such as effluents, agricultural runoff and chemical spills (Cohen *et al.,* 1986, Rahman *et al.,* 2006). Their toxicity, stability to natural decomposition and persistence in the environment have been the cause of much concern to the societies and regulation authorities around the world (Qamar and Muneer, 2009). The control of organic pollutants in water is an important measure in environmental protection. Among many processes proposed and /or being developed for the destruction of the organic contaminants, biodegradation has received the greatest attention. However, many organic chemical, especially those that are toxic or refractory, are not amenable to microbial degradation. Recently considerable attention has been focused on the use of a semiconductor as a means to oxidize toxic organic chemicals (Blake, 2001, Herrmann, 1999, Litter, 1999, Vidal *et al.,* 1999, Alfano *et al.,* 2000, Fujishima *et al.,* 2000, Macounova *et al.,* 2001, Topalov *et al.,* 2001, Li *et al.,* 2001).

Synthesis of noble metal nanoparticles for applications such as catalysis, electronics, textiles, environmental protection, and biotechnology is an area of constant interest. Recently, an awareness of general sanitation, contact disease transmission, and personal protection has led to the development of antimicrobial textiles. The development of antimicrobial using Zinc Oxide nanoparticles has been investigated in various works. The ZnO nanoparticles were prepared by wet chemical method in this work (Rajendra *et al.,* 2010, Rajendra, 2009 #552, Basheer et al.)

Objectives

- Synthesis of Zinc Oxide (ZnO) nanoparticles by bottom-up process and then Selenium Doped.
- Characterization of the synthesized Zinc Oxide nanoparticles using XRD.
- Propose further use of Zinc Oxide in redox reaction

MATERIALS AND METHODS

Zinc nitrate $(Zn(NO_3)_2,6H_2O)_2.(H_2O)_2$, Oxalic acid dehydrate, Selenium (Se) metal powder (99.9%), Sodium hydroxide (NaOH), Zinc acetate and Ethanol. All precursors used in this study were of analytical grade and the solvents were of laboratory grade, which were used without further purification.

Synthesis of ZnO Nanoparticles by Sol-gel Method: ZnO nanoparticles were synthesized by sol-gel method using Zinc nitrate and NAOH as precursors. In this work, the aqueous solution (0.2M) of Zinc |nitrate ($Zn(NO_3)_2.6H_2O$) of NaOH were prepared with deionized water. The Zinc nitrate weighed around 2.9749g and it is transferred into a beaker and exactly 1.22 g of NaOH pellets were taken another beaker measure 50 ml of deionized water and pour in to respected beakers. The NaOH solution is taken in the beaker and slowly added into the Zinc nitrate solution which is taken in another beaker and it is kept on magnetic stirrer which helps in the proper mixing of both the solution at room temperature which resulted in the formation of a white precipitation. The white product was centrifuged at 5000 rpm for 30 min and washed three times with distilled water. Then it is filtered by Whatman's filter paper. This filter paper was removed after filtration and kept for drying in hot oven at the temperature of 60 - 80°C for 3 hours. The calcinations were carried out for the obtained product at 400°C in the presence of atmosphere air for 4 hr (Dutta *et al.,* 2012). The reaction between Zinc nitrate and Sodium hydroxide for the synthesis of Zinc oxide nanoparticle can be represented as:

$$Zn(NO_3)_2+2NaOH \rightarrow ZnO +2NaNO_3+H_2O$$

Synthesis of Selenium doped pristine ZnO NPs: To synthesise the pristine ZnO NPs, 5.48g of Zinc acetate dehydrate and 3.78 g of Oxalic acid were mixed and ground in a mortar at room temperature for 30 min. the smell of acetic acid ceased indicating the completion of the reaction to form hydrated ZnC_2O_4. The thermo gravimetric analysis of the synthesized ZnC_2O_4 revealed mass loss at about 400°C sample(4) in Table (1), suggesting formation of ZnO NPs and agreed well with the reported literature (Shyni *et al.,* 2016). Consequently, the ZnO NPs were prepared by heating the synthesized hydrated ZnC_2O_4 in a quartz crucible at 450°C for 30 min in a temperature controlled muffle furnace. Similarly batches of Se doped ZnO NPs (10%) were synthesized by mechanochemical method after adding required amount of selenium metal powder with the Zinc acetate and oxalic acid precursors. The synthesized NPs were washed with ethanol and kept overnight for drying at 60^{o}C.sample (5) see Table (1).

