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RESEARCH PAPER

Optical Constants and Dispersion Parameters of $(NiO)_{1-x}(Co_3O_4)_x$ Nano Composite Thin Films Prepared by Chemical Spray Pyrolysis

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ABSTRACT

A 0.05 M $NiCl_2.6H_2O$ and 0.05 M $CoCl_2.6H_2O$ dissolved in distilled water to make it ready for next step of our preparation were used to produce $(NiO)_{1-x}$ $(Co_3O_4)_x$ nano composite thin films on a glass substrate using a spray pyrolysis process. With increasing Co_3O_4 concentration, the reflectivity, real and imaginary dielectric constants, and refractive index all increased. This may be due to the homogeneity and surface roughness of the films, a result that is beneficial for gas sensor applications. The energy difference decreased from (3.75 to 2.1) eV. With increasing Co_3O_4 concentration in the $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films, the dispersion coefficients decreased also. Energy dispersive X-ray spectroscopy (EDX), the analysis revealed the prominent presence of the components Ni, O, and Co. The field emission scanning electron microscopy (FESEM) confirmed our prepared surface it is agglomerate and nanosurface, It can be noted thin films have a common surface shape comprising many randomly placed chunks or aggregates of Co_3O_4 on the top surface of the films.

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INTRODUCTION

A material's optical characteristics define its relationship to light. Reflectance, absorption, dispersion transmittance, and refractive index are among the material's optical characteristics [1].

With a large band gap (3.6-4.0) eV [2], ferromagnetic characteristics, a working temperature of 523 K, and a p-type semiconductor that has demonstrated remarkable chemical stability, nickel oxide is a green crystalline solid. It has been the subject of numerous scientific studies due to its unique electrical, magnetic, and optical characteristics.

It was an interesting research material because of its excellent ion storing qualities and low cost. NiO nanostructures, for example, are p-type semiconductors with peculiar electric and magnetic characteristics that change depending on the particle size [3].

Cobalt oxide is one of the most researched oxide materials because of its uses in many different technological fields, including the absorber layer in solar cells [4], dye for ceramics and glass [5], electrode for electrochemical devices [6], gas sensors [7,8], super capacitors [9], smart glasses, and electrochemical devices [9,10]. The three distinctive crystalline phases of cobalt oxide are

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cobalt(II) oxide (CoO), cobalt(III) oxide (Co_2O_3), and cobalt(II,III) oxide (Co_3O_4). Since cobalt can absorb oxygen (O) even at room temperature, high temperatures (about 1173 K) are required to make pure cobalt oxide. Co_2O_3 completely transforms into Co_3O_4 without changing its lattice structure when sufficient oxygen is collected at temperatures higher than 573 K [11–13]. Thin films of the composite $(NiO)_{1-x}(Co_3O_4)_x$ were prepared in this investigation using the spray pyrolysis process.

MATERIALS AND METHODS

Spray pyrolysis was used to create the $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films on a glass substrate. The spraying solution was prepared from 0.05 M of $NiCl_2.6H_2O$ and 0.05 M of $CoCl_2.6H_2O$ dissolved in 100 ml distilled water. $(NiO)_{1-x}$ $(Co_3O_4)_x$ composite thin films prepared with various concentration (0, 25, 50,75,100)%,Fig. 1 shows solutions with different volume ratios (A-NiO pure, B- $(NiO)_{75}$ $(Co_3O_4)_{25}$,C- $(NiO)_{50}$ $(Co_3O_4)_{50}$, D- $(NiO)_{25}$ $(Co_3O_4)_{75}$

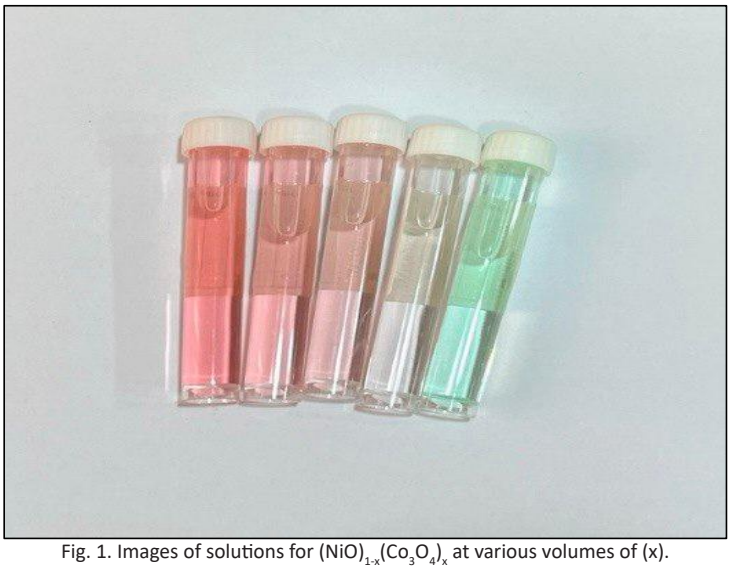

Fig. 1. Images of solutions for $(NiO)_{1-x}(Co_3O_4)_x$ at various volumes of (x).

