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Research Article

EFFECT OF TRANSITION METAL DOPING ON THE STRUCTURAL, OPTICAL, THERMAL PROPERTIES, AND ANTIMICROBIAL ACTIVITY OF ZINC OXIDE NANOPARTICLES

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ABSTRACT

Effect of transition metal dopant on structural, optical, thermal properties, and antimicrobial activities of synthesized zinc oxide nanoparticles (ZnO NPs) were investigated in this study. undoped, cobalt, and nickel doped ZnO nanoparticles are successfully synthesized using chemical co-precipitation method. The structure and morphology of these nanoparticles are characterized using X-ray diffraction (XRD), atomic force microscope (AFM), scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), as well as an appropriate physical measurement; (Vibrational and electronic) spectroscopy. The Results obtained from XRD indicate that the particles are possessed in wurtzite structure , SEM and AFM studies reveal that the particle size is in the 45-65 nm range. The EDS confirmed the Zn, O, Co, and Ni as major elements of as-synthesized nanoparticles. Ultraviolet-visible (UV-Vis) spectra show that doping Co and Ni, lead to an increase in the absorption edge wavelength and a decrease in the band gap energy, Fourier- transform infrared (FT-IR) spectra give the presence of functional groups and chemical bonding as well as doped sub-lattice. To measure the biological activity and potential antimicrobial efficiency of these undoped, Co, and Ni-doped ZnO nanoparticles on the Candidates.

Keywords: Doped, ZnO nanoparticle, co-precipitation method, and antimicrobial activity.

INTRODUCTION

The nanotechnological applications in medicine have yielded a completely new field of technology that is set to bring significant advances in the fight against a range of diseases[1]. Nanoparticles have risen as novel microbial activity owing to the high surface area to volume ratio, which will become of utmost importance to the researcher due to the fact of the developing microbial resistances against metallic element ion, antibiotics, and the advancement of safe strains[2]. Numerous nano- particles display incredible antimicrobial productivity and used to control medicate safe microbial populations[3]. Different inorganic metal oxide nanoparticles such as ZnO, MgO, TiO_2 and SiO_2 exhibit significant antimicrobial activities and used in therapeutics, diagnostics, and Nanomedicine-based antimicrobial agents[4,5]. Inorganic nanoparticles show greater effects on resistant strains of microbial pathogens, less toxicity, heat resistance and provide mineral elements essential to human cells [6]. Among these metal oxide nanomaterials, ZnO should be given a special attention, due to its essential role in healthcare products, UV blocking capability, biocompatibility and modest cost [7].

Furthermore, microbial contamination effects should not be underestimated in the healthcare and food industry so that the development of antimicrobial agents and surface coatings have been a motivating factor for many researchers in the recent decades[8]. ZnO nanoparticles are known to be one of the multifunctional inorganic nanoparticles with powerful antibacterial action. Antibacterial and antifungal activities of zinc oxide nanoparticles are observed even at very low concentrations and also the antifungal activity does not influence soil fertility compared to the conventional antifungal agents [9]. A large number of studies have been performed over the last decades on the properties of ZnO that can be tuned by doping with various metals ions[10, 11]. The transition metals (Ni, Co, Al, Cu, Ag) doped ZnO display best properties, as transition metal elements have close ionic radius parameter to that of Zn^{2+}, which means that these elements can easily penetrate into ZnO crystal lattice or substitute Zn^{2+} position in crystal[12]. Doped transition metals in zinc oxide optimize the electronic, magnetic, optical, and electrical characterization that is useful for various practical applications[12]. Doped ZnO demonstrates a greater effect against pathogenic organisms when contrasted with ZnO, in this way using nanoparticles as an antimicrobial agent and considered one of the most useful techniques to minimize the cost and chemical waste [13]. Ni is an essential depend on these magnetic materials. Besides, Ni^{+2} (0.69 A °) has the same valence as Zn^{+2} and its radius is close to that of Zn^{+2} (0.74 A °), so it is workable for Ni^{+2} to replace Zn^{+2} in ZnO lattice. Some researchers reported that the luminescence properties of zinc oxide are changed after doping of Ni [14].

In this study, an experimental investigation in order to synthesize and characterize the structure of zinc oxide nanoparticles doped with cobalt and nickel is performed using a chemical co-precipitation method. It highly predicted to become the useful tool to fight against microbial infections in the near future.

MATERIALS AND METHODS

All the starting materials, solvents as well as reagents, were purchased commercially and then used without any further purification.

