



Influence of temperature and concentration on biosynthesis and characterization of zinc oxide nanoparticles using *cherry* extract

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Abstract

Due to distinguishing characteristics of nanoparticles (NPs) in terms of size, shape, chemical composition, transmittal and different applications, nanotechnology is considered as an interesting domain of research. Application of metallic NPs is important because of the diminution of dimensions and thus the unique thermal, optical and electronic properties. This research attempts to explore the synthesis of zinc oxide NPs. Zinc oxide NPs have been synthesized using *cherry* extract under different pH, temperature and concentration and then optimum conditions for the synthesis of them were determined. For further investigations, UV–Vis spectroscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD) and Fourier infrared transformation spectroscopy (FTIR) were used. The solution containing zinc oxide NPs showed a major absorbance of 378 nm which confirmed the synthesis of zinc oxide NPs, and spherical morphology of NPs was observed in SEM images. Zinc oxide NP sizes were 6.5 and 20.18 nm which are obtained by UV–Vis spectra and XRD spectrum, respectively. Also, based on the FTIR spectra of the extract obtained before and after the synthesis, the existence of the reducing agents in herbal extract was confirmed. According to this study, the biological synthesis of NPs using plant extracts can be considered as a cost-effective and efficient method of biological synthesis of NPs and it could be an appropriate replacement to typical chemical methods for the synthesis of NPs.

Keywords Biosynthesis · Zinc oxide nanoparticles · *Cherry* extract

Introduction

Throughout the history of humanity, since the time of Greek, scientists believed that the material can be divided into small pieces so far as the smallest part could not become smaller, and the foundation of the material is based on these particles. Perhaps the Greek philosopher, Democritus, was the father of nanotechnology, since about 400 BC. He was the first person which used the term of “atom” to describe the particles that makes matter, which in Greek means indivisible. The starting point and primary development of nanotechnology are not exactly clear. Maybe it can be said that the first nanotechnologists were medieval glaziers. At that time, church glasses were made using gold nanometric particles and very

beautiful colored glasses were obtained. The color of these glasses is based on this fact that nanoscale materials do not have the same properties as micro-sized materials [1].

Nanoparticles are the most common elements in nanoscience and nanotechnology, and their interesting properties have led to numerous applications in chemical, medical, pharmaceutical, electronic, and agricultural industries. According to the chemical composition, these particles are divided into metal, ceramic, polymer, and semiconductor. Chemical preparation and solid-state processes, such as grinding and steam condensation, are the usual methods for nanoparticles production, but these methods are not affordable [2]. Nanotechnology has significant advances in nanomaterial production and the use of new methods and materials. By the development of new methods and materials, concern about environmental pollution with nanoparticles produced by chemical methods and the production of harmful side-effects has been doubled. Safe biological methods can be considered as a substitute for conventional chemical methods for nanoparticles preparation [3]. In recent years, scientific advances in nanotechnology and the use of nanoparticles

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have expanded dramatically, and among nanoparticles, zinc oxide is most likely to be of interest for researchers. Zinc oxide with a broadband gap of (3.3-eV) is a semiconductor, with special usages compared to the micron-sized particles. It is used in various industries such as burning ointments, antibacterial treatments and sunscreen, as strong absorbent UV rays, transistors manufacturing, diodes, props, atomic force microscopes, and display screens. It is used as catalyst [4], photocatalyst [5], absorbent, dental cement manufacturing, solar cell electrodes [6] and gas sensors [7, 8]. They are also used in rubber manufacturing, in the varistors and fire extinguishers, as pigments for dyeing, anti-corrosion coatings, in the glass, ceramic and glaze industry because it can reduce the thermal expansion coefficient and melting point and increase chemical resistance [9]. There are various physical–chemical methods for zinc oxide nanoparticles production [10]; for example, synthesized zinc oxide nanoparticles by ionic liquids, hydro-thermal, direct precipitation, and microwave-assisted methods, respectively [11–14].

It should be noted that synthesized nanoparticles using physical and chemical methods have some limitations, including use of toxic solvents and hazardous chemicals, which is non-eco-friendly [15].

In recent years, green synthesis of nanoparticle has become an interesting topic in nanoscience for researcher. The main goals of green synthesis are (a) producing nanoparticles that are not harmful to environment or human health and (b) producing eco-friendly nanoparticles that can solve environmental problems. Synthesized metallic nanoparticles using plant extracts have attracted great interest with the initiation of green nanotechnology for nanoparticle synthesis, [16]. So there is a great attention to synthesize metal nanoparticles using bio routes [17, 18].

During the green synthesis of metal nanoparticles, redox reactions happen in saline solutions, such that the extracts of various organisms include reducing agents that transfer electrons to metal ions and ultimately produce metallic nanoparticles [10, 19, 20].

