Original Article: Synthesis of ZnO nanoparticles via flaxseed aqueous extract

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ABSTRACT

Synthesis of widespread types of nanoparticles including metal oxides by plant extract is considered as alternative chemical synthesis methods. ZnO nanoparticles are synthesized by a biological process using flaxseeds (*Linum usitatissimum L.*) extract. The biosynthesized ZnO NPs were characterized by XRD and SEM, EDX, DLS spectroscopy. According to the SEM images, the synthesized nanoparticles are spherical shape with agglomeration and based on XRD, average crystallite size of the ZnO NPs is 15.45 nm

Introduction

Nanoparticles are of great importance for their potential applications in the emerging areas of nanoscience and technology [1,2]. Size, shape, and surface morphology play essential roles in controlling the physical, chemical, optical, and electronic properties of these nano materials [3]. Generally, nanoparticles can be prepared and stabilized by physical and chemical methods such as laser ablation [4], lithography [5], high energy irradiation [6], electrochemical methods [7] and photochemical reduction [8]. These methods are expensive and toxic to the environment; therefore, the use of plant extracts could be an alternative to physical and chemical methods for the production of nanoparticles in an eco-friendly method. ZnO has many applications owing to its unique characteristics which include low cost, nontoxicity, abundance in nature and the ability to prepare compounds with varying morphologies having different prosperities. ZnO is an n-type semiconductor with wide direct band gap energy (3.37 eV), a large bond strength, and large excitation binding energy (60 mev) at room temperature. Nano sized ZnO has extensive applications in solar cells [9], gas sensors [10],

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photocatalytic, antibacterial, electrical and optical devices [11], electrostatic dissipative coatings [12], degradation of environmental pollutants [13,14] and external uses as antibacterial agents in lotions, mouthwashes, ointments and surface coatings to prevent microbial growth [15]. ZnO nanoparticles exhibits a high degree of cancer cell selectivity with the ability to surpass the therapeutic indices of some commonly used chemotherapeutic agents [16,17]. To avoid these disadvantages, several

methods for the synthesis of ZnO NPs using plants extract, fungi, and bacteria have been developed as green approaches [18-37].

Flax (*Linum usitatissimum L.*), also known as linseed, is a plant widely cultured all over the world due to its seeds and fiber [38]. Flaxseed has been used for centuries for the manufacture of oil [39]. Flaxseed is a valuable source of bioactive compounds such as unsaturated fatty acids, dietary fiber and phenolic compounds [39-43].

Flaxseed, especially flaxseed hulls, are particularly a rich source of phenolic compounds [39,40]. One of the main phenolics are lignans, which compared to other edible sources are present in very high amounts [39,43]. The principal lignan found in flaxseed is secoisolariciresinol diglucoside (SDG) [39,44-45] with traces of secoisolariciresinol, matairesinol, pinoresinol, lariciresinol, hidrossimataresinol and isolariciresinol [46]. The SDG is able to inhibit the development of hormone-related breast and prostate cancers, as well as non-hormone-related colon cancers [43,46]. Additionally, flaxseed contains substantial amounts of phenolic acids (mainly p-coumaric, vanillic, chlorogenic, gallic, sinapic and ferulic, which are present as glycosides with ester and ether bonds) and flavonoids (C- and O-glycosides of flavones and tannins) [39-41]. In this research, we report the synthesis of ZnO NPs using flaxseed (*Linum usitatissimum L.*) extract as reducing agent due to the presence of phenolic compounds in flaxseed (Figure1). The synthesized ZnO NPs were subjected to different characterizations.