Instrumentation

X-ray Diffraction (XRD)

The crystal structure and lattice parameters of the synthesized of undoped ZnO, Co-doped, and Ni-doped ZnO nanoparticles were used Philips (X-ray diffractometer XRD- 6000).

Atomic force microscopy (AFM)

The particle sizes and morphology of the nanoparticles were estimated by atomic force microscopy (AFM). The AFM images were performed with an (AFM AA3100) microscope.

Scanning electron microscopy (SEM)

The morphology of the prepared nanoparticles was estimated using an Oxford instrument attached to JSM-5300 scanning microscope equipment.

Energy dispersive X-ray Spectroscopy (EDS) Analysis

The elemental analysis of the particles was performed using the scanning electron microscope (SEM) that attached with an electron dispersion spectrometer (EDS).

Ultraviolet-visible (UV–Vis) and Fourier Transform Infrared, (FTIR) spectroscopy

Ultraviolet-visible Uv-visible spectra were measured using Shimadzu Uv-vis. 160 A-Ultra-violet spectrophotometer in the range (200-1000) nm. Fourier transform infrared analyzed spectra in the frequency range (400-4000) cm^{-1} were recorded as KBr discs on FTIR.8300 Shimadzu spectrophotometer**.**

Thermal gravimetric analyses (TGA)

Thermal analyses (TGA) were achieved in N_2 atmosphere with a heating rate 10℃ min^{-1} using a Perkin- Elmer model 4000 instrument.

Synthesis of ZnO nanoparticles

Zinc Oxide nanoparticles were prepared using the chemical precipitation method. A suspension of (0.2 M) zinc acetate dihydrate dissolved in (50ml) deionized water and was stirred for a half hour. Then (1M) of sodium hydroxide was added dropwise to zinc acetate solution and mixed at 70℃ with constant stirring to get the solution with the pH (12). A white gel precipitate was formed, washed with deionized water and followed by ethanol. After that, a white powder was obtained after drying at 70 ℃ in the vacuum oven overnight.

Synthesis of ZnO: Co^{+2} and ZnO: Ni^{+2} nanoparticles

For the synthesis, transition metal doped ZnO nanoparticles, (0.2M) of transition metal salts dissolved in (50ml) deionized water was added dropwise to the zinc acetate solution after stirring for a half hour. Then (1M) of sodium hydroxide was added dropwise to the solution and mixed at 70℃ with constant stirring to obtain the solution to with the pH (12). The obtained precipitate was filtered, then washed with water and ethanol, and finally dried at 50℃. The prepared samples were then used for further experimental analysis.

Biological Studies

The *in vitro* antimicrobial activity of the synthesized compounds were tested against two types of fungi which are (Candida albicans and Candida tropicalis) using the disc diffusion method. Each one of the prepared compounds was first dispersed in methanol to form monodispersed and then each nanoparticles were was sonicated for 30min.

The antifungal test was performed according to the disc diffusion method. The culture was inoculated and then was well formed in the medium. Moreover, the plates were incubated at room temperature and after 48 hours of incubation, the growth inhibition was examined utilizing the zone formation.

RESULTS AND DISCUSSION

Characterization

X-ray diffraction (XRD) analysis

The XRD pattern of the prepared ZnO, Co- doped, and Ni-doped ZnO is shown in Figure 1 (a, b, and c). The XRD of the ZnO shows a diffraction peak at $2\theta = 31.80°, 34.45°, 36.28°, 47.57°$, and 56.62° which can be indexed to (100), (002), (101) (102) (110) and (200) crystal plane respectively. The position of these peaks indicates the formation of hexagonal wurtzite structure which generally is in agreement with standard JCPDS (NO-36-1451) (2157-7439). Similar characteristic peaks and assigned lattice plane are observed in some other studies too[15]. The X-ray diffraction pattern of Co-doped ZnO consists of diffraction peaks located in 2θ = 19.7785, 23.4242, 27.5667, 28.9447, 33.2112, 33.6991, 48.5689, 59.1846, and 69.5614 respectively as shown in (Figure 1 (b)). Furthermore, no peaks associated with Co are detected in the XRD pattern indicating that Co^{2+} ions have substitutionally replaced the Zn^{2+} [16]. The XRD pattern of Ni-doped ZnO(Figure 1 (c)) show peaks in (100) and (101). No signals of the metallic Zn are detected by XRD. However, there is no peak attributed to Ni, suggesting that the Ni^{+2} ion may be doped into ZnO[17]. It can be concluded from the results that incorporation of Co and Ni into ZnO lattice enhances crystalline quality.