Biosynthesis of nanoparticles using plants has a priority compared to the chemical and physical methods because of the energy and time costs reduction, and also is an eco-friendly method. There are many reports that plant extracts / different parts of plant have been used for the synthesis of ZnO NPs, such as *Trifolium pratense* flower extract [21], *Aloe vera* leaf extracts [22], *Rosa canina* fruit extract [23], *Physalis alkekengi* L. seed extract [24], and *Citrus aurantifolia* (lemon) peel extract [25], *C. aurantifolia* [26], dried sap of shoots and roots of *Astragalus gummifer* [27], *Hibiscus rosasinensis* [28], leaf extract of *Azadirachta indica* [29], Leaf extract of *Camellia sinensis* [30], *Zea mays* leaf extract [31], aqueous extract of *Abutilon indicum* [32] and *Dittrichia graveolens* aqueous extract [33]. The synthesis of ZnO NPs was studied with the leaf extract of *Ocimum tenuiflorum* and was characterized

by scanning electron microscopy (SEM), Fourier infrared transformation spectroscopy (FTIR) and X-ray diffraction (XRD). The SEM image showed hexagonal shape and NP with diameter range of 11–25 nm [34]. The synthesized ZnO nanoparticles from leaf extract of *Olea Europaea* were characterized using by FTIR, UV–Vis, XRD and SEM techniques. The average size of particles was found to be 500 nm and the thicknesses was about 20 nm by SEM studies. FT-IR analysis of aqueous *Olea Europaea* leaf extract showed the presence of surface-active molecules (phytoconstituents) such as aldehydes, amines, phenols, and alcohols that caused zinc oxide nanosheet stabilization [35, 36].

Biosynthesis of ZnO nanoparticles was widely studied before, but to our best knowledge there are no reports on its biosynthesis using *cherry* extract. In this study, the biological synthesis of zinc oxide NPs using *cherry* fruit extract has been investigated. The reason for this choice is that the *cherry* contains niacin, protein, vitamins (A, B, C) and riboflavin as well as mineral salts such as protein, sugar, water, nitrogen, hydrocarbon, fatty substances, arabinose, cellulose, carotene and cyanuric acid [37]; this plant has some bio-molecules that can act as capping and reducing agents which increase the rate of stabilization and reduction of NPs. In this study, the effects of temperature, concentration and pH were investigated, eventually; synthesized NPs were evaluated by ultraviolet (UV–Vis) spectroscopy, SEM, XRD and FTIR.

Experimental

Materials and methods

In this study, *cherry* fruit was purchased from local suppliers (Arak—Iran). All chemicals were purchased from Merck Co. Doubly distilled water was used in all the experiments.

The powder X-ray diffraction (XRD) analysis was performed by means of a XPERT Pro XRD Machine, manufactured by Panalytical, Netherlands. The morphologies were characterized by a scanning electron microscope model VEGA3-TESCAN manufactured by Elektronen-optik-service GmbH Germany, and Shin SAENG Furnace Model SEF-201 manufactured by Muffle Furnace Korean Company, and 632 Metrohm pH meter, Herisau, Switzerland, the Varian Cary-300 spectrophotometer, American Shaker made by IKA, and Perkin Elmer RXI FT-IR spectrometer were used in this study.

The extract preparation

2.5 kilograms of *cherry* water were filtered by Whatman filter paper 42 then it was boiled for 15 min, so extract solution was prepared from concentrated solution.

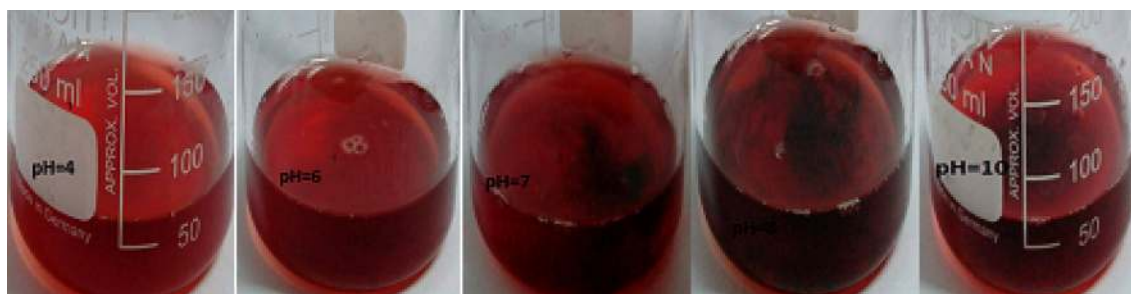


Fig. 1 Influence of pH on ZnO nanoparticles synthesis via green route using *cherry* extract