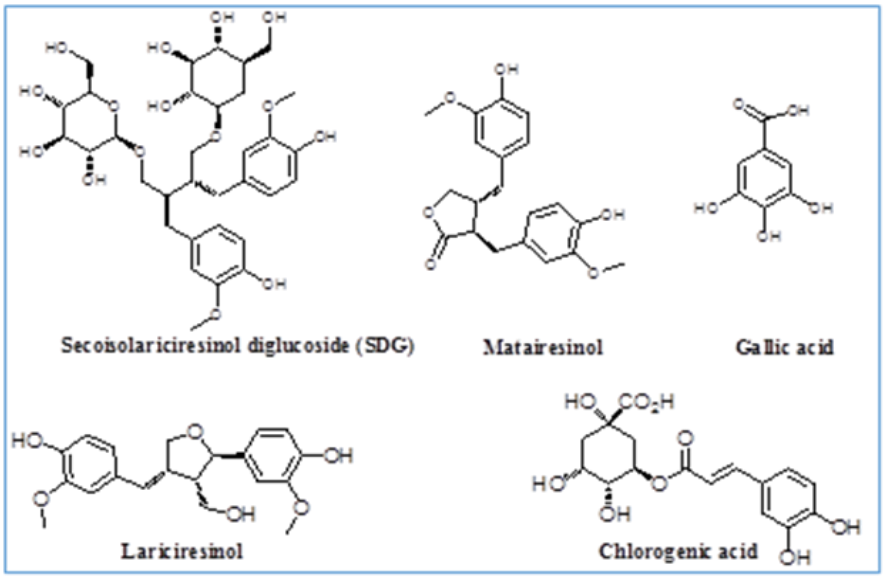

Figure 1. The structure of pheolic compounds in Flax

Experimental

General

The reagents used in this study, Zinc (II) acetaste dihydrate (99%purity), and solvents were purchased from Sigma and Merck Chemical Co.

Biosynthesis of ZnO NPs

The flaxseed (*Linum usitatissimum L.*) is prepared from the market. The flaxseed is crushed into finest powder. 10 g of flaxseed powder was refluxed in 200 mL of distilled water for 2 h. Then, the extract was filtered through Whatman No. 1 filter paper. For the synthesis of ZnO NPs, the 30 mL of flaxseed extract was mixed with 30 mL zinc acetate 0.3 M in Erlenmeyer and the mixture was heated in at 85-90 °C for 2 h. The residual solution were centrifuged at 4000 rpm for about 15 minutes for removing the unwanted organic matters and were filtered. The samples were heated at 400 °C into a for 2 h [28].

Results and discussion

FE-SEM image is used to predict the morphology of the ZnO NPs. SEM images of the synthesized ZnO NPs is represented in Figure 2. It is observed that in this method, most of the nanoparticles are spherical in shape with agglomeration. EDX spectrum (Figure 3), shows the high values of zinc and oxygen respectively. These results confirmed the presence of zinc as a majority of label compared to oxygen in precursor material. The EDX analysis displays the optical absorption peaks of ZnO NPs and these absorption peaks were due to the surface plasmon resonance of zinc oxide NPs. The origin of these elements lies in the biological components; mostly aligned along with ZnO NPs [47].