The average crystalline size is calculated using Debye–Scherrer equation [18],

$$D = \frac{k\lambda}{\beta \cos\theta} \qquad (1)$$

Where; D = is the average crystalline size of the nanoparticles, K = is a geometric factor (0.94), λ= is the wavelength of X-ray radiation source, β = is the angle (full-width at half maximum)

FWHM of the XRD peak at the diffraction angle. The calculated average crystalline size of the undoped, Co-doped,and Nidoped ZnO is about (2-13) nm. From the results in Table.1, the incorporation of Co and Ni into ZnO lattice improves crystalline quality that calculated using Debye–Scherrer formula.

Atomic Force Microscopy (AFM)

The Atomic Force Microscopy image is used to determine the distribution of the particle size and estimate the mean size of the particles. Figure (2a, 2b, and 2c), show the two-dimensional (2D) and three-dimensional (3D) AFM images of pure ZnO, Co doped ZnO, and Ni doped ZnO respectively. It can be observed that the average size is about 50 nm,45nm and 65nm for ZnO, ZnO: Co^{+2} and ZnO: Ni^{+2} nanoparticles respectively.

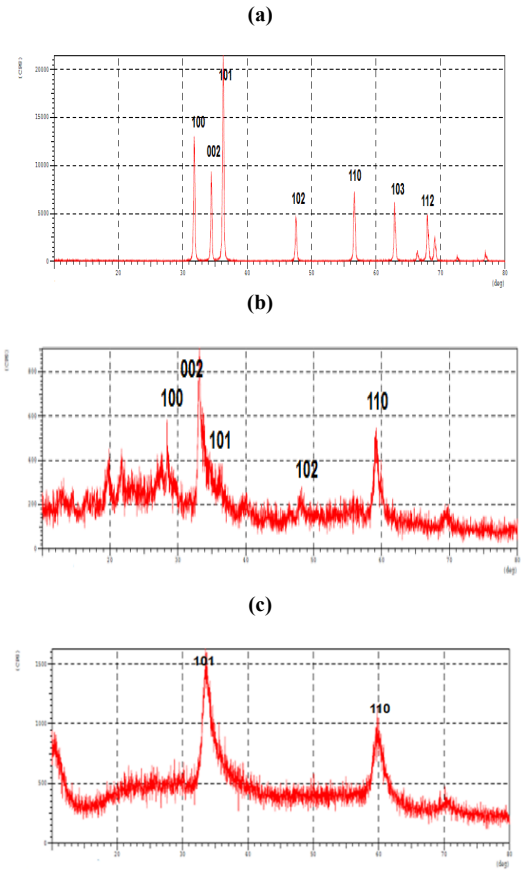


Fig. 1: The X-ray diffraction of (a) Undoped, (b) CO doped, and (c) Ni doped ZnO.