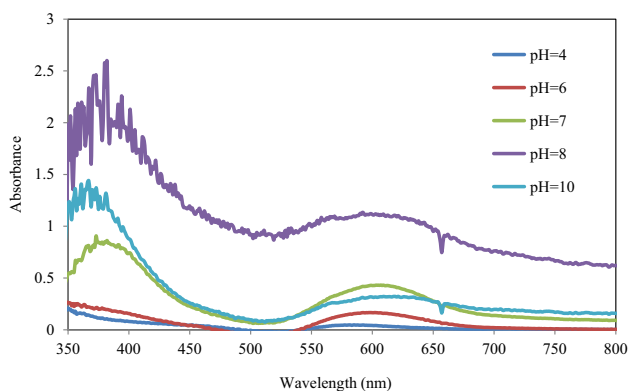


Fig. 2 UV-Vis absorption spectrum of synthesized ZnO nanoparticles at different pH using *cherry* extract

Influence of pH on zinc oxide nanoparticles synthesis by *cherry* extract

According to studies by other researchers [38], in low pH regions, ZnO is highly soluble while at high pH, partial disaggregation occurred. So we chose the pH domain ranging between 4 and 10.

10 ml of as-prepared extract solution is poured into the 5 beaker and then 30 ml of 0.3 M $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was added to them and stirred with the stirring rate of 150 rpm for 30 min at 25 °C with pH value of (4–6–7–8–10) (Fig. 1), then left them in a dark place for 12 h; after that solutions were centrifuged with stirring rate of 5000 rpm to separate the obtained precipitate. For this purpose UV-Vis spectrophotometry has been used in the range of 350–800 nm (Fig. 2). Finally, the optimum pH was selected (Fig. 3). To adjust pH, 0.1 M Nitric acid and 0.1 M NaOH were used.

Influence of temperature on zinc oxide nanoparticles synthesis by *cherry* extract

10 ml of as-prepared extract solution is poured into the 3 beaker and then 30 ml of 0.3 M $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was added to them and stirred with the stirring rate of 150 rpm for

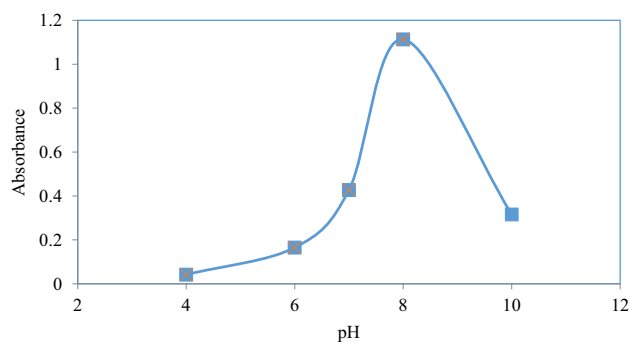


Fig. 3 pH vs adsorption spectrum for ZnO nanoparticles synthesized using by *cherry* extract

30 min at different temperatures (25, 60 and 90 °C) then left them in a dark place for 12 h; after that solutions were centrifuged with stirring rate of 5000 rpm to separate the obtained precipitate. After drying and calcining, the precipitate was characterized by SEM, according to the results, the optimum temperature was selected for further experiments (Fig. 4a–c).

Influence of metal salt solution concentration on zinc oxide nanoparticles synthesis by *cherry* extract

To study the effect of concentration of zinc nitrate solution on biosynthesis of ZnO nanoparticles, according to previous studies [39], different concentrations were selected. During the synthesis, 10 ml of as-prepared extract solution is poured into the 4 beaker and then 30 ml of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ with different concentration (0.005, 0.02, 0.05 and 0.3 M) was added to them and stirred with the stirring rate of 150 rpm for 30 min at 25 °C (Fig. 5) then left them in a dark place for 12 h; after that solutions were centrifuged with stirring rate of 5000 rpm to separate the obtained precipitate. After drying and calcining, the sediment was characterized by SEM (Fig. 6a–d). According to the results, the optimum metal salt solution concentration was selected for further experiments.