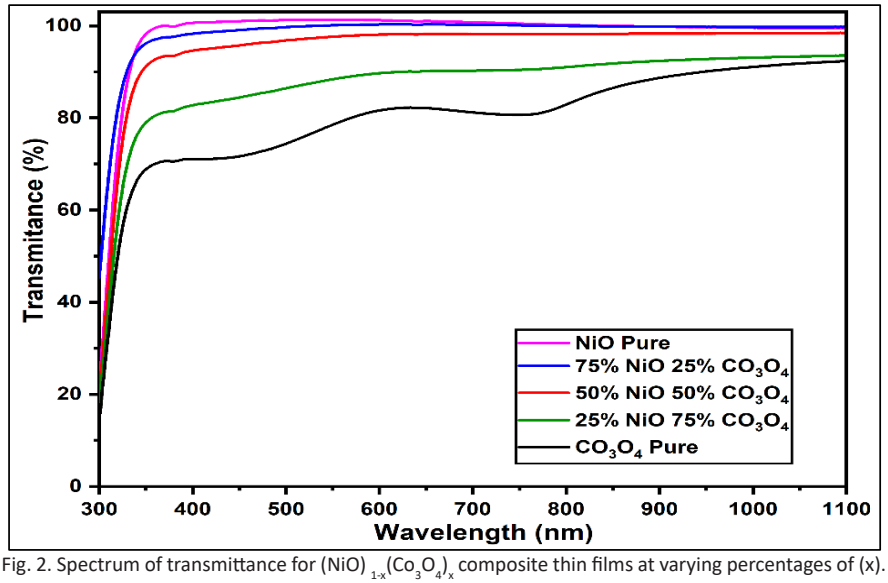

Fig. 2. Spectrum of transmittance for $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films at varying percentages of (x).

and E- Co_3O_4 pure) depending on the value x in $(NiO)_{1-x}(Co_3O_4)_x$.

The deposition parameters, including the stopping time of two minutes, the deposition rate of two milliliters per minute, and the space between the nozzle and the substrate of thirty centimeters, were maintained at optimal values. A UV-visible spectrophotometer is used to calculate certain optical parameters and record absorption spectra in the (300–1100) nm range.

RESULTS AND DISCUSSION

Optical Constants

Fig. 2 displays the $(NiO)_{1-x}(Co_3O_4)_x$ composite thin-film optical transmittance spectra in the wavelength range of 300–1100 nm. For all deposited thin films, it was found that the transmittance increase as the wavelength increased, while transmittance decreased with increasing the concentration of the Co_3O_4, which led to a decrease in the energy gap

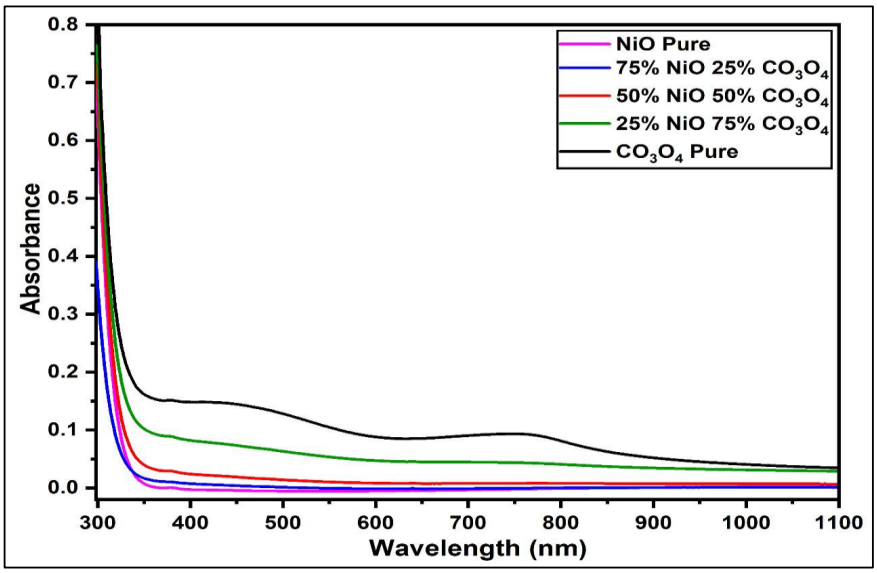

Fig. 3. absorbance spectrum for composite thin films of $(NiO)_{1-x}(Co_3O_4)_x$ at varying volumes of (x).