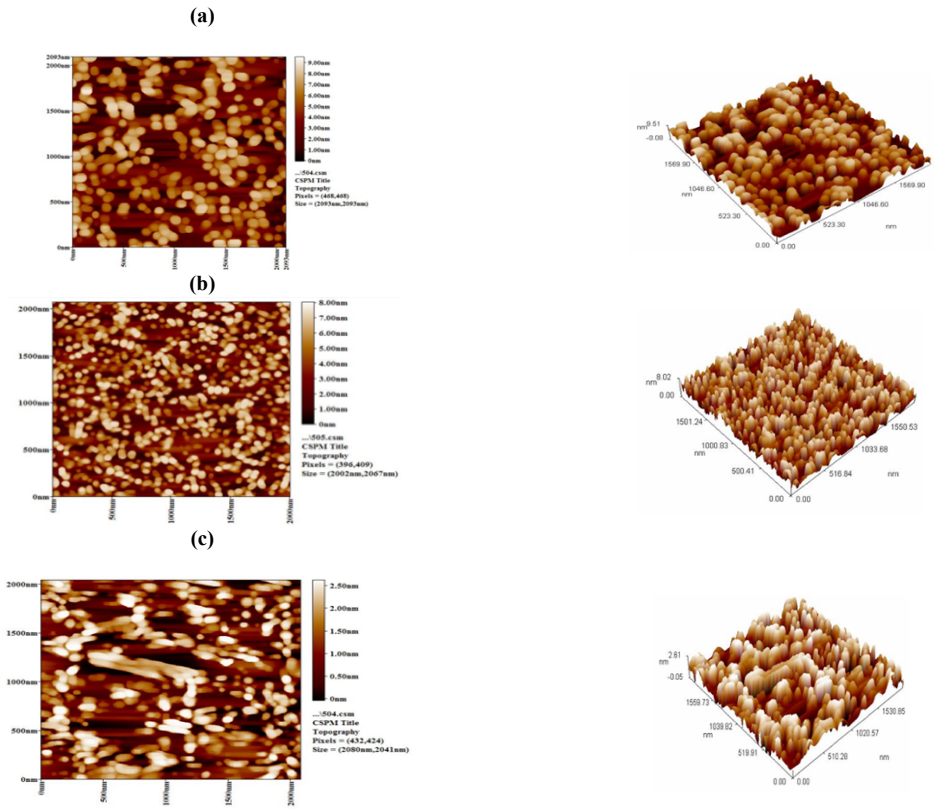


Fig. 2: AFM 2D and 3D images of: (a) pure ZnO, (b) Co doped ZnO and (c) Ni doped ZnO.

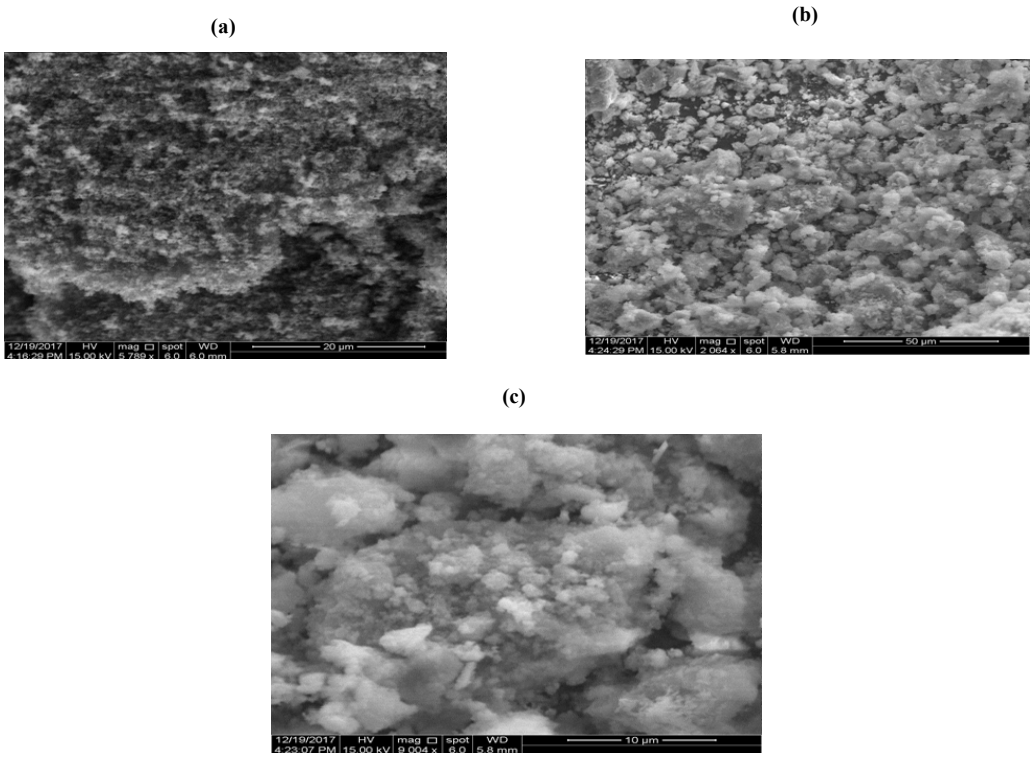


Fig. 3: Scanning electron microscope image of (a) UndopedZnO, (b) CO doped ZnO, and (c) Ni doped ZnOnanoparticles