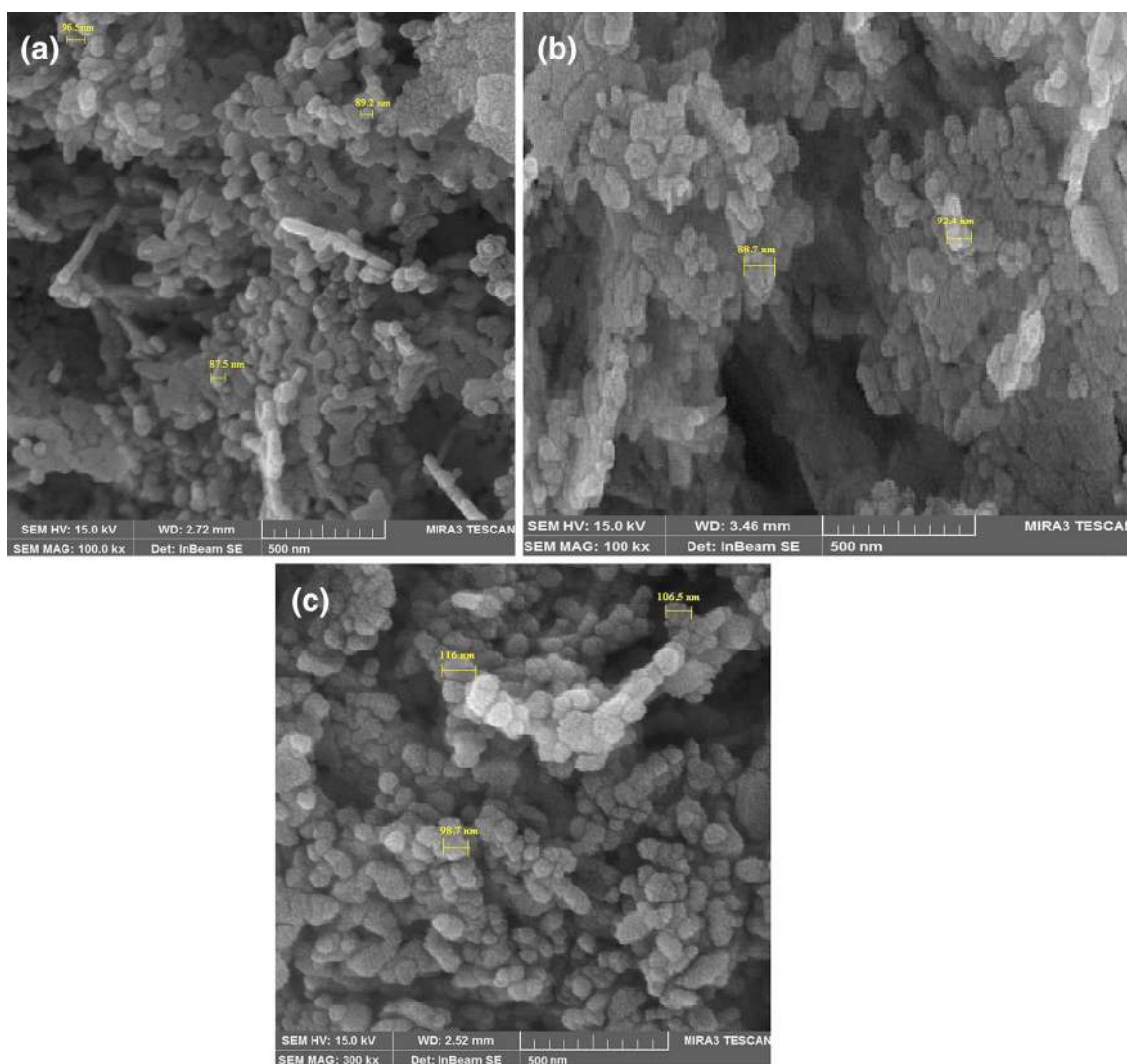


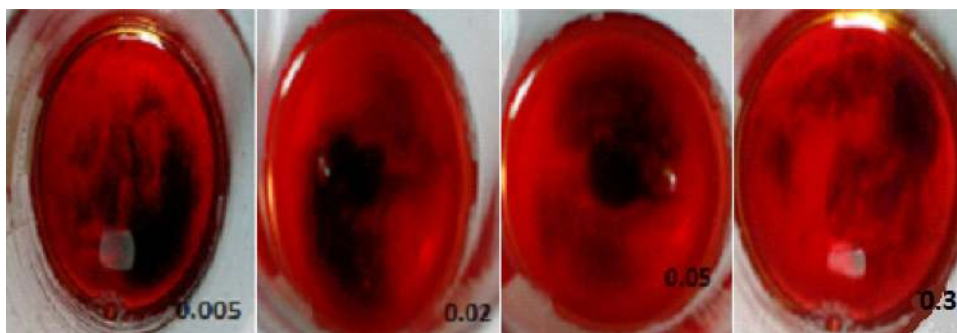
Fig. 4 Scanning electron micrograph (SEM) of synthesized ZnO nanoparticles using zinc nitrate (0.3 M) with pH = 8, **a** at 25 °C, **b** 60 °C and **c** 90 °C

Characterization of metal nanoparticle

After determining the optimum conditions (pH = 8, $t = 25$ °C, $C = 0.005$ M), ZnO nanoparticles were again

synthesized using *cherry* extract and evaluated by UV–Vis (between 350 and 500 nm), FTIR and XRD techniques.

