



## Green biosynthesis of zinc oxide nanoparticles *via* aqueous extract of cottonseed

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### Abstract

Green synthesis of nano-particles by using of aqueous extract of *cottonseed* as antioxidant materials is an efficient method along with advantages such as economically, simplicity of work-up process and eco-friendly approaches. Also, this study was focused on synthesis of ZnO nanoparticles (ZnO-NPs) with using an aqueous extract of *cottonseed*. The synthesized ZnO-NPs were characterized by using XRD, EDX, SEM, FTIR and UV-VIS spectroscopy. By the collected results, especially from Fourier transform infrared spectroscopy (FTIR) data was concluded the presence of heterocyclic, aromatic and phenolic compounds in water extract of plant are acts as antioxidants for nano-particle synthesis. In other side, the SEM pattern analysis showed that ZnO-NPs have a crystalline wurtzite phase. Finally the XRD spectrum identified the average particle size of ZnO NPs about 18.97 nm.

## 1. Introduction

In recent years employing of nano-science has been considered as multi applied technology. In the field of nano-science, the particles with size of 1-100 nm, at least one side of their dimensions is called nanoparticles class [1,2]. Furthermore nano has been discovered on the planet from million years ago, but it has been highlighted by the researchers at during of these years. The range of sizes is different but usually the particles under 100 nm is aimed, because in this range of size, essentially many different properties of the material could be appeared [3]. By morphology deforming of particle size from micro to nano, some important physical properties as alteration are mentionable: one is increasing the ratio of surface area to volume and other side entering particle size into the domain of quantum effects [4]. By increasing the surface to volume ratio, which step by step is arised by decreasing the size of the particle, can be affected the behavior of the atoms in the outside from surface of the particle to compare the internal atoms activation. This affects on the size and some physical and chemical properties of nano particles [4, 5]. Also, there are many materials that have properties in the scale of nano, but the name of the nanoparticles does not relate to them. Nanotechnology is seeking to use the powering and strange properties of the materials in the small scale. When the objects become smaller, in the range of nano, the fraction of surface area to their volume increases [6]. Hereby the importance of material in nano-scale, there are several methods reported in the literatures for synthesis of ZnO nanoparticles (ZnONPs), such as: chemical vapor deposition, gas phase method, hydrothermal synthesis, electrochemical method, microwave synthesis and the sol- gel method. Among the reminded methods the green synthesis is more beneficial than the others because of its simplest work-up, short step, costly and eco-friendly aspects. One of the problems with using chemical methods for synthesis of NPs, is the absorbed chemicals on the surface of the synthesized NPs which cause some unwanted effects in sensitive materials which are used for medical proposes [7,8]. For more information about the structure and morphology of zinc oxide, Zn atoms is located a half space of the tetrahedral places and existing octahedral locations are empty on the network [9]. Some of the other ZnO properties are

attributed about semiconductors include a broadband gap and straight [10]. For expressing more on ZnO importance there are some possess for that, such as high piezoelectric constant, intense luminescence, high thermal conductivity, resistance to radiation and superficial strong susceptibility to the presence of absorbing species [11]. By this study, ZnONPs were synthesized using cotton seed aqueous extract. The main natural compounds inside of the cotton seeds contain some special organic materials by the references and include of gossypol, camphorol, quercetin and other flavonoids are active more in oxidation and reduction process [12]. It is noteworthy the affected extracted compounds have protective effects against pathogens and antioxidants Cotton seeds contain high levels of that [13].

## 2. Material and Methods

### 2.1. Material

Sodium hydroxide (NaOH), Zinc acetate dehydrate  $[\text{Zn}(\text{CH}_3\text{COO})_2] \cdot 2\text{H}_2\text{O}$ , Ethanol ( $\text{C}_2\text{H}_5\text{OH}$ ), Whatman Filter Paper (WHA10348903) was bought from Merck company, Germany. Cottonseed was provided from cotton center of Iran (Golestan species, collected from Golestan province area). In all experiments for making of solution, the deionized water was used. Furthermore to preventing of the experimental errors, all glassware, dishes and used equipment's in the experiments, were properly washed, sanitized and dried.

### 2.2. Preparation of cottonseed extract

For the extraction process 10 g of peeled cottonseed by identified source was grinded and was placed in 100 ml of deionized water. Then by warming of the mixture to 50 °C for 4 hours, a yellowish-green solution was appeared. In next step by cooling of the wormed mixture to room temperature followed by the filtration process, a shiny yellowish green solution was collected. Finally the extracted solution was stored in a refrigerator for synthesis of target nanoparticles.