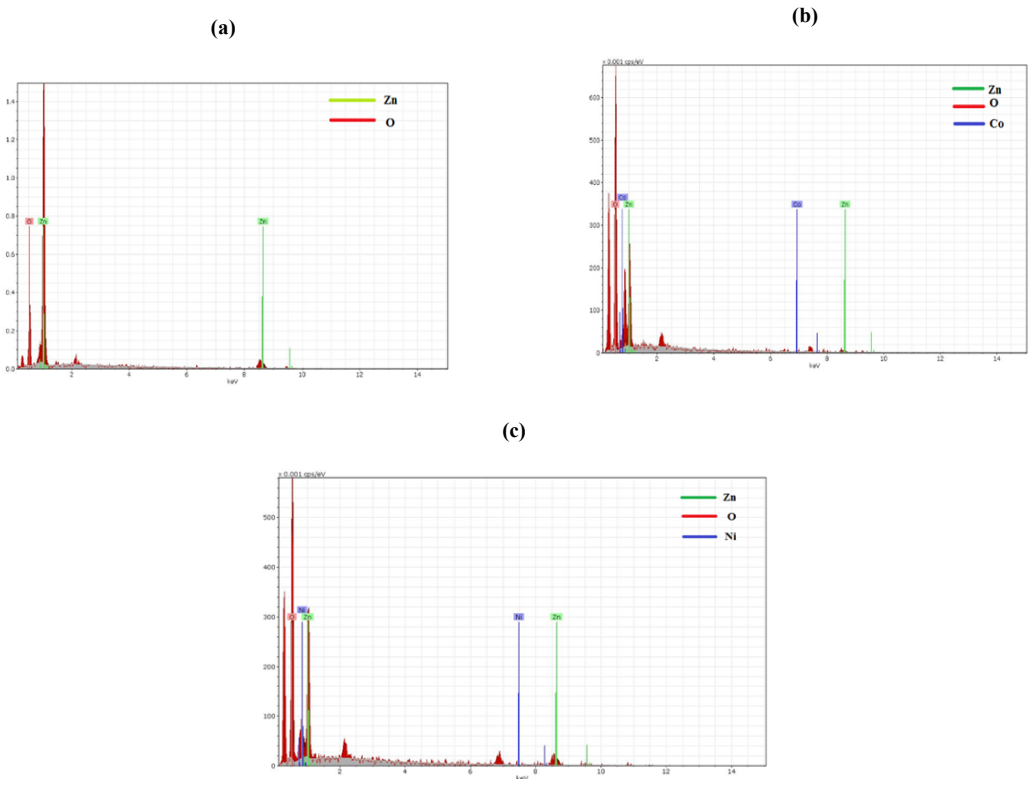
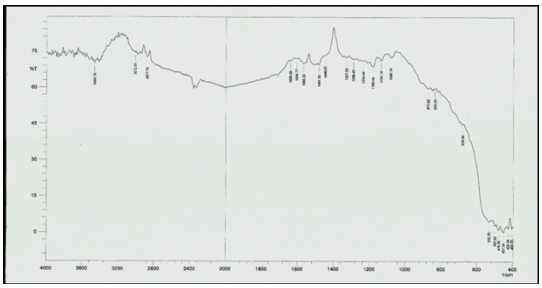
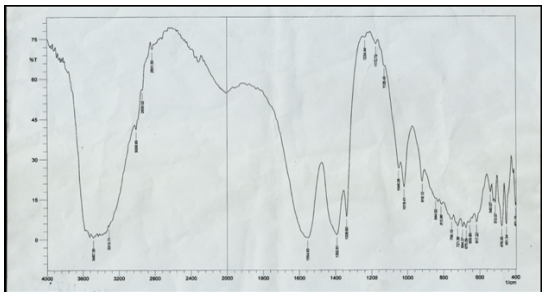


Fig. 4: Energy dispersive X-ray spectroscopy Spectrum of (a) UndopedZnO, (b) Co-doped ZnO, and (c) Ni-doped ZnO

(a)



(b)



(c)

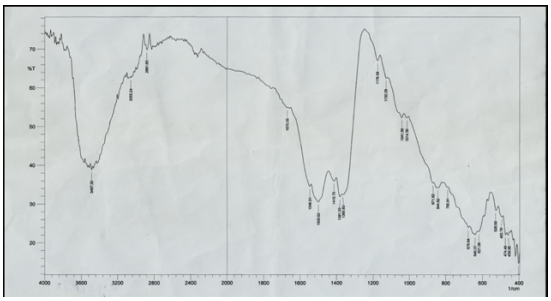
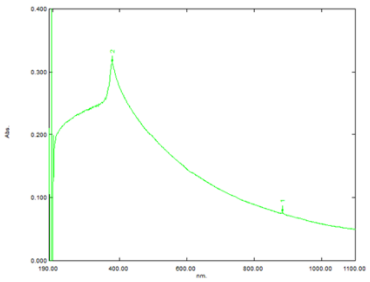
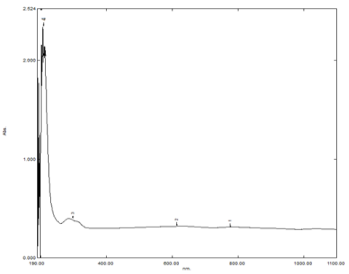


Fig. 5: Fourier Transform Infrared spectrum of (a) UndopedZnO, (b) Co doped ZnO, and (c) Ni doped ZnO nanoparticles.