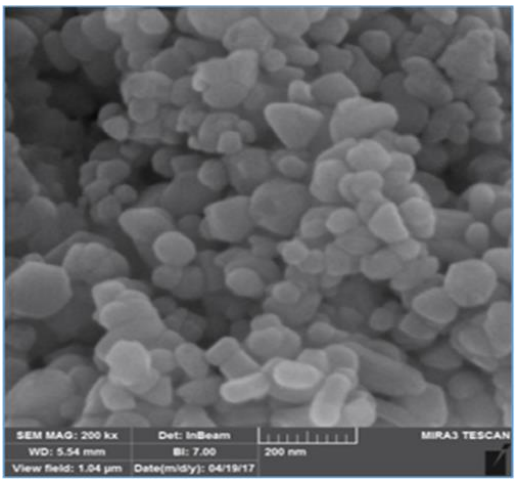

Figure 2. SEM images of synthesized ZnO NPs

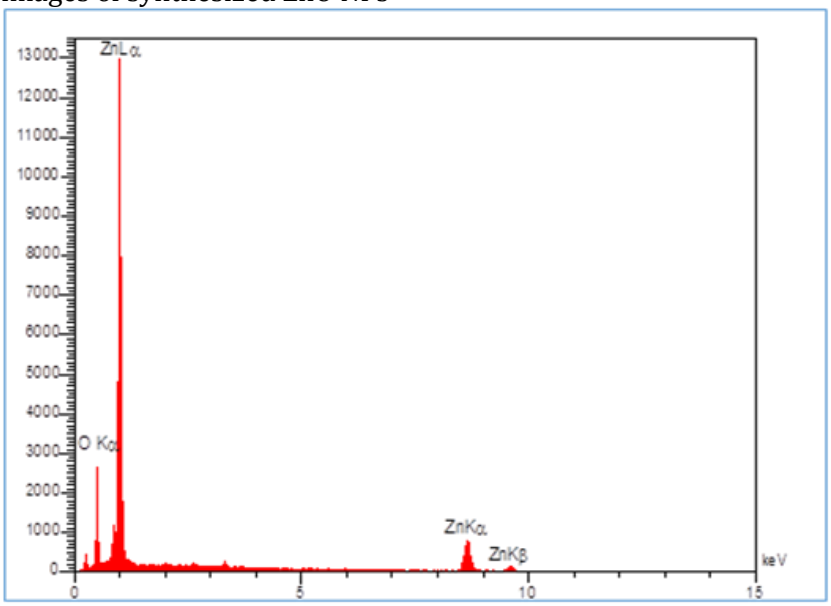

Figure 3. EDX spectrum of synthesized ZnO NPs

Structure and phase purity of ZnO NPs is shown in Figure 4, from the diffractogram of XRD, are very well matched with the hexagonal phase (wurtzite structure) by comparison with the data from JCPDS card No. 75-0576 [48] and no indication of a secondary phase or impurity peaks were obtained. The strong and narrow diffraction peaks indicate that the product has good crystalline structure (Figure 4). The detected peaks corresponded to the hexagonal phase ZnO nanoparticles are found in the lattice planes (h, l, k) of (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (2 0 0), (1 1 2), (2 0 1), (0 0 4) and (2 0 2) in the 2θ value: 31.95°, 34.56°, 36.50°, 47.65°, 56.78°, 62.93°, 66.47°, 67.96°, 69.12°, 72.61° and 76.94° respectively. The average size of ZnO NPs was calculated using the Scherer's Equation [49]:

$$d = k\lambda / ß\cos\theta$$

where d is particle size of the crystal, k is Scherer's constant (0.9), λ is X-Ray wavelength (0.15406 nm), ß is the width of the XRD peak at half height and θ is the Bragg diffraction angle. The average crystallite size of the ZnO NPs prepared by the two methods is ca. 15.45 nm.

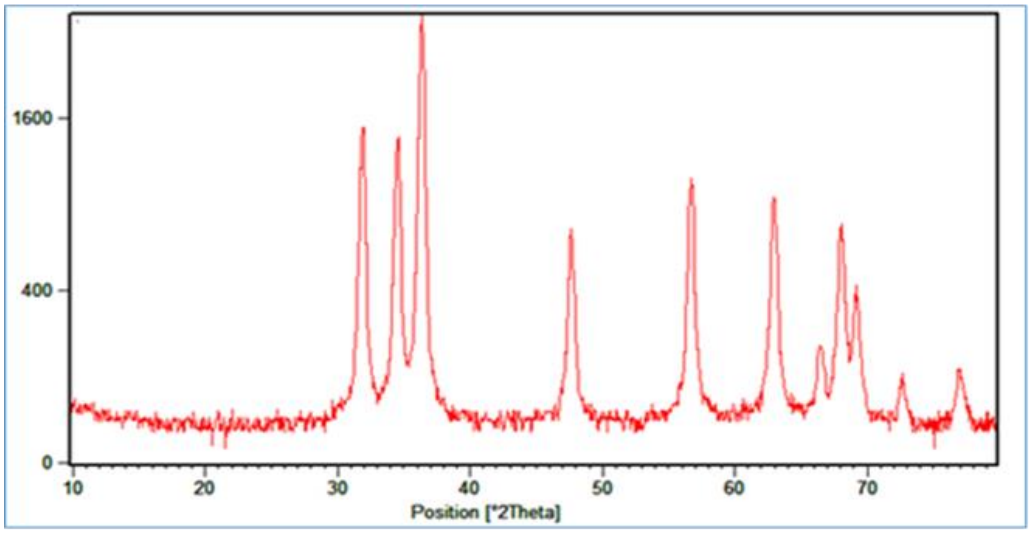

Figure 3. XRD pattern spectrum of synthesized ZnO NPs

ZnO NPs were characterized using dynamic light scattering (DLS) analysis. The particle sizes starting from 30 to 60 nm with an average size 43.5 nm (Figure 5) are detected. These values are larger than those measured by XRD method. This can be attributed to the dispersion of ZnO NPs powder in ethanol succeeding sonication. Furthermore, since the attractive forces between the dry particles are larger, the complete dispersion of the ZnO NPs powder to its primary size has not occurred [50].

The UV–Visible spectra of ZnO NPs prepared by flaxseed extract showed a characteristic absorption peak at 305 nm (Figure 6). The stretching of ZnO NPs were found around 400–800 cm^{-1}. The peaks at 420 cm^{-1} in IR spectrum (Figure 7) is corresponding to ZnO stretching. The metal oxygen frequencies observed for the respective metal oxide is in accordance with literature values [49,51].