Preparation of Selenium Doped Zinc Oxide Nanocomposite: The synthesis of ZnO-Se has been carried out using Mory autoclaves provided with the Teflon. The condition selected for the synthesis of ZnO – Se particles are T = 150°C temperature. ZnO nanoparticles prepared by the sol gel method were taken and around O.2 g of this ZnO is weighed and taken in the Teflon liners. Around 0.2 g Selenium is weighed and taken in the same Teflon lines. Then exactly measure 15 ml of Ethanol and it is poured into the seam Teflon liners. Stir the content for 10 minutes. Then these Teflon liners are placed in a Moray autoclave. Then the autoclaves were placed in the oven provided with a temperature controller, the temperature of the oven was raised up to a predetermined temperature for a desired period (5 hours). After 5 hours the autoclave is cooled to the room temperature. The contents of the liners are thoroughly washed in ethanol repeatedly and later drive in an oven at 60^{o}c for 3 hours. These dried ZnO–Se nanoparticles were characterized by XRD. (Shyni *et al.,* 2016).

RESULTS AND DISCUSSION

Typical XRD patterns of ZnO nanoparticles which are shown in Fig(1). The different peaks at angles 2Θ of 32.08°, 34.70°, 36.60°, 48.00°, 56.80°, 36.00°, 66.70°, 68.20° and 69.40° correspond to the respective reflections from the (100), (002), (101), (102), (110), (103), (200), (112) and (201) crystal planes of the hexagonal Wurtzite Zinc Oxide structure (JCPDS 36-1451) can be seen at the spectrum of ZnO. The XRD for the different experiments are shown in Table (1). The lattice parameters are obtained using the formulae (Zak and Abd, 2011).

Table 1. The XRD for the different experiments

Planes (*hkl*)	1	2	3	4	5
			2Θ°		
100	32.08	32.00	32.10	31.94	31.96
002	34.70	34.60	34.60	34.62	34.60
101	36.60	36.50	36.52	36.44	36.42
102	48.00	47.80	47.70	47.76	47.82
110	56.80	56.80	56.80	56.86	56.80
103	63.00	36.00	63.00	64.08	63.08
200	66.70	66.70	66.60	66.68	66.64
112	68.20	68.20	68.24	68.18	68.18
204	69.40	69.30	69.34	69.22	69.18

Table 2. The lattice constant value for the different samples

Sample	2Θ°	$\beta_{(FWHM)}$	D (nm)	a	c	c/a
1	36.6	0.2526	33.125	3.2191	5.1662	1.6049
2	36.4849	0.5366	15.588	3.2172	5.1807	1.6103
3	36.4849	0.5366	15.588	3.2175	5.1807	1.6015
4	36.4840	0.2782	30.067	3.2328	5.1807	1.6025
5	36.4092	0.3535	23.657	3.2309	5.1807	1.6035

Table 3. The lattice constant value for the different samples

Sample	ε Strain	™ $(nm)^{2-}$	d (Bragg) (A°)	d (formula) (A°)	Reduction %
1	$3.331x10^{-3}$	$9.1136x10^{-4}$	2.4532	2.4529	0.5
2	$7.101x10^{-3}$	$4.1154x10^{-3}$	2.4597	2.4537	0.4993
3	$7.101x10^{-3}$	$4.1154x10^{-3}$	2.4584	2.4537	0.4995
4	$3.679x10^{-3}$	$1.1061x10^{-3}$	2.4636	2.4626	0.4999
5	$4.689x10^{-3}$	$1.8005x10^{-3}$	2.4650	2.4616	0.4997

Table 4. The lattice constant value for the different samples

Sample	L(A°)	μ	V (A°)3	σ (Gpa)
1	1.9602	0.3794	46.362	-1.7857
2	1.9612	0.3785	46.437	-1.1368
3	1.9612	0.3786	46.445	-1.1368
4	1.9676	0.3798	46.888	-1.1368
5	1.9669	0.3796	46.833	-1.1368