(a)



(b)



(c)

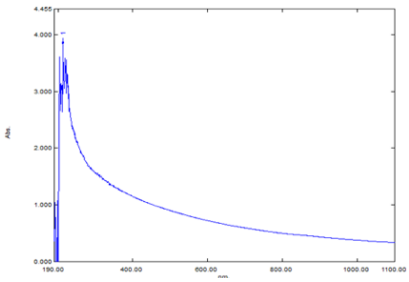
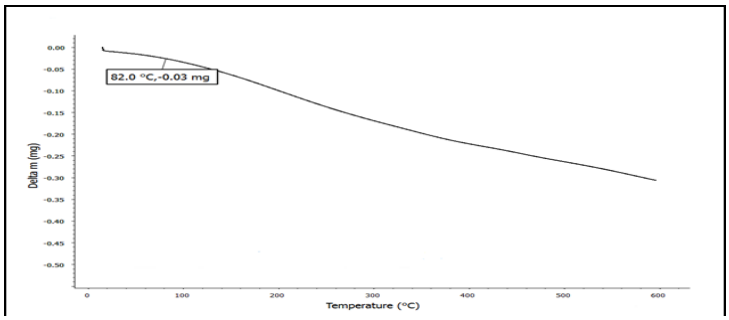
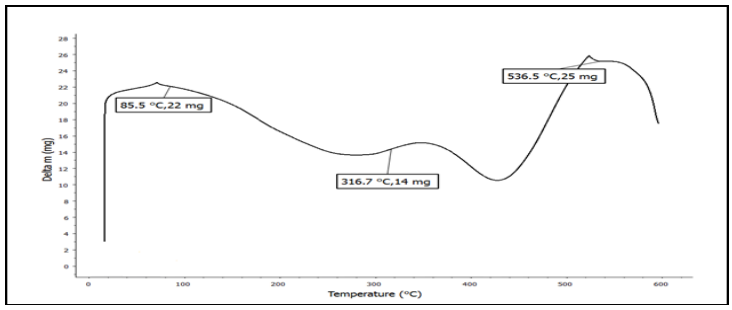


Fig. 6: Electronic spectra of the (a) undoped, (b) Co doped, and (c) Ni dopedZnO nanoparticles

(a)



(b)



(c)

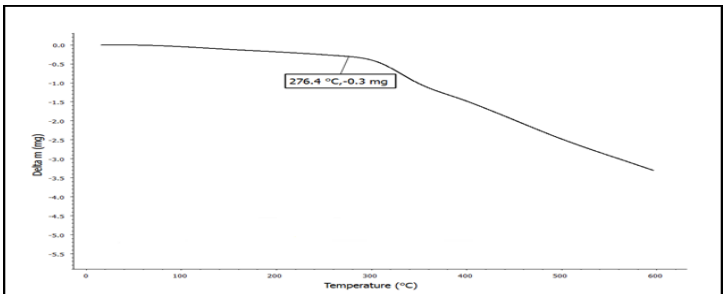


Fig. 7: The TGA curve of the prepared (a) undoped, (b) Co doped, and (c) Ni dopedZnO nanoparticles .

Table 1: The lattice constants as obtained from Rietveld refinement and particle size as deduced from X-ray line broadening.

Nanoparticles	Lattice Plane	2θ (Deg)	FWHM (Radian)	d(h k l) (A°)	Grain size (nm)
ZnO	101	36.28	0.21710	2.47356	13
Co-Doped ZnO	101	33.6991	0.76000	2.65749	3
Ni-Doped ZnO	101	33.7441	1.43000	2.65405	2

Table 2: The atomic percentage of elements in undoped ZnO, Co-doped and Ni-doped ZnO nanoparticles.