Fig. 5 Influence of metal salt solution concentration on ZnO nanoparticles synthesis via green route using *cherry* extract



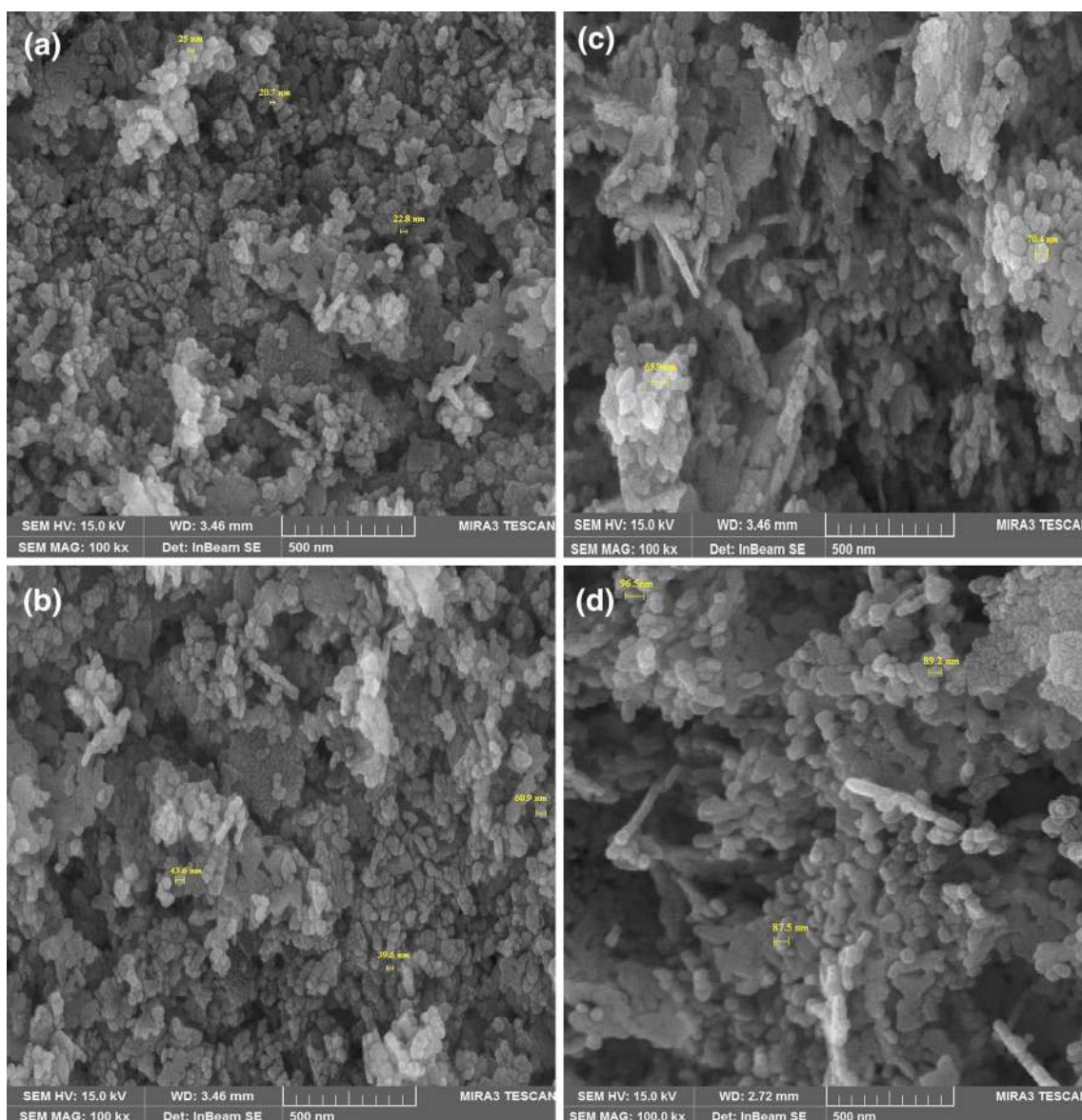


Fig. 6 Scanning electron micrograph of biosynthesized ZnO nanoparticles at 25 °C and pH=8 using different concentration of zinc nitrate, **a** 0.005 M, **b** 0.02M, **c** 0.05 M and **d** 0.3 M

