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Preparation and Characterisation of ZnO Nanoparticles

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ABSTRACT

In this work, spherically shaped ZnO nanoparticles of size around 100 nm have been synthesized by sol gel process using zinc acetate dehydrate and sodium hydroxide as starting materials at room temperature. Surface morphology and microstructural characterization of the particles are carried out using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS), particle size analysis by dynamic light scattering (DLS), grain size and crystal structure using X-ray diffractometer (XRD) and Fourier transform infrared spectroscopy (FTIR). The X-ray diffraction results indicated that the synthesized ZnO particles are of single phase and the average particle sizes were about 100 nm. The synthesized nanoparticles could be applied in the treatment of industrial waste water.

1. Introduction

Nanotechnology deals with the preparation, design, and characterization of materials and devices on the nanoscale**.** Nanotechnology is an emerging field, which plays an important role in the development of innovative technology to produce new products with improved performance and utilize less energy and reduce harm to the environment [1]. ZnO is an environmental friendly metal oxide that could be suitable for industrial, technical and medical applications [2]. Many researchers have synthesized ZnO nanostructures using different techniques. Synthesis of ZnO nanostructures using Low-temperature aqueous chemical growth method was found to be superior method, due to its advantages such as low cost, low temperature, non-toxic operation and environmental friendliness [3]. Other methods of preparation include micro emulsion hydrothermal synthesis [4] direct deposition in aqueous solution [5] surfactant assisted hydrothermal orientation growth [6] alcohol solution refluxing [7] and simple chemical sol-gel process [8]. Recent research work on ZnO synthesis finds applications in lasers and optical amplifiers based on electronic transitions [9].

The piezo electric properties of ZnO finds applications as a sensor, converter, energy generator and photo catalyst in hydrogen production [10, 11] and also applied in the ceramic industry due to its superior qualities such as hardness, rigidity and piezoelectric constant, while its low toxicity, biocompatibility and biodegradability make it a material of interest for biomedicine and in pro-ecological systems [12-19].

The present study focusses on the preparation of nano sized ZnO using environmental friendly sol-gel method. Zinc acetate dehydrate and sodium hydroxide are used as starting materials. The prepared nanoparticles could be employed as a cost effective photo catalyst for the treatment of waste water from textile industry. The research work was carried out in the sultanate of Oman during April 2016.

2. Experimental Methods

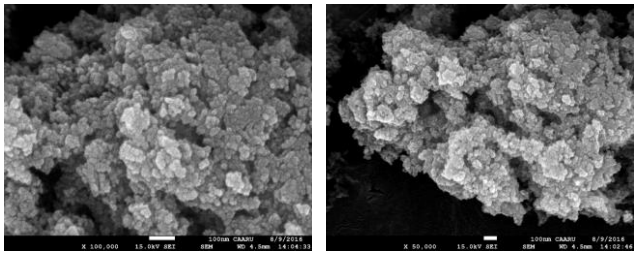
The precursors employed for the preparation of ZnO nanoparticles are zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$] and sodium hydroxide (NaOH). The synthesis methodology used was sol-gel process and the experiment was carried out at room temperature. Equal volumes of zinc acetate dehydrate (0.05 M) and sodium hydroxides (0.1 M) are allowed to react for 24 hours under continuous stirring and the resulting mixture was centrifuged at 3000 RPM for 30 minutes. The resulting particles are washed three times with ethanol to remove the unreacted materials attached to the surface of ZnO nanoparticles. The washed particles are calcined at 400 °C for 2 hours in a drying oven to produce pure ZnO. The calcined ZnO particles are powdered using a mortar and a pestle to obtain the desired size range.

The synthesized nano particles are characterized in terms of surface morphology, micro structures and particle size using SEM JEOL JSM-7600F (Field Emission Scanning Electron Microscope) and X-ray (X-ray diffraction (XRD) instrument) Rigaku, and Mini Flex 600 DLS (Dual Scattering Particle Size Analysis) CILAS Nano DS and FTIR (Fourier transform infrared spectroscopy) Frontier is Perkin Elmer's. All characterizations were carried out at room temperature.

3. Results and Discussion

3.1 SEM Characterization

The surface morphology of the particles are examined using SEM and the micrographic images of the ZnO nanoparticles are shown in Fig. 1a and b. It is observed from the morphology of the ZnO nanoparticles that all particles are distributed uniformly. Furthermore, these particles are much more uniform in size and shape compared with the products of conventional method. The average crystal size was found to be 100±4 nm by selecting few numbers of crystals, which are in agreement with DLS measurement.



(a) (b)

Fig. 1 SEM Micrograph of pure ZnO nanoparticles a) at high magnification (100000 X) and at 15 kV and b) at a magnification of 50000 X and at 15 kV

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The elemental compositions of the ZnO nanoparticles prepared by sol-gel method were determined by Energy dispersive X-ray diffraction (EDX INCA Penta FETx3 OXFORD Instruments) as shown in Fig. 2 indicating the high purity of ZnO nanoparticles. The elemental analysis of the prepared ZnO nano particles are summarized in Table 1.