Element	Atomic Percentage (%)		
	ZnO	Co-Doped ZnO	Ni- Doped ZnO
Zn	76.87	33.50	39.18
O	23.13	64.43	58.50
Co	-	2.01	-
Ni	-	-	2.31

Table 3: Evaluation of the antimicrobial activity of the undoped, Co, and Ni doped ZnO nanoparticles, and determination of the IZ (inhibition zone) in mm.

Compound	Diameter of inhibition zone growth (mm/mg sample)	
	Candida albicans	Candida tropicalis
ZnO	9	8
ZnO: Co^{+2}	15	13
ZnO: Ni^{+2}	11	10

Scanning Electron Microscopy (SEM)

The morphology of the prepared nanoparticles was investigated by scanning electron microscope (SEM) as shown in the Figure 3 (a,b, and c) . In has been noticed from the SEM micrographs that the ZnO nanoparticles (Figure 3a) have a spherical nature which is in agreement with the shape of the surface plasmon resonance band of the UV–Vis spectra . While, Figure (3b) and Figure (3c) present SEM micrograph of cobalt and nickel doped ZnO and can clearly be seen that the sampls are composed of nanoparticles are uniformly distributed over the entire surface. The morphology of Co and Ni doped ZnO nanoparticles completely differ from that of undoped ZnO nanoparticles. This may be assigned to the effect of doping in ZnO nanoparticle

Energy dispersive X-ray spectroscopy (EDS) analysis

The EDS measurements were performed to emphasize the presence of cobalt and nickel ions in the prepared nanocrystalline ZnO particles, Figure 4 (a) shows the EDS spectrum proven that the nanoparticles suspension contains nothing but zinc and oxygen. The elemental composition of ZnO nanoparticles with two major peaks was observed to have an atomic percentage of 70.87 of Zinc and 29.13 of oxygen. Figure 4 (b) shows the EDS spectra of cobalt doped ZnO nanoparticles, and this spectrum confirmed the presence of zinc, oxygen , and cobalt. It was found that Co-doped zinc oxide nanoparticles have an atomic percentage of 13.73 of Zinc, 70.27 of oxygen, and 7 of cobalt. Figure 4 (c). Shows the atomic percentage of 36.61 of Zinc, 56.81 of oxygen and 6.58 of Nickel. This indicated the doping of Ni ion into the ZnO lattice.

Fourier transform infrared (FTIR) spectroscopy

Fourier Transform Infrared analyzed were performed in order to study the surface changes on the particles, as well as to determine the presence of functional groups and chemical bonding. These analyses can reveal the quality or consistency of samples. Figure 5 (a), 6 (b), and5 (c) show the FTIR spectrum of undoped and doped ZnO with Co^{+2} and Ni^{+2}. The spectrum of the samples, show a band observed at (3350-3450) cm-1 assigned to the *v*-OH which refers to the presence of H2O. Whereas, the presence of a band in the range 1624-1635 cm^{-1} is due to bending vibration of adsorbed H_2O molecules [19]. Hence, the presence of an OH band in the spectrum is owing to chemically and physically adsorbed water molecule on the surface of nanoparticles [20]. The band that observed at 1338 cm^{-1} and 1546 cm^{-1} is assigned to the C=O asymmetric and symmetric vibration mode respectively, probably from the un-reacted acetates [21]. The absorption band in the range 400-678cm^{-1} are mainly ascribed to ZnO stretching mode[22].These bands undergo slight shifting.

When Co doped in ZnO indicates the incorporation of Co dope in the lattice structure of ZnO [22]. The bands located near 790 and 844 cm^{-1} are assigned to the vibrations of ZnO-Ni local bonds and defect states respectively [23].

UV-Vis spectroscopy

Electronic spectra of the prepared nano compounds were dispersed individually in methanol with 0.1% wt to form a monodisperse solution. After that, each nanoparticle was sonicated for 30min Figure 6(a) shown the electronic spectra of ZnO nanoparticles revealed one band at 378nm emerge from the electronic transition from valence band to the conduction band (O2p → Zn3d) [24,25], as shown in Figure 6(a)). The Co-doped ZnO, (see Figure 6(b)) shows the presence of three bands attributed to d-d transition in the region 514,625, and 794 nm which were assigned to $^4A_2(F) \rightarrow {}^2A_1(G)$ and $^4A_2 \rightarrow {}^4T_1(P)$ respectively.

