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Green Synthesis by Zygophyllum Coccineum Leaves Extract for Preparing ZnO Nanoparticles, and Characteristics Study

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Abstract

In this paper, we used the aqueous extract of the Zygophyllum Coccineum Plant Leaves for the synthesis of ZnO-NPs by a green method utilizing zinc acetate dihydrate as a precursor. The synthesized zinc oxide NPs was characterized by AFM (Atomic Force Microscopy), UV-Vis. (UV-Visible spectroscopy), FT-IR (Fourier Transform Infrared Spectroscopy). XRD (X-Ray Diffraction), FE-SEM (Field emission Scanning Electron Microscopy), and EDX (Energy Dispersive X-Ray Spectroscopy). We investigate the topography of the ZnO-NPs surface and it gave an average diameter of 67.42 nm. The wavelength of the sample was 368 nm and Eg was 3.3714 e. V. The absorption peak appears at 561 cm^{-1} which confirms the presence of ZnO-NPs nanoparticles. X-ray diffraction studies represent the size of crystallites 12.01 nm using the Debby-Scherrer formula that proposed ZnO-NPs have a hexagonal structure. The purity of zinc oxide NPs formation and the percentage of elements in nanoparticles structure were proven by EDX. SEM testing using the images of the ZnO-NPs proved that spherical shapes of nanoparticles agree with XRD analysis.

Keywords: Zygophyllum Coccineum, Zinc oxide Nanoparticles, Green method, biosynthesis, Flavonoid

1. Introduction

Green synthesis methods of ZnO nanoparticles are considered promising methods due to they are eco-friendly and applied due to the lowest possible number of toxic chemical products used, energy-efficient and cost-effective [1]. Therefore, plants and herbal extracts are more attractive, as the methodology is simpler and more economical and the presence of effective components such as flavonoids, terpenoids, and polysaccharides [2]. In chemical /physical synthesizing NPs, these types require poisonous materials or use a large amount of energy. Therefore, green synthesis methods are used as an alternate method via utilizing plant's metabolites, microorganisms' compounds to produce effective NPs [3]. The Zygophyllum coccineum plant is a halophytic type with the ability to live in tropical areas arid, high-salted, and marshy regions. it belongs to the Zygophyllaceae kind which includes about 200 species [4]. The compounds extracted from Zygophyllum coccineum was included dihydroxy flavones, flavonol glycosides, flavonol aglycones, flavones, flavone glycoside, isoflavone, anthocyanin. Other compounds from the extract included alkaloids, stilbene glycoside, sesquiterpene, aldehydes like syringaldehyde and cinnamaldehyde, and quinovic acid-based triterpenoid saponins as the total plant extract's constituents [5]. ZnO-NPs can be considered important between the nano metal oxides due to their unique chemical/physical properties, which gives them more chance of application [6]. It can be used in the industry of rubber and plastic due to it furnish waterproof and able to increase the intensity of the rubber [7]. Furthermore, ZnO-NPs molecules have the ability to adsorption of high UV, ZnO-NPs activation the products of sunscreen and cosmetic industries [8], also it appears a good semiconductor due to unique properties in nanoscale such as highly visible transparency, band gap Eg, and high electron mobility [9]. ZnO-NPs are added to finished textiles

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later in the fabrics industry to increase ultraviolet resistance [10]. It was used in various industries such as solar [11], electronics [12] antifungal, [13] concrete, [14], and photocatalysis [15], in the last years it was used as a food product additive to increase growth efficiency [16]. ZnO-NPs are odor-free and white, [17] solid powders of zinc blende, hexagonal wurtzite crystals, and cubic crystals rock salt [18]. It has a wide band gap energy (~3.3 e.V) [19]. Different methods were used to synthesize ZnO-NPs including chemical, physical, and biological methods [20]. Chemical methods involved sol-gel, vapor deposition, hydrothermal, co-precipitation, microemulsion, and solvothermal. Physical methods involved arc plasma, laser ablation, ultrasonic irradiation, thermal evaporation, physical vapor deposition [21]. The production rate for zinc oxide NPs is high in most physical and mechanical processes to use in practical operation [22]. The biogenic method uses the extract of the leaves, flowers, and roots [23], also uses Microorganisms (phage, yeast, bacteria, algae, and fungi) [24]. Classification methods of zinc oxide nanoparticles can be widely divided into two types, the bottom-up type according to a route of the growth and synthesis of nanoparticles, and the top-down class [25]. The bottom-up route has been using atoms to build nanoparticles by chemical or biological methods and controlling deposition and growth, while the top-down route involves cutting bulk materials to nano-scale materials [26]. The semiconductor substances are distinguished by the presence of band gap energy Eg, which explained the energy required to excite the electron and move it from the valence band towards the conduction [27]. A perfect estimation of E_g is important in predicting the photophysical and photochemical characteristics of semiconductor materials [28]. E_g was been calculated by different methods: using an equation of energy as [29]:

Eg= h. C/ ג --------------(1.1)

where h: Planck constant: 4.1356*10-15 eV. S, C: speed of light =3.00*108 m/s, and ג: maximum wavelength of ZnO

2. Materials and Methods

2.1.Materials

Zinc acetatedihydrateZn $(CH_3COO)_2.2H_2O$, 99.9% and sodium hydroxide (NaOH)were purchased from BHD,. *Zygophyllum Coccineum* leaves plucked from south of Iraq, Al-Shatrah city،ethanol (70%) purchased from Fluka.

2.2.Methods

2.2.1. Synthesis of ZnO-NPs by using Zygophyllum Coccineum leaves.

In this method Zygophyllum coccineum plant was used to synthesis zinc oxide nanoparticles. Zygophyllum coccineum plant was collected from the south of Iraq, AL-Shatrah city. The leaves have been washed thoroughly by tap water to remove any dust and rubble, then washing with distilled water, dried at room temperature with manual stirring without using any heat source or exposure it to the sun. Finally, the leaves are grinded to a powder by mortar, pestle and kept at room temperature away from moisture and light. Fig. (1) shows the steps of ZnO-Z preparation.25 g of the leaves powder is soaked with 250 ml of distilled water for 24 hours to extract by the cold maceration method with shaking it every 4 hours, then filtering by nomination paper [30]. The aqueous solution kept in the refrigerator at 15 C° until use. Zinc acetate dihydrate Zn $(CH_3COO)_2.2H_2O$ has been used as a precursor. 3.75 g has been taken from zinc acetate dihydrate and added to 100 ml of distilled water by stirring at 60 C° for 15 minutes. The precursor solution added to 37ml from crude plant juice that prepared previously and mixed by magnetic stirrer at 60°C for about 75 minutes, then separation by centrifugation at 3000 rpm, the dark plant paste is eliminated and the solution part taken to be placed in an open oven at 75 C° for48 hours until it turns into a thick creamy solution tinged with white, then it washed with distilled water twice to remove the remaining plant sap does not turn into charcoal during the high heating process, after that, the remaining has taken and heated to a temperature of 150 C° for three hours to obtain the yellowish-white ZnO which is washed again with water and ethanol (3:1) and heated again in 500 C° for 90 minutes to obtain the white ZnO-NPs.

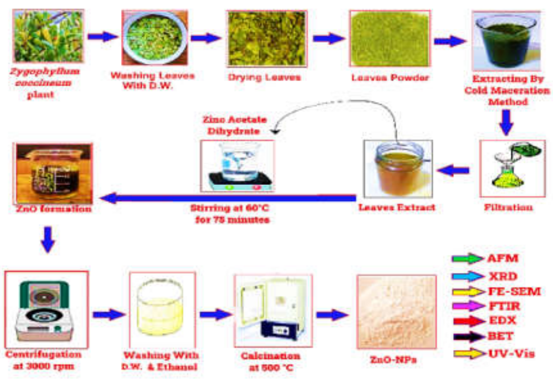


Fig. 1 Steps of synthesis of ZnO-NPs

Zygophyllum coccineum leaves extract contains various phytochemicals such as dihydroxy flavones, flavonol glycosides, isoflavone, anthocyanin, and cinnamaldehyde which act as reducing agents and significantly reduces the particle sizes [31]. After the successive reduction of particle sizes, the NPs are also affected by the terpenoids (stilbene glycoside, and sesquiterpene), terpenoids are effective covering and stabilizing agents by the interaction between them and the ZnO-NPs and which that acts to protect particles inside the reaction medium, ZnO-NPs become stabilized [32].