Table 1 Elemental composition of ZnO nanoparticles

Element	Weight%
Zn	61.5
O	21.8
C	12.9
Al	3.7

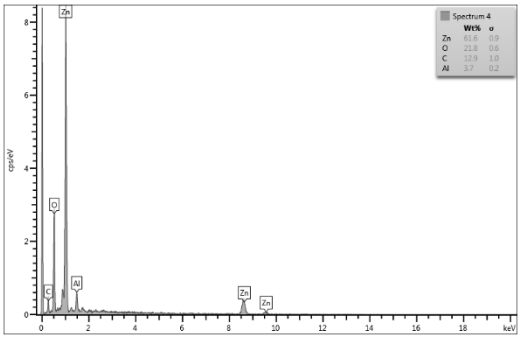

Fig. 2 EDX spectra and elemental analysis of ZnO nanoparticles

3.2 XRD Analysis

The phase identification and formation of ZnO nanoparticles are confirmed using XRD analysis and the diffraction data were recorded by using Cu Kα radiation. The intensity data were collected over a 2θ range of 10-90°. Fig. 3 shows the powder X-ray diffraction patterns, crystalline structure and broadening of the characteristic line of the ZnO nanoparticles. The diffraction patterns and inter plane spacing are well matched to the standard diffraction pattern of wurtzite ZnO, demonstrating the formation of ZnO Nano crystals. No characteristic peaks were observed other than ZnO, which indicates the confirmation of high purity ZnO nanoparticles. Diffraction peaks related to the impurities were not observed in the XRD pattern.

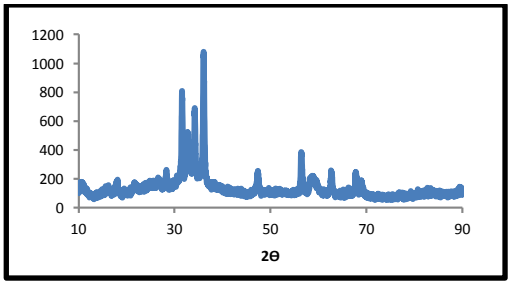

Fig. 3 X-ray diffraction spectra of ZnO nano particles

XRD pattern designates the formation of hexagonal wurtzite phase of ZnO which is compatible with the electron diffraction results. The peak broadening in the XRD pattern clearly shows that small nano crystals are present in the samples and no evidence of bulk remnant materials and impurity. Nine peaks appear at different 2θ values as shown in the Fig. 3.

As ZnO crystallizes in the wurtzite structure in which the oxygen atoms are arranged in a hexagonal close packed type with zinc atoms occupying half the tetrahedral sites. Zn and O atoms are tetrahedrally coordinated to each other and have, therefore, an equivalent position. The zinc structure is open with all the octahedral and half the tetrahedral sites empty. Values for some major XRD peaks are summarized in Table 1.

Table 1 Interplanar spacing (d_{hkl}) from XRD, JCPDS data card for corresponding hkl planes, percentage of variation of *d*, and FWHM

(d_{hkl})	d_{XRD} (Å)	d_{JCPDS} (Å)	% of contraction in d	FWHM (Degree)
⟨100⟩	2.7964	2.8135	0.6078	0.295
⟨002⟩	2.5871	2.6027	0.5994	0.289
⟨101⟩	2.4624	2.4751	0.5131	0.318
⟨102⟩	1.9030	1.9106	0.3978	0.372
⟨110⟩	1.6196	1.6244	0.2955	0.402
⟨103⟩	1.4727	1.4769	0.2844	0.447

3.3 DLS Analysis

Dynamic light scattering is a widely used technique for the determination of particle size in colloidal solution. The particle size distribution of ZnO nanoparticles in Fig. 4 indicates that the average size of particles is about 100 nm which indicates moderate distribution of the nanoparticles. A single peak in the Fig. 4 designates the particles are of uniform size distribution, which is one of the desired qualities for photo catalysis application.

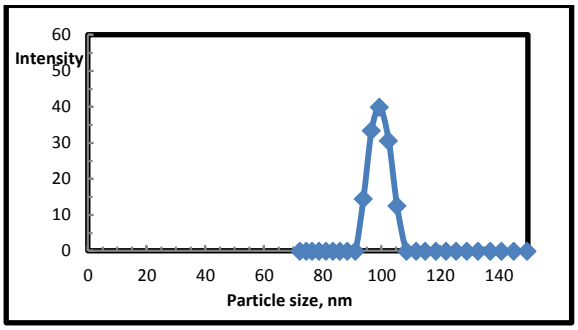

Fig. 4 Particle size analysis using DLS

3.4 FTIR Analysis

The purity and nature of the ZnO nanoparticles are carried out using Infrared studies. The FTIR spectrum of the pure and ZnO, synthesized by sol-gel method, which was acquired in the range of 500 - 4000 cm^{-1} as indicated in the Fig. 5. The peaks observed at 3478 cm^{-1} and 889 cm^{-1} are due to O-H stretching and deformation, respectively assigned to the water adsorption on the metal surface. The peaks at 1516 cm^{-1} and 523 cm^{-1} are corresponding to ZnO stretching and deformation vibration, respectively. The absorption at 3769 cm^{-1} is attributed to stretching vibration of OH groups.

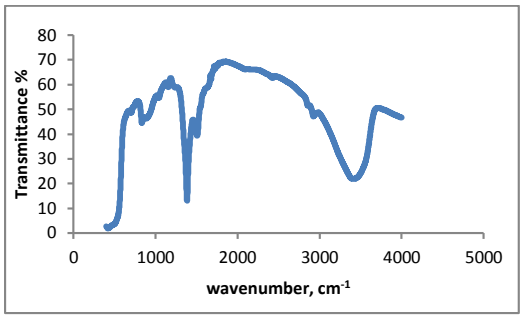

Fig. 5 FTIR spectra of ZnO nanoparticles

4. Conclusion

ZnO nanoparticles of size around 100 nm have been successfully fabricated by sol-gel method using zinc acetate dihydrate and sodium hydroxide as precursors. The surface morphology of the resulting nanoparticles was characterized using SEM, crystallinity using XRD, particle size analysis using dynamic light scattering, and bond stretching by FTIR analysis. The synthesized ZnO nanoparticles were found to be of uniform size and without any aggregation. The same particles could be employed in the treatment of waste water from textile industries. Currently the research team is investigating the possibility of using surface modified ZnO nanoparticles in the textile industry waste water treatment.

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