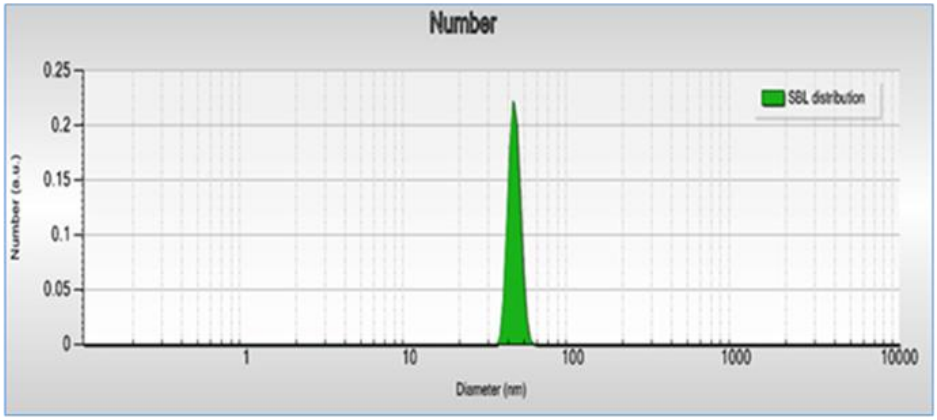

Figure 5. DLS spectrum of synthesized ZnO NPs

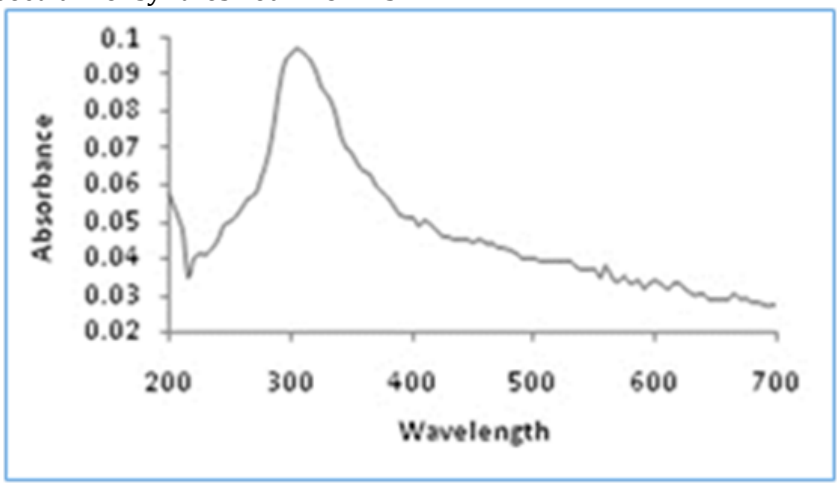

Figure 6. UV–visible spectrum of synthesized ZnO NPs

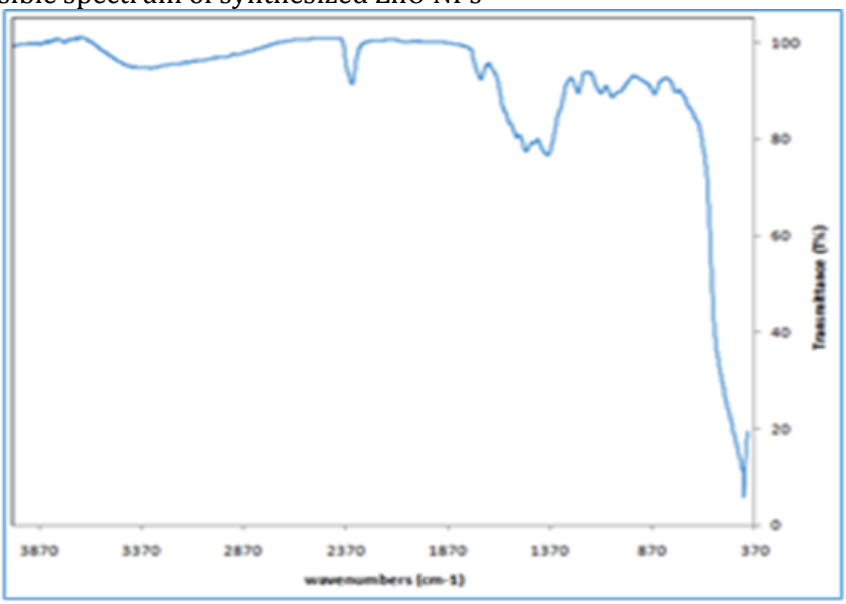

Figure 7. IR spectrum of synthesized ZnO NPs

Conclusion

In this study, a simple method has been reported to synthesize hexagonal phase ZnO nanoparticles *via* green method using Flax (*Linum usitatissimum L.*) extract.

Structure and phase purity of the ZnO NPs are evaluated by XRD. The morphology of the ZnO NPs was predicted by SEM techniques.

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