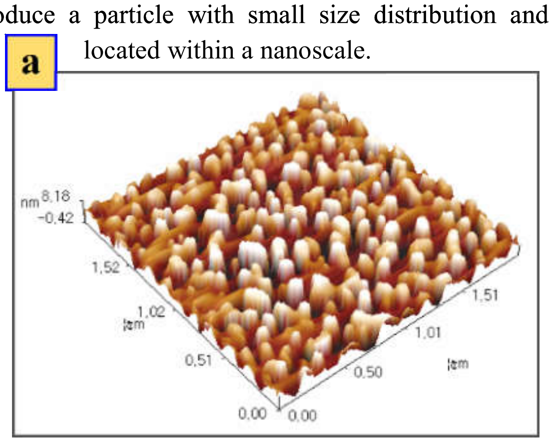
2.2.2. Characterization of ZnO-NPs

The AFM 3D images has been taken by AA3000-angstrom- USA model. The absorption spectra were measured on model Sartorius AG Göttingen BL210S Germany at wavelengths between 200-800 nm using 1 cm optical path length quartz cuvettes. Shimadzu IR-Affinity-1-Japan FTIR spectrometer was used to measure FTIR spectra at room temperature. The dried ZnO samples are mixed with KBr in the form of a round disk and it was measured in the range of 400-4000 cm^{-1}.XRD measurement of the ZnO-NPs was done on a P-Analytical XRD UK diffractometer working with Cu Kα radiation (λ= 1.54 °A). The morphology, the size of ZnO-NPs, and the elementary components were examined by FE-SEM and EDX images obtained by a ZEISS Gemini-Germany.

3.Results and Discussion

3.1. AFM analysis

The Information was getting via the AFM method related to the three-dimensional images of the surface topography of the granules and the distribution of cumulating granularity for the zinc oxide NPs [33]. Fig.2a, b shows the surface obtained from images of technique, and the granularity cumulating distribution for related to AFM measurements the average diameter is found to be 59.42 nm and the average volume of the particles is 3.16 nm3, this agreed with the fact that the prepared ZnO-NPs by green method produce a particle with small size distribution and located within a nanoscale.



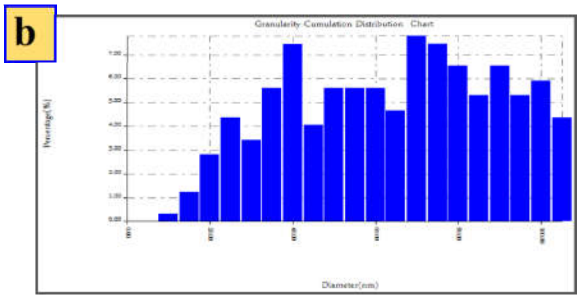


Fig. 2 (a) AFM image (b) granularity cumulating distribution chart

3.2. UV-Visible spectroscopy

The UV-visible spectrum is represented by the absorption peak at 368 nm as shown in fig. 3 which refers to the lower particle size of zinc oxide NPs sample according to the absorption peak which a wavelength it's consistent with that was mentioned in literature [34]. Eg value was reported by using an equation of energy (1.1). The calculated value of the band gap was 3.3714 e.V.