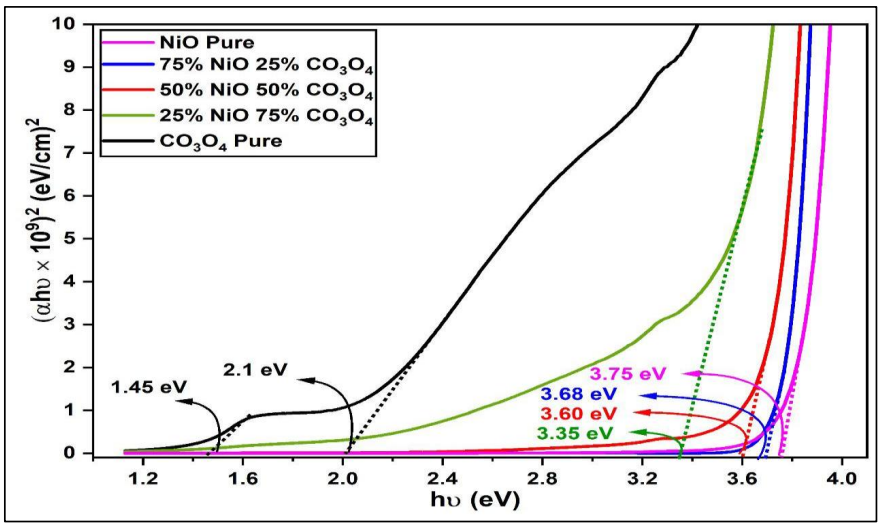

Fig. 4. Images of the energy gap for $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films at varying volumes of (x).

The values for the prepared films as in Fig. 2 are a result of the rise in the produced films' thickness as the proportion of Co_3O_4 rises, which is in line with the findings of the study. [14].

Fig. 3 shows a strong absorption that subsequently dropped as the wavelength increased. In contrast, transmittance and reflectance exhibit the reverse tendency. It is evident that the films exhibited a greater capacity to absorb electromagnetic radiation at longer wavelengths than NiO as the concentration of Co_3O_4 increased. This suggests that Co_3O_4 can decrease the band gap of NiO. According to the findings, the Eg value of the unaltered NiO was between (3.6 - 4) eV [15, 16].

As the concentration of Co_3O_4 increased from 25% to 100%, the energy band gap of $(NiO)_{1-x}(Co_3O_4)_x$ thin films reduced from 3.75 to 2.1 eV, as illustrated in Fig. 4, where some of the unpaired electrons join with the unpaired electrons on

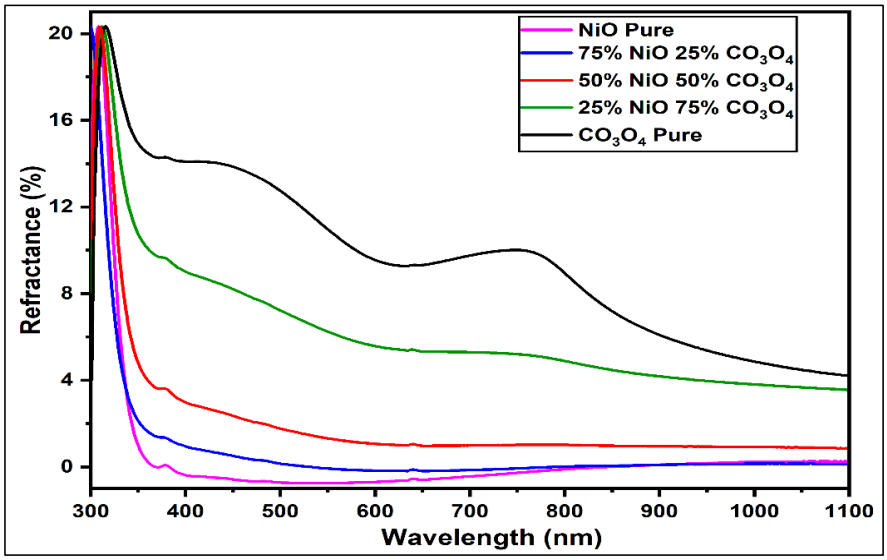

Fig. 5. Reflectance spectrum for composite thin films of $(NiO)_{1-x}(Co_3O_4)_x$ at varying volumes of (x).