### 2.3. Synthesis of ZnO nanoparticles

Zinc acetate dehydrate (0.02 M) was prepared in 50 ml of deionized water at a room temperature. During 4 hours, 10 ml of peeled *cottonseed* extract was mixed with 50 ml of Sodium hydroxide and then 50 ml of zinc acetate slowly added to that. The mixture was placed in an ultrasonic bath for two hours for reduction process of zinc ion. The resulted white solid was centrifuged for 15 minutes at 15,000 rpm. Finally the produced nanoparticles were washed with distilled water and ethanol to elution of its impurities. Eventually the white powder was dried in an oven for one night.

### 2.4. Characterization

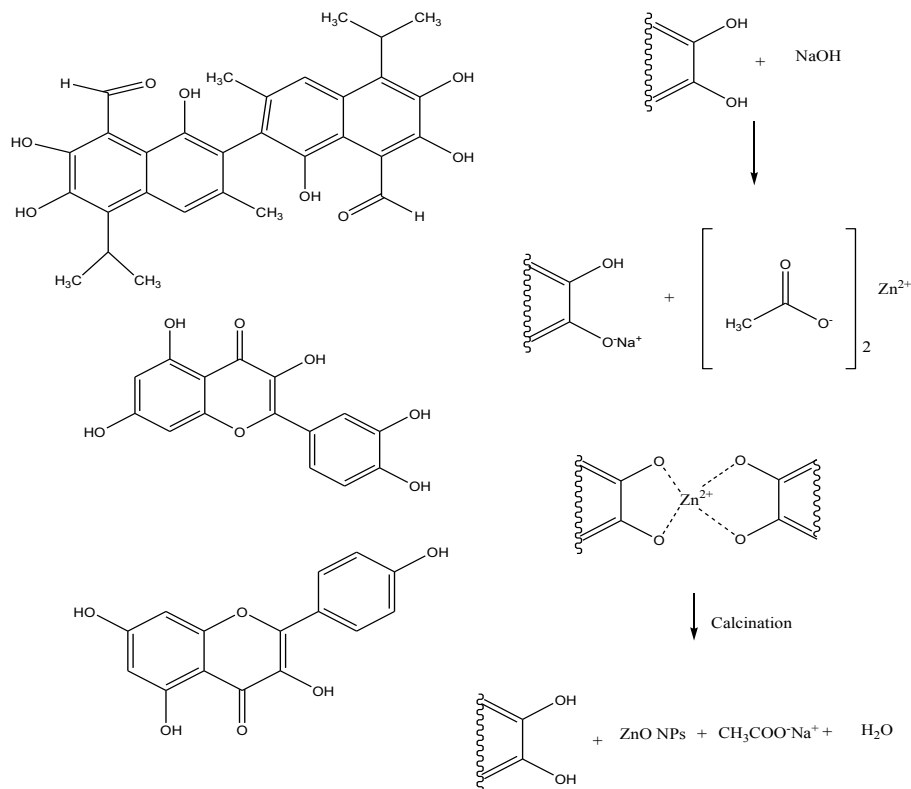
Nanoparticles were characterized using different methods such as UV-VIS or infrared spectroscopy (FTIR) [14]. The size of synthesized ZnO-NPs was performed by employing of X-ray diffraction (XRD) [15]. Also the morphology of synthesized particles was studied by scanning electron microscopy (SEM) [8].

## 3. Results and discussion

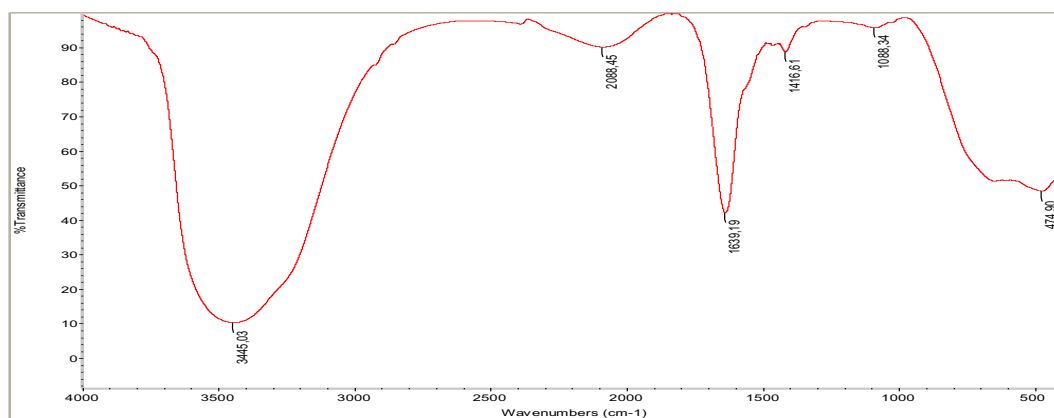
According to the probably mechanism that was showed in Figure 1, the existing biomolecules in the *cotton seed* water extract act as ligand agents. The hydroxyl groups in the aromatic moiety of the biomolecules lead to formation of zinc ions complex. This phenomenon starts the nucleation process that caused to reverse micellization and shape of the NPs [16].