These minima are in agreement with the Co^{+2} d-d (tetrahedral symmetry) [26]. For Ni-doped ZnO case the absorption band is shifted to a lower wavelength at 214 nm. " These electronic transitions indicate that Ni^{2+} ions are incorporated Zn^{2+} ions in the octahedral ZnO. Usually, divalent Ni ions in an octahedral symmetry show three main bands at about 8300, 14080, and 24995 cm^{-1}. These bands are commonly attributed due to the spin-allowed triplet-triplet transitions" [27]:

$^3A_2g(F) \rightarrow {}^3T_2g(F)$, $^3A_2g(F) \rightarrow {}^3T_1g(F)$ and $^3A_2g(F) \rightarrow {}^3T_1g(P)$.

The obtained data detect that the single observed absorption band is most probably due to $^3A_2g\ (F) \rightarrow {}^3T_1g\ (P)$. Electronic transitions occur between split d levels of the central atom giving rise to so-called d-d transition or ligand field spectra. Where this electronic transition attributed to d–d transition is localized at (26525 cm^{-1}) [28]. The band gap energies are calculated using the equation of the reference"[29]:

$$Eg = 1240 / \lambda \text{ max} \qquad (2)$$

Where Eg: is the band gap energy, the value of (1240) is the Hz, and λ max is the wavelength in nanometer. The calculated values are 3.27 eV (378 nm), 3.17 eV (390nm) , and 3.13 eV (396 nm) for undoped ZnO , Co and Ni- doped ZnO respectively. These values are obviously red-shifted. This shift might be due to the sp-d exchange interaction between the band electron and the localized d-electron of the Co^{+2} ion and Ni^{+2}ion.

Thermal analysis

The thermogravimetric analysis (TGA) carried out for further confirmation of cobalt and nickel doped ZnO nanoparticles. Figure 7 (a) shows the TG curve of ZnO which was heated from room temperature to 600 ℃ at 10 ℃/min under a nitrogen atmosphere. TGA curve shows that the decomposition started at in the temperature range of 250–450℃, where a water molecule (found 0.03%) was lost. The temperature was performed in TGA of ZnO nanoparticles up to 400 ℃. It indicates there is no change in the structure and indicates the formation of nanocrystalline ZnO, as confirmed by XRD analysis. For cobalt doped ZnO, the result of the TG curve, Figure 7 (b) shows that the weight loss for Co-doped sample decomposition stated at 85.5℃ and was completed at 536.5℃. Whereas, the weight loss for Ni-doped samples at 276.4 ℃ is assigned to the release of absorbed water on the surface.

Biological activity

Many studies have concluded that the zinc oxide nanoparticles an effective antimicrobial against pathogenic microorganisms [30,31]. In addition, the antifungal activity of ZnO nanoparticles against Botrytis cinerea and Penicillium expansum has been found in the same studies of the references[30, 31]. "They proposed that the cell functions of Botrytis cinerea were influenced by zinc oxide nanoparticles due to increased produce of nucleic acids through the stress response in fungal hyphae leads to the death of cells [32]. Whereas the treat cells of Penicillium expansum by ZnO nanoparticles, the damaged cell membrane results in the decreased amount of proteins, carbohydrates and lipids in fungal cells lead to death of the cells" [33].

The antimicrobial activity results for the synthesis of nanoparticles are shown in Table.3.The undoped ZnO showed moderately antimicrobial activity against both kinds of fungal i.e.

Candida albicans and Candida tropicalis. The Ni doped Zno nanoparticles show a slight effect compared with undoped ZnO in all organisms. While, Co doped ZnO nanoparticles reveal the maximum antifungal activity against all organisms that have been used in this study, and recorded high inhibition activity against Candida tropicalis comparable with undoped ZnO and Ni-doped ZnO. It is clear, from the experimental results, that the transition metal doping in ZnO nanoparticles shows higher antifungal activities as compared to undoped ZnO. In general, good agreement between our results and Sankara et al results 33 results, regarding to enhance the antimicrobial activity. Thus, It can be said that in this study, transition metal doped ZnO nanoparticles have shown the best antifungal behavior compared to undoped ZnO nanoparticles.

CONCLUSION

Undoped, Co doped, and Ni doped ZnO nanoparticles were synthesized by chemical co-precipitation method and the prepared nanoparticles are characterized by XRD, AFM, SEM, EDX, UV-vis, FTIR spectrophotometer. The zone of inhibition was observed against pathogenic fungal strains and suggests that ZnO: $Co^{+2}$and ZnO: Ni^{+2} showed good antifungal activity adjacent to Candida albicans and Candida tropicalis than the undoped ZnO nanoparticles.

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