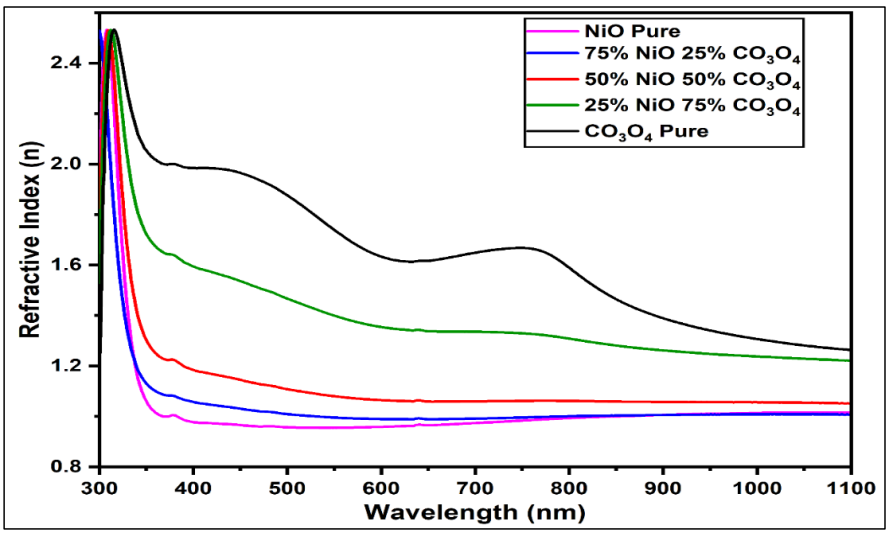

Fig. 6. Spectrum of the refractive index (n) for $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films at varying volumes of (x).

the NiO surface to form a structure. The upward displacement of the valence band edge is what causes this decrease in the band gap [17–19].

We notice from the figure that with increasing the concentration of Co_3O_4, the energy gap decreases. The rise in electronic transitions can be attributed to the doping process, which produced new levels near the valence band inside the energy gap. As a result, bridges were created that made it possible for electrons to flow between the conduction and valence bands. Consequently, the produced film's structural qualities were enhanced by the addition of Co_3O_4.The electron was unable to vaporize and travel from the valence pack to the conduction beam because the energy of the falling photon was less than the semiconductor's energy value. Consequently, raising the wavelength lowers the absorption. We note that the absorption increased with the concentration of the Co_3O_4, which led to a decrease in the energy gap values

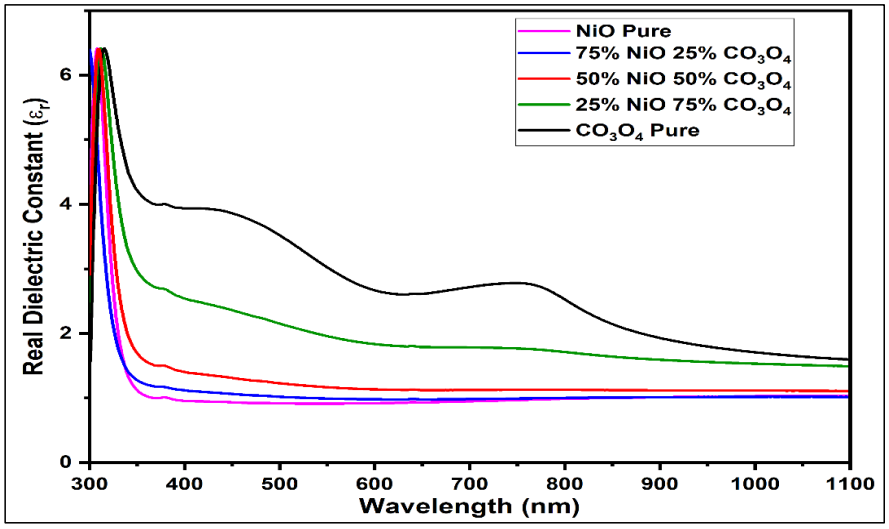

Fig. 7. The dielectric constant spectrum's real component for $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films at varying voltage percentages of (x).