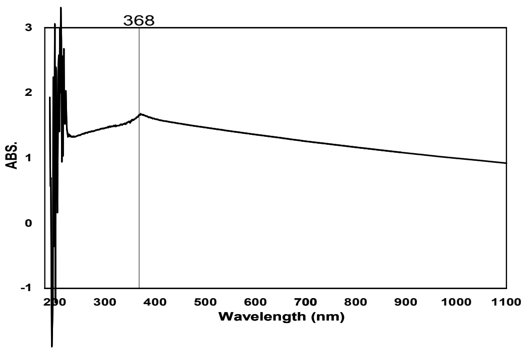


Fig. 3 Absorption max of ZnO-NPs

3.3 ATR–FTIR Spectroscopic Investigation

The FTIR spectra were estimated over a wavelength range between 400 to 4000 cm^{-1}. The characteristic bands of the zinc oxide NPs shown in Fig. (4). The broadband between 3751-3000 cm^{-1} corresponds to the hydroxyl group stretching mode of the O-H band according to the adsorption of water by zinc oxide NPs surface, this broad peak could be referred to as the effect of intermolecular hydrogen bonds and intra between it. The broad O–H peaks become narrower with an increase or decrease in the pH value due to the additional amount of NaOH added in synthesis stages, which reacts with the Zn $(CH_3COO)_2.2H_2O$ at alkaline medium [35]. The chart showed uniqueness in some bands according to the influence of the biomaterials like flavonoids, polyphenols, and amino acids, present in leaves extract. The 2881 cm^{-1} and 2823 cm^{-1} bands were due to C-H and C-C aromatic stretching vibration in aromatic aldehyde respectively, C-O in amino acid stretching bond and stretching of C-H bonds have seemed at 1096 and

1051 cm^{-1} bands respectively, also The 1600-1520 cm^{-1} band seemed due to the aromatic stretching vibration of C=C ring including C=O stretching vibration bond of polyphenols, as well as assigned to the occurrence of amide I and II groups and methylene from the proteins in the solution and C-N vibration stretching of amine due to the proteins and polysaccharides molecules present in plants extracts. This route to NH2 and free carbonyl and groups from proteins and amino acid residues refer that they have the ability to bind to zinc and that the proteins and biomaterials could possibly form a layer around the metal for preventing agglomeration and thereby stabilizing the nanoparticles. FTIR spectra revealed the fact, the protein molecules and biomaterials present in the leaf extract possibly cause the reduction of metal ions in the synthesis samples [36]. These results propose that not only OH^{-1} ions of flavonoids but also other functional groups like protein molecules play the main role in the bio-reduction of salts and capping of nanoparticles. The bands 1229 and 1327 cm^{-1} were linked to the stretching vibration of the C-N bond. The band compiled between 1000 cm^{-1} and 400 cm^{-1} were majorly due to metal and oxygen group bonding that is, ZnO presence can be reinforced. FTIR spectrum has been demonstrated various Zn–O band positions at 561 cm^{-1}. In this research, the band absorption peak associated with ZnO–NPsstretching band clearly appears at confirming the formation of ZnO-NPs.

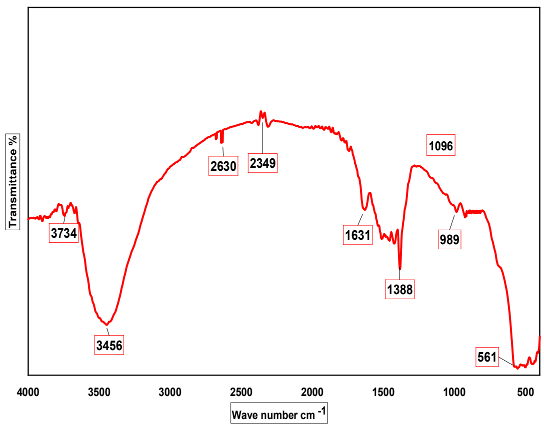


Fig. 4 FTIR spectra for ZnO-NPs

3.4 X-ray diffraction analysis

The crystalline structure of synthesized zinc oxide NPs was illustrated using measured XRD diffraction patterns, as shown in Fig.5. The XRD pattern for zinc oxide NPs were consistent with the Miller indices, and consistent with the reference cards of ZnO-NPs. Therefore, the structure of ZnO-NPs is likely to be polycrystalline with a shape that likes the hexagonal wurtzite shape. The biological method synthesized by aqueous extract of leaves of *Zygophyllum Coccineum* was consistent with reference cards (JCPDS card: 01-079-0208). ZnO-NPs sample shows the following values at the peaks 31.93, 34.58 and 36.40, which corresponds to (100), (002), and (101) of Miller indices. The crystallite size is calculated using the Debye Scherrer formula [37];

$$D = k\lambda / (\beta \cos \theta) \quad .... (3.1)$$

where, k is the constant considered as 0. 9, λ is Cu Kα radiation wavelength (1.54 °A), β is the full width athalf maximum (FWHM) of the peak in radian and θ is the diffraction angle and D is the crystal size. The crystallite size (D) for ZnO-NPs was (12.01) nm and the average crystallite size was (18.78) nm.