Results and discussion

Influence of pH on zinc oxide nanoparticles synthesis by *cherry* extract

pH is another important factor affecting the reduction of metal ions. The major effect of pH is changing the electrical charges of biomolecules which might change their reducing and capping ability and then the growth of nanoparticles [40]. The effect of pH on the reduction of metal ions was studied by UV–Vis spectroscopy and is shown in Fig. 2. It is well known that a strong absorption band in the range of 300–550 nm is due to ZnO nanoparticles [41]. At pH=4.0

in the range of 350–550 nm, no main absorption peak was observed but a main absorption peak appeared about 373 nm when pH increased from 4 to 10 indicating the formation of ZnO nanoparticles. It was observed that higher pH (pH 8) enhances the rate of reduction as the colour of the solution turned colloidal brown more quickly compared to a solution of lower pH (Fig. 1). With increase of pH from 4 to 8, surface plasmon resonance (SPR) absorption peak shifted to larger wavelength indicating the formation of larger size ZnO nanoparticles and the red shift; however with increase of pH from 8 to 10, SPR absorption peak shifted to the blue shift that will be due to smaller size of ZnO nanoparticles [42]. It is found that at the strongly alkaline condition, the

more abundance OH ions in the solution, form a complex with Zn^{2+} cations as the molecules $Zn[OH]_4^{2-}$ and $Zn(OH)_2$, which could restrain the production of ZnO nanoparticles [43, 44]. The area under the SPR-peak in the UV–Vis spectra directly depends on the shape and size of the metal nanoparticles as well as dielectric constant of the metal and the surrounding medium in aqueous suspensions [45] and causes the spectrum to be noisy. The optimum pH for nanoparticles synthesis depends on the substrate or stabilizing agent which is used in the synthesis. According to the obtained results from UV–Vis spectroscopy and pH-absorption spectrum of synthesized ZnO nanoparticles (Fig. 2), it was found that the best pH for synthesis of hexagonal small size nanoparticles via green route using *cherry* extract as a reducing and stabilizing agent is pH = 8 (Fig. 3).

Influence of temperature on zinc oxide nanoparticles synthesis by *cherry* extract

It has been demonstrated that biosynthesized nanoparticles size can be controlled by changing the solution temperature [42, 46, 47]. Figure 4 shows SEM images of ZnO nanoparticles at different temperatures (25, 60, and 90 °C). It can be observed that the increase in temperature caused the increase in the size of ZnO NPs (87.5–116 nm) which indicate the reduction rate of metal ions increased by temperature; because high temperature will lead to too high reaction kinetics. It is impossible to control the growth step of the crystallization process in reactions with fast kinetics; on the other hand, an explanation for this is that *cherry* extract is a reducing agent which is rich in ascorbic acid [48] and this acid becomes slightly unstable at higher temperatures [49] and leads to a poor reduction process, uncontrolled and fast aggregation.

SEM images of nanoparticles (Fig. 4) in this synthesis show their dimensions and according to SEM images, the morphological surface of ZnO nanoparticles was optimized at 25 °C.

Influence of metal salt solution concentration on zinc oxide nanoparticles synthesis by *cherry* extract

Figure 5 shows effect of zinc nitrate concentration in the ZnO NPs biosynthesis using extract of *cherry* at different concentration (0.005, 0.02, 0.05 and 0.3 M). When zinc ions concentration was increased from 0.005 to 0.3 M, biochemical reduction starts immediately, and the lower quantities of hexagonal structural nanoparticles formed with an average size of 20.7–96.5 nm (Fig. 5a–d) that this is mainly due to the formation of large anisotropic particles. This perhaps may occur because by fixing the quantity of *cherry* extract, the concentration of groups/bio-molecules such as protein,

vitamins (A, B, C) and sugars which capped and stabilize the nanoparticle. Also, these SEM images show that the concentration of biosynthesis ZnO nanoparticles occurred quickly and the larger size of them formed that due to the occurrence of competition between zinc ions and functional groups of *cherry* extract and rate of reduction also increased [50, 51]. Thus, the optimum concentration of zinc nitrate for ZnO biosynthesized nanoparticle via green route using *cherry* extract was found to be 0.005 M.

UV–Vis analysis

The UV–Vis spectra of ZnO nanoparticles biosynthesized using *cherry* extract is shown in Fig. 7. The metal nanoparticles have a SPR broad absorption in the UV–Visible region [52, 53]. From this UV spectra, SPR peak for ZnO nanoparticles is obtained at 378 nm indicating combined vibration of electron of nanoparticle with the light wave [54]. Similar results were reported by Ghorbani et al. [41]. The following equation (Eq. 1) is used to find the size of the nanoparticle from the absorbance spectra (Fig. 7) [55, 56].

$$r(\text{nm}) = \frac{-0.3049 + \sqrt{-26.23012 + 10,240.72/\lambda_p(\text{nm})}}{-6.3829 + 2483.2/\lambda_p(\text{nm})} \quad (1)$$

where r and λ_p are the particle radius and the peak absorbance wavelength (nm), respectively. The absorbance peak at 378 nm is related to the particle size of 6.5 nm.