### 3.1. Fourier transform infrared spectroscopy (FT-IR)

Infrared absorption spectroscopy was used for consideration the intensity of the absorption of mid-infrared light by functional groups of the sample content [17]. According to Dobrucka et al. 2016, the reading of IR spectrum includes the interpretation of the interdependence between the absorption bands (vibrational bands) and the chemical compounds in the sample. By refer of this method, a number of active molecules could be absorbed of infrared light and identified in different wavelength [18]. FTIR spectrum analysis of zinc oxide nanoparticles was showed in Figure 2. Herein the area between 400 and 600  $\text{cm}^{-1}$  is assigned to Zn-O [19]. The band 3445.03  $\text{cm}^{-1}$  is attributed to the stretch hydroxyl group (O-H) which is indicated in the Figure 2. The other peak located in 1639.19  $\text{cm}^{-1}$  probably related to the C = C stretch of the aromatic ring and the band of 1088.34  $\text{cm}^{-1}$  may be related to the presence of the C-O group.



**Figure 1.** Possible mechanism for synthesis of ZnO-NPs



**Figure 2.** FT-IR spectrum of synthesized ZnO-NPs from aqueous extract of *cottonseed*.

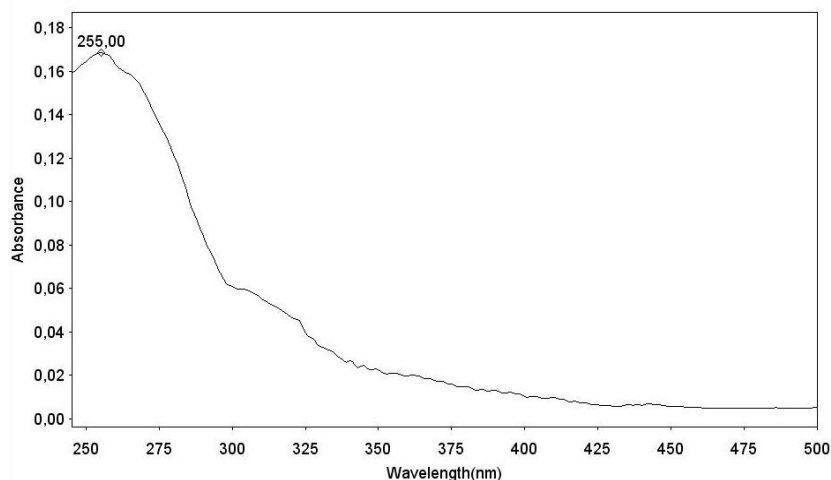
### 3.2. *Uv-vis Spectrophotometry*

The UV spectrum was measured at room temperature using a quartz cell with a path length of 1 cm. Herein the absorbance of the reaction mixture in nano-process was performed after preparation collecting of a ZnONPs solution. Typical absorption of nanoparticles was observed at 255 nm at room temperature which is attributed to the ZnO NPs (Fig. 3). The absorption edge of UV curve systematically shifts to the lower wavelength or higher energy with the decreasing size of the nanoparticle [20].

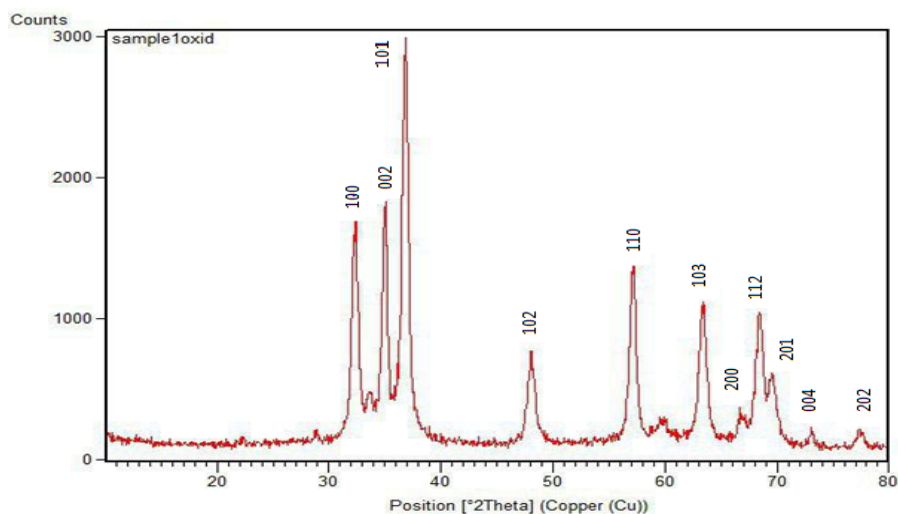
### 3.3. *X-ray Powder Diffraction (XRD)*