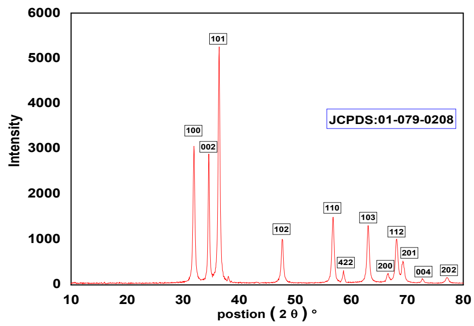


Fig.5 X-ray diffraction patterns for ZnO-NPs

3.5. EDX analysis

The purity of green biogenic zinc oxide NPs sample was determined via the EDX analysis, as shown in Fig. (6). EDX method was used to determine the composition ofthe zinc and oxygen elements that were present in the sample and has confirmed that the ZnO-NPs have high purity [38] The ratio of the percentage weight of composition atoms were (62.68 and 37.32 %) respectively.

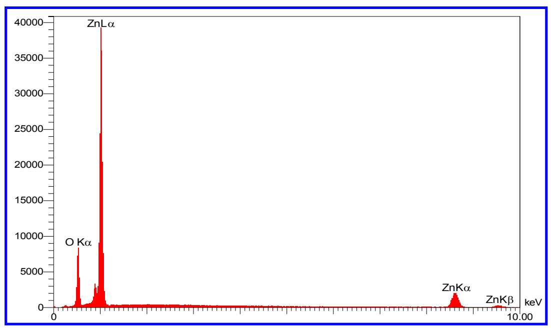


Fig. 6 EDX spectra for ZnO-NPs

3.6. FE-SEM Microscopy

The ZnO-NPs were tested under scanning electron microscopy of type (ZEISS Gemini-Germany) to explore the morphology, surface features, crystalline structure and porosity. It can be seen that the sample demonstrated excellent crystallinity as shown in Fig. (7), the size was 33.5691 nm which that calculated by IMAGE J software. The shape of the particles is often spherical with a small ratio of nanorods [39].

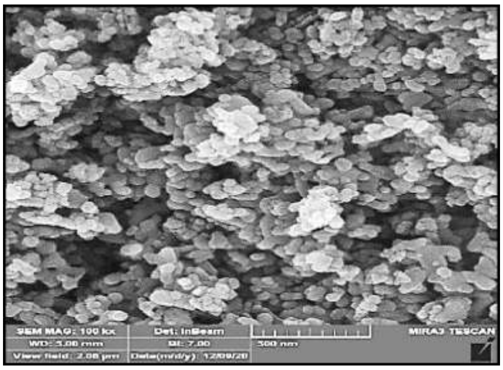


Fig. 7 FE-SEM microscopy for ZnO-NPs

4. Conclusions

The green method of preparing ZnO-NPs from the aqueous extract of Zygophyllum Coccineum leaves is considered a promising method compared with other preparation methods because it was eco-friendly without a toxic chemical agent. Among different diagnostic techniques used to characterize these particles AFM,Uv-Vis., FT-IR, XRD, EDX, and SEM.The band gap energy E_g was calculated by Einstein's energy equation (3.3471e. V). The Zn-O bond confirmed the ZnO-NPs formation at 561 cm $^{-1}$. The crystallite size wasdetermined by Scherrer equation and the size of ZnO-NPs equals to 12 nm and the structure was wurtzite hexagonal. The purity of the compound was proven by knowing the weight percentages of zinc and oxygen using EDX technique. SEM analysis supposed the morphological structure for ZnO-NPs which that a hexagonal spherical.

5. Conflicts of interest

"There are no conflicts to declare".

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