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Effect of Annealing Temperature on the Optical Properties of Cobalt Oxide Thin Films

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Abstract
A spectrometric technique, UV-Vis spectroscopy was used to measure the optical properties of cobalt oxide (Co_3O_4) thin films synthesized via the sol-gel spin coating technique. Thin films were annealed at temperatures ranging from 250 °C to 400 °C and characterized by spectrophotometer for their optical absorption, transmittance, reflectance, absorption coefficient, extinction coefficient, optical band gap, refractive index, and optical dielectric constant. The results reveal that increasing the annealing temperature enhances the optical absorption while reducing both transmittance and reflectance, indicative of improved crystallinity and surface morphology. The optical band gap decreased from 2.75 eV at 250 °C to 1.80 eV at 400 °C, corresponding to a red shift in the absorption edge. A progressive increase in the refractive index and optical dielectric constant was also observed, suggesting a denser and more compact film structure at higher temperatures. These results demonstrate that thermal treatment plays a crucial role in tailoring the optical performance of cobalt oxide thin films. With increasing annealing temperature, absorption intensity and absorption coefficient increased, while transmittance and reflectance decreased, indicating enhanced structural uniformity and reduced surface scattering. These findings highlight the potential of thermally treated cobalt oxide thin films for use in optoelectronic, photovoltaic, sensor, and solar energy conversion.

Keywords
Cobalt oxide thin films, Annealing temperature, Optical properties, UV-V is spectroscopy, Sol-gel spin coating, Optical band gap

Introduction

The study investigates the effect of annealing temperature on the optical properties of cobalt oxide (Co_3O_4) thin films. Although cobalt oxide thin films have promising applications in optoelectronic devices, solar cells, sensors, and other optical technologies, there is limited understanding of how annealing temperature influences important optical characteristics such as absorbance, transmittance, refractive index, and energy band gap. This knowledge gap restricts the ability to optimize the films for specific technological applications.

The main objective of the study is to investigate the effect of annealing temperature on the optical properties of cobalt oxide thin films using spectrometric techniques. To achieve this, cobalt oxide thin films were synthesized using the sol-gel spin coating method, annealed at different temperatures (250–400 °C), and characterized using UV-Vis spectroscopy to determine their absorbance, transmittance, reflectance, refractive index, optical dielectric constant, and optical band gap.

The significance of the study lies in providing valuable insights into how annealing temperature affects