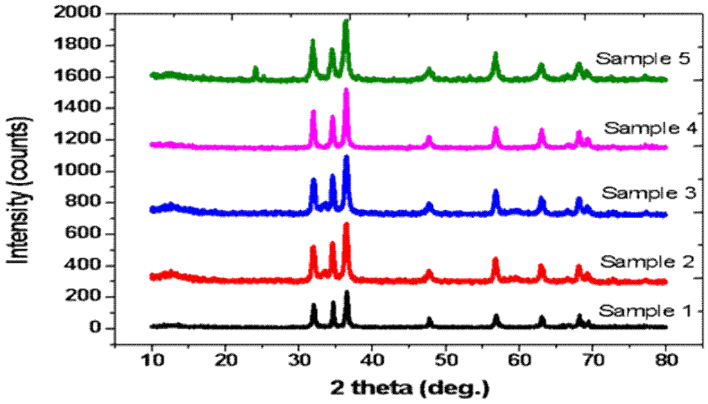

Fig. 1. The different peaks at angles 2Θ for the different samples

$a = \frac{\lambda}{\sqrt{3}sinsin\,\theta 101}$ and $c = \frac{\lambda}{sinsin\,\theta 002}$

The crystal sizes (D) of the ZnO nanoparticles were calculated using Scherrer equation (Patterson, 1939)

$$D = \frac{k\lambda}{\beta_{hkl}coscos\,\theta}$$

where λ is the wavelength of the incident X-ray (1.5406 A°) for Cu Kα, k is a constant equal to 0.90 (Damonte *et al.,* 2004), β_{hkl} is the peak width at half-maximum intensity, and θ is the peak position.

The (101) plane (the strongest) was selected to calculate the crystal size as can be seen in Table (2).The lattice parameters are shown in table (2), the lattice parameter a and c have determined for all samples. The values for the lattice parameters a and c are identical to those reported (Damonte *et al.,* 2004), for ZnO nanoparticles prepared by standard methods. The lattice parameter obtained in this work highly related to those previously reported (Saleem *et al.,* 2012). The d-spacing was calculated by both methods using the Bragg's law:

$$d = \frac{\lambda}{2sin\,sin\,\theta}$$

as well as the formula:

$$\frac{1}{d^2_{(hkl)}} = \frac{4}{3}\left(\frac{h^2+hk+k^2}{a^2}\right) + \frac{l^2}{c^2}$$

that relates it to the lattice constants. The values obtained were almost similar Table (3). The d_{hkl} for the different samples calculated by theoretical methods

The micro-strain is given by the formula,

$\varepsilon = \frac{\beta}{4tantan\,\theta}$ and dislocation density (↗), that indicate the amount of defects is defined as the length of dislocation line per unit volume of crystal, is calculated via relation $= \frac{1}{D^2} = \frac{1}{D^2}$ (Barrett and Massalski, 1980). The Zn ⟶O bond length L is given by (Seetawan *et al.,* 2011), $L = \sqrt{\left(\frac{a^2}{3}\right) + \left(\frac{1}{2} - \mu\right)^2 c^2}$ here μ is the measure of an atom displacement to the neighboring one along the "c" axis. The value of μ is calculated by the equation, $\mu = \frac{a^2}{3c^2} + 0.25$. The unit cell volume is calculated as follows $V = 0.866a^2\,a$ (Srinivasan *et al.,* 2007). The stress σ (GPa) in the crystallite's planes can be determined using the formula, $\sigma = -233\left(\frac{c_{bulk}-c}{c_{bulk}}\right)$, where c is the lattice parameter crystallite's planes calculated from XRD analysis, c_{bulk} is the strain-free lattice parameter of ZnO (5.2061A°). That calculations are summarized in table (4). The parameter is changed with c/a ratio in away to maintain the tetrahedral dimension due to the angle distortion. From table (3), it can be seen that μ differ with c/a ratio. On the other hand the L value obtained is almost equal to the 1.9767A° value reported by Seetawan et al. (Seetawan *et al.,* 2011).

Conclusion

In the present study, different chemical methods were used to synthesis Zinc Oxide(ZnO) nanoparticles (NPs) and Selenium were also used to dope the Zinc Oxide (ZnO-Se) at nanoscale ere it has been shown that control is possible by bottom-up process. ZnO and ZnO-Se nanoparticles were characterized using X-ray diffraction (XRD), whe are the average crystallite size of obtained ZnO nanoparticles range is 16-33 nm. The average c/a ratio was consistent with the values for the hexagonal wurtzite structure of ZnO demonstrating that the composite still retain the ZnO structure. D-space (d) was calculated theoretically by using Bragg's law and practically by the formula and there was no difference existed and as supported byanalysis. Some lattice also were calculated micro strain (ε), potential parameter (μ), bond length (L), unit cell volume (V) and stress (σ), these parameters result that highly stable nanoparticles were synthesized. Some reduction in crystal size of ZnO were shown when doped with Selenium.

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