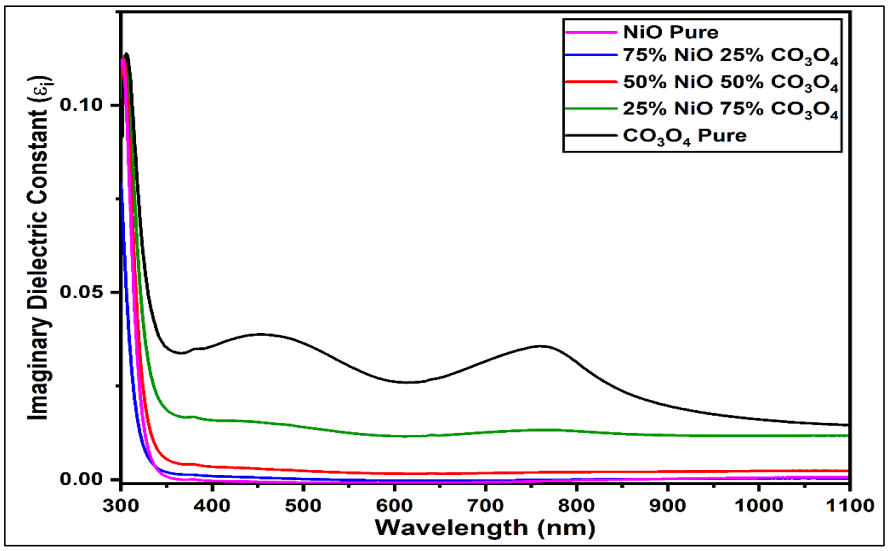

Fig. 8. The dielectric constant spectrum's imaginary component for $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films at various vol.% of (x).

of the prepared films [20,21].

Fig. 5 displays the reflectance of $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films. According to this figure, the reflectance rises as the Co_3O_4 concentration does, which is in line with the researchers' findings [22].

It is known that the refractive index is related to reflectivity, the n results of prepared films in Fig. 6 at cutoff wavelength (λc). It was also observed that the refractive index increases for Co_3O_4 ratios this is consistent with the results of the researchers [22], which may be due to the increasing homogeneity and the surface roughness of the films.

According to [23], the dielectric constant's real (ε_1) and imaginary (ε_2) components are written as follows:

$$\varepsilon_1 = n^2 - k^2 \quad (1)$$

$$\varepsilon_2 = 2nk \quad (2)$$

where (k): is the extinction coefficient and (n): is the refractive index. The imaginary part quantifies the rate of wave dissipation in the medium, whereas the real (ε_1) component is associated with dispersion. Figs. 7 and 8 show the real and imaginary dielectric constants. Based on these figures, the values of ε_1 and ε_2 increase with increasing wavelength.

Dispersion Parameters

A single-term Sellmeier relation was created by Wemple and Didomenico [24] utilizing an outstanding long-wavelength approximation:

$$n^2-1=E_d E_o / E^2_o - E^2 \quad (3)$$

where (E_o): is the single oscillator energy of the electronic transitions, (E_d): is the dispersion energy, and n is the refractive index.

A plot of $(n^2 - 1)^{-1}$ against E^2, see Fig. 9, was used to estimate Eo and Ed which were calculated from the slope $(E_oE_d)^{-1}$ and intercept (E_o/E_d). The