X-ray diffraction analysis

According to the XRD analysis and the obtained results pattern, the sharp and high peak indicates the crystallinity of the prepared nanoparticle (Fig. 8).

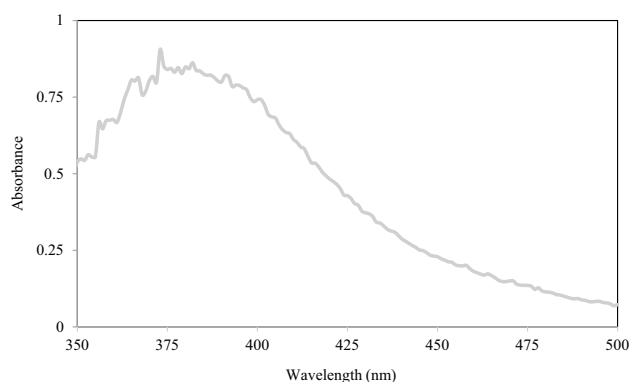


Fig. 7 UV–Vis spectra of ZnO nanoparticles synthesized by *cherry* extract at optimal condition, (10 ml of *cherry* extract, 30 ml of 0.005 M Zinc nitrate solution, pH = 8, temperature 25 °C, time = 12 h)

The diffraction peaks at 2θ values of 32.04, 34.675, 36.502, 47.773, 56.799, 63.100, 68.204, 69.394, 72.764, 77.147 correspond to (100), (002), (101), (102), (110), (103), (112), (201), (004) and (202), respectively, which are characteristic of the hexagonal structure of ZnO nanoparticles. All peaks were well matched with JCPDS card No. 80-0075.

Also, according to the Scherer–Debye equation (Eq. 2):

$$D = \frac{K \times \lambda}{\beta \times \cos \theta} \quad (2)$$

in which the parameters are as follows, respectively: D is the size of the crystal, its unit is equal to λ unit and is usually angstrom or nm; λ is the X-ray wavelength; K is a dimensionless shape factor, with a value close to unity; β is the full width at half maximum (FWHM); θ is the peak position on the horizontal axis of diffraction pattern, which, if the horizontal axis is 2θ . It should be divided into two to get θ . With the necessary calculations, the zinc oxide nanoparticle average size is 20.18 nm. Similar results of *Nyctanthes arbor-tristis* flower extract [57] and *Corymbia citriodora* leaf extract [58] have been reported for synthesizing zinc oxide nanoparticles.

Fourier transform infrared spectroscopy analysis

The FT-IR spectrum of the extract sample before and after the synthesis of metal nanoparticles is shown in Figs. 9 and 10. A broad peak within the wavenumbers ranging from

3650 to 3200 cm^{-1} attributed to the O–H functional group, peak in the range of 2850–3000 cm^{-1} can be assigned to the C–H alkane functional group. Six peaks within the wavenumbers ranging from 3500 to 3300, 1630 to 1850, 1680 to 1600, 1450 to 1340, 1350 to 1000 and 800 to 600 cm^{-1} can be assigned to the N–H, C=O (The carbonyl stretch of a carboxylic acid), C=C, CH_3 , C–N and Cl respectively. The band at 825 cm^{-1} is due to asymmetrical and symmetrical stretching of zinc carboxylates resulting in the involvement of carboxylic groups in protein of *cherry* leaf extract. The peak which is observed from 400 to 500 cm^{-1} is due to Zn–O vibrations of ZnO nanoparticles, as observed earlier [31, 59–61]. Peak at 1627.24, 3416.92 cm^{-1} corresponds to C=O stretching and O–H stretching of organic compound. FTIR analysis of zinc nanoparticles indicated that they might be surrounded by these organic molecules [62].

According to the comparison of two spectra, the absence of some peaks or the presence of some new peaks in the spectrum of the extract after nanoparticle synthesis is an evidence for the performance of the reducing agents and nanoparticles formation.