Obviously, in the XRD analysis results (Fig. 4) is observed in the presence of several dispersed couriers for the sample at 32.38, 34.96, 36.84, 48.08, 57.17, 63.38, 66.74, 68.46, 69.54, 73.07 and 77.39 Which respectively corresponds to Peaks 100, 002, 101, 102, 110, 103, 200, 112, 201, 004, and 202. Dispersed couriers indicate that the product is a crystalline phase and also confirm to be zinc oxide wurtzite [7, 16]. The size of nanoparticle was obtained using the Debye-scherrer equation as following:

$D = K \lambda / \beta \cos\theta$ . In this formula,  $D$  is the crystal size,  $K$  is the crystal shape factor usually 0.9,  $\lambda$  is the wavelength of the x-ray radiation (if used with copper radiation  $K\alpha$ , 0.154 nm),  $\beta$  is the peak width in half the maximum height (FWHM), and  $\theta$  is the diffraction angle (In degrees). The estimated size of the synthesized ZnO-NPs crystal was calculated to be 30.5 nm.



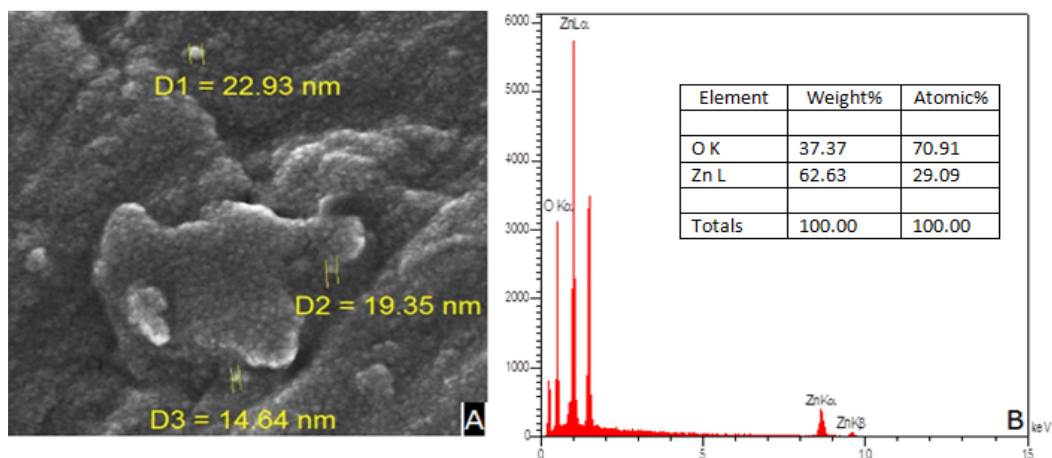
**Figure 3.** UV-VIS absorption spectra of ZnO-NPs



**Figure 4.** XRD pattern of synthesized ZnONPs from aqueous extract of cottonseed

### 3.4. Field Emission Scanning Electron Microscope (FESEM) and EDX Pattern

The morphology of the synthesized ZnO-NPs was evaluated by scanning electron microscopy (SEM) to confirm the presence of ZnONPs. As shown in the Figure 5, hexagonal particles of ZnONPs are synthesized with reduction the  $Zn^{2+}$  metal particle biomolecules by aqueous extract of cottonseed. The particles size was calculated as 22.93, 19.35 and 14.64 and the average mean particle size was about 18.97 nm. In the EDX pattern, there are two zinc and oxygen elements in zinc oxide nanoparticles. The percentages of zinc and oxygen found were 62.63 and 37.37, respectively (Figure 5).



**Figure 5.** Scanning electron microscope (Fig. A) and EDX pattern (Fig. B) ZnONPs synthesized from aqueous extract of cottonseed.

## Conclusion

In the present study it can be concluded that ZnO-NPs was synthesized from *cottonseed* water extract, which has advantages such as inexpensive, simple work-up, costly and safe method. According to the FTIR spectrum analysis, because of the presence oxidation-reduction activated compounds in the aqueous extract of *cottonseed* could be as a driving force to promoting formation of zinc oxide nanoparticles. Finally by the obtained results from XRD, a hexagonal structure of ZnO-NPs with an average size of 18.97 nm was detected. This method as a green synthesis suitable and available practical way to rich a special structure of nano-materials and could be extended to preparation others nano-scale compounds simpler and more natural for the synthesis of ZnO-NPs. It is also less polluted than the chemical method.

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