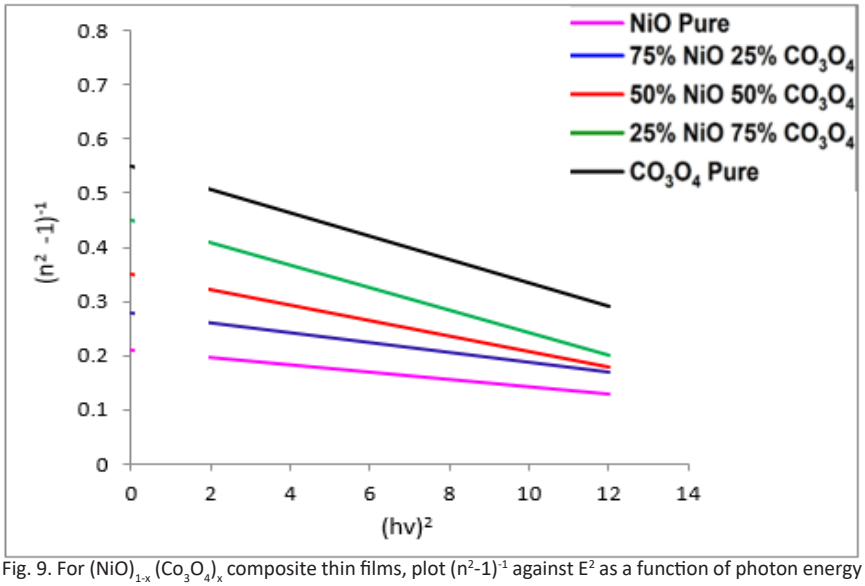

Fig. 9. For $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films, plot $(n^2-1)^{-1}$ against E^2 as a function of photon energy squared at varying vol.% of (x).

Table 1. the optical characteristics of composite thin films of $(NiO)_{1-x}(Co_3O_4)_x$ at varying volumes of (x).

Samples	E_d (eV)	E_o (eV)	E_g (eV)	ε∞	n_o	M_{-1}	$M_{-3}(eV)^{-2}$
NiO Pure	26.726	5.612	3.75	5.76	2.4	4.761	0.151
$NiO_{75}Co_3O_{4\,25}$	19.738	5.526	3.68	4.57	2.1	3.571	0.116
$NiO_{50}Co_3O_{4\,50}$	14.201	4.970	3.60	3.85	1.9	2.857	0.115
$NiO_{25}Co_3O_{4\,75}$	10.327	4.647	3.35	3.22	1.7	2.222	0.102
Co_3O_4 Pure	9.160	5.038	2.1	2.82	1.6	1.818	0.071

calculated values, which were displayed in Table (1), decreased as the concentration of Co_3O_4 increased. The optical energy gap value derived from the Tauc relation was in agreement with the Wemple-DiDomenico energy gap estimate [25]. (n_o) The static refractive index and the static dielectric constant can be obtained from the following relationships[26]:

$$n^2(o) = 1+ E_d/E_o \quad (4)$$

$$\varepsilon_\infty = n^2)o) \quad (5)$$

The calculated values were listed in the Table (1) showing a decrease in their values with increasing concentration of the Co_3O_4.

The relationships listed below [27, 28] The (M_{-1}) and (M_{-3}) moments of the imaginary part of the optical spectrum of $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films may be found using it:

$$E^2_o = M_{-1}/M_{-3} \quad (6)$$

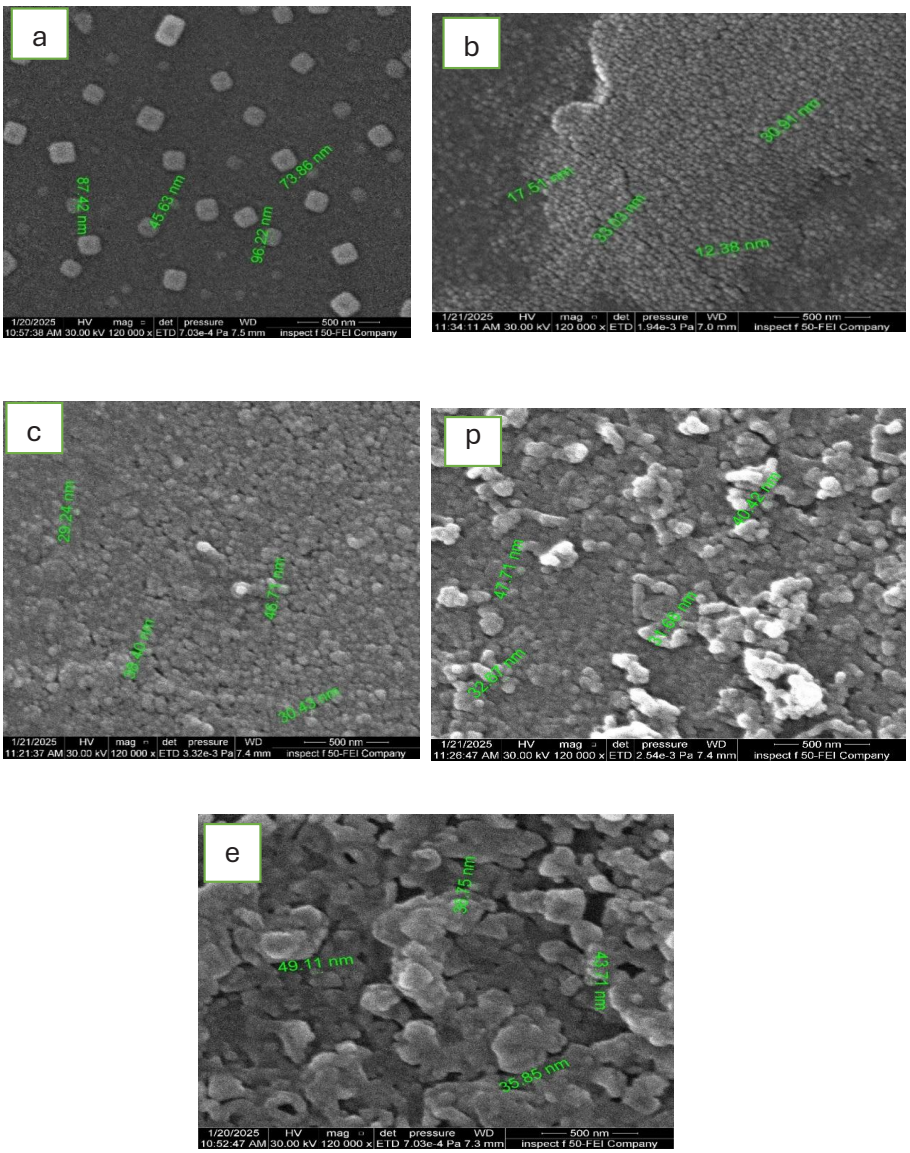

Fig. 10: FESEM pictures of composite thin films of $(NiO)_{1-x}(Co_3O_4)_x$ at varying volumes of (x).

$$E^2_d = M^3_{-1}/M_{-3} \quad (7)$$

Table 1 indicates that the optical spectrum moments decrease when the concentration of Co_3O_4 thin films increases.

Structural Properties

Field Emission Scanning Electron Microscopy (FESEM)

FESEM images of $(NiO)_{1-x}(Co_3O_4)_x$ thin films at various Co_3O_4 volume ratios (0,25,50,75, and100)%