Conclusion

Green nanotechnology means “the development of clean technologies” for reducing human and environmental health threats associated with the fabrication and use of nanotechnology products, as well as replace existing products with

Fig. 8 X-ray diffraction pattern of zinc oxide nanoparticles synthesized by green route using *cherry* extract at optimal condition, (10 ml of *cherry* extract, 30 ml of 0.005 M Zinc nitrate solution, pH = 8, temperature 25 °C, time = 12 h)

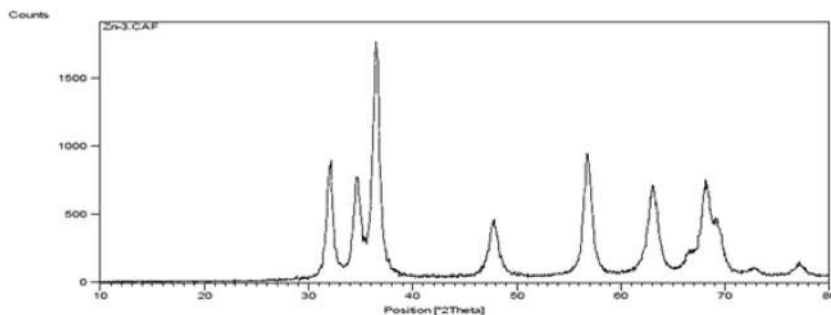


Fig. 9 FT-IR spectra of ZnO nanoparticles synthesized by *cherry* extract at optimal condition, (10 ml of *cherry* extract, 30 ml of 0.005 M zinc nitrate solution, pH = 8, temperature 25 °C, time = 12 h)

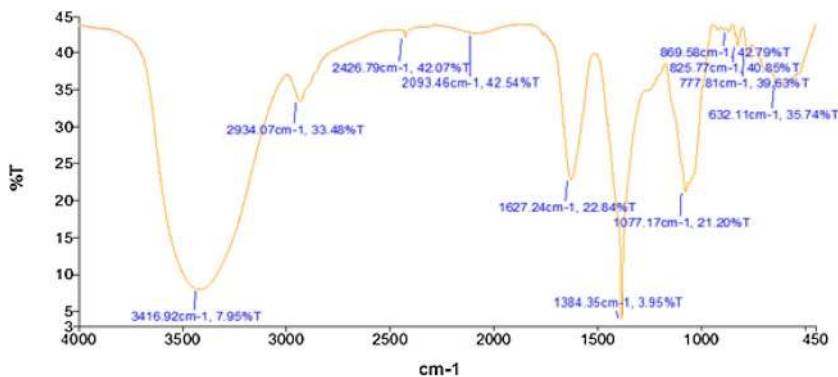
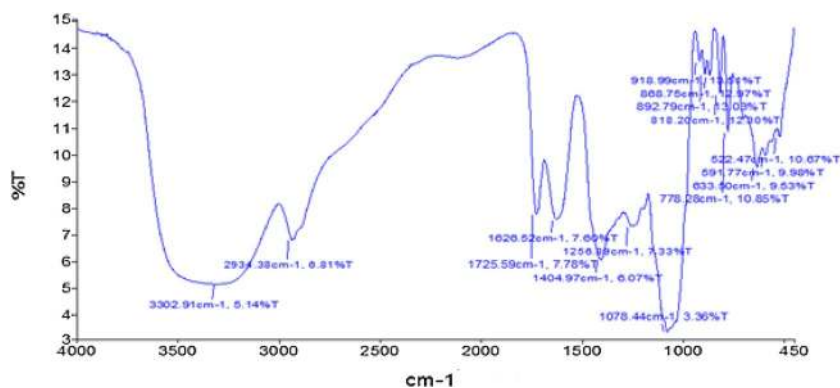


Fig. 10 FT-IR spectra of pure *cherry* extract



new nano-products that are eco-friendly. Main goals of green nanotechnology are: (a) production of nano-materials and products which are not harmful to environment or human health, (b) production of nano products that provide solutions for environmental problems.

In this work, using UV–Vis spectrophotometry in the range of 350–800 nm, biosynthesis of zinc oxide nanoparticles by *cherry* extract was studied. To achieve optimal synthesis conditions, effective parameters such as pH, temperature and concentration of zinc nitrate were investigated. In optimum conditions, hexagonal zinc oxide nanoparticles with an average size of 20.18 nm, using 10 ml of *cherry* extract and 30 ml of 0.005 M zinc nitrate at pH = 8, temperature = 25 °C and time = 12 h were synthesized. The obtained results from UV–Vis spectrophotometry, SEM, XRD and FTIR analysis confirmed the efficiency of *cherry* extract in the synthesis of high purity, crystalline zinc oxide nanoparticles. Also, in the FTIR spectrum of the extract, reducing functional groups for zinc ions reduction was observed. According to the results, this fruit is capable of synthesizing nanoparticles, and since there are no reports about this plant, the results clearly show the good functioning of this plant. Therefore, it can be concluded that synthesis of nanoparticles using herbal extracts instead of chemical and physical methods for preparation of these nanoparticles is an appropriate alternative. Finally, in this study, it was found that zinc oxide nanoparticle can be synthesized by green chemistry route without any harmful chemicals.

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