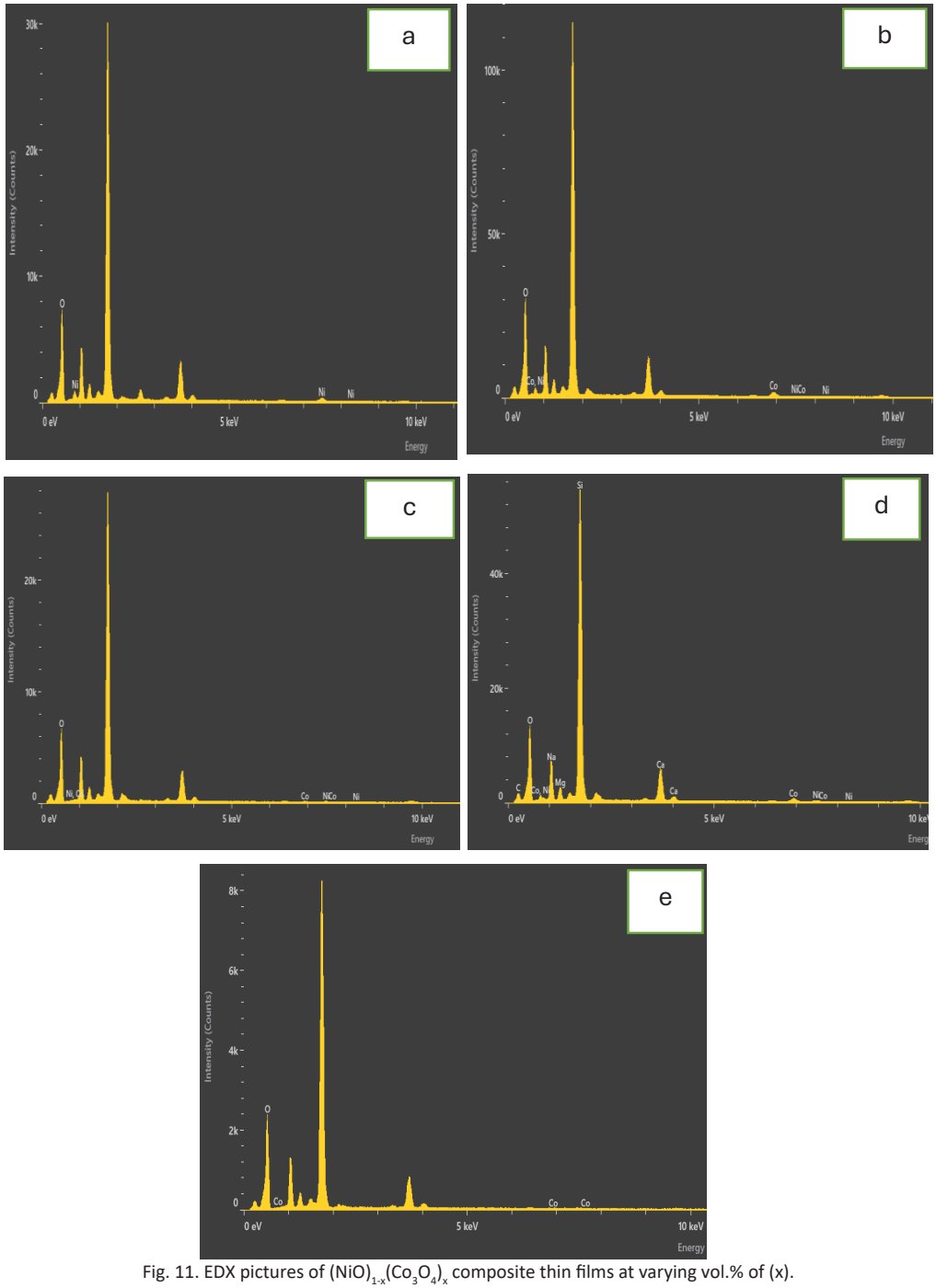

Fig. 11. EDX pictures of $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films at varying vol.% of (x).

Table 2. EDX analysis for prepared films elements compositions.

Samples	Element	Weight %	Atomic %
NiO Pure	O	90.3	97.2
	Ni	9.7	2.8
$NiO_{75}Co_3O_{4\ 25}$	O	87.4	96.2
	Co	11.1	3.3
	Ni	1.6	0.5
$NiO_{50}Co_3O_{4\ 50}$	O	94.5	98.4
	Co	2.1	0.6
	Ni	3.4	1.0
$NiO_{25}Co_3O_{4\ 75}$	O	86.5	95.9
	Co	9.4	2.8
	Ni	4.2	1.3
Co_3O_4 Pure	O	99.0	99.7
	Co	1.0	0.3

are displayed in Fig. 10.(a-e). As can be seen, thin films share a common surface form with several Co_3O_4 aggregates or chunks arranged haphazardly on the top surface. The image displays a cubic nanocrystal. The results indicate that Co_3O_4 had a propensity to cluster and disperse effectively in the $(NiO)_{1-x}(Co_3O_4)_x$ thin films. Cobalt oxide developed a continuous network with $(NiO)_{1-x}(Co_3O_4)_x$ as its concentration rose. The findings presented herein are consistent with the conclusions reached [29]. From the Fig. 10, we notice that the size of the particles in the membranes is nano-sized, which means that the membranes can be used as a sensing application because the nano-sized size provides a high adsorption area, and this is one of the conditions for high sensitivity, this is consistent with the work of researchers. [30]

Energy Dispersive X-ray Spectroscopy (EDX)

Fig. 11 (a-e) shows the energy-dispersive X-ray analysis (EDX) spectra of the $(NiO)_{1-x}(Co_3O_4)_x$ thin films that were formed on a glass substrate. Fig. 4 shows the $(NiO)_{1-x}(Co_3O_4)_x$ thin films' energy-dispersive X-ray analysis (EDX) spectrum for various Co_3O_4 concentrations (0,25,50,75, and 100)% that were deposited on a glass substrate using the spray pyrolysis approach. The components Ni, O, and Co were found to be highly prevalent. Additionally, as seen in Table 2.

CONCLUSION

Using 0.05 M of $NiCl_2.6H_2O$ and 0.05 M of $CoCl_2.6H_2O$ diluted in distilled water, the chemical spray pyrolysis approach has successfully produced the $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films. As the concentration of Co_3O_4 grows, the reflectance, real and imaginary dielectric constants, and refractive index all rise. This could be because the films become more homogeneous and rough on the surface, which makes them appropriate for sensing applications. As the concentration of Co_3O_4 in the $(NiO)_{1-x}(Co_3O_4)_x$ composite thin films increases, dispersion parameters decrease also. Energy dispersive X-ray spectroscopy (EDX), the analysis revealed the prominent presence of the components Ni, O, and Co, field emission scanning electron microscopy (FESEM), It can be noted thin films have a common surface shape comprising many randomly placed chunks or aggregates of Co_3O_4 on the top surface of the films.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this

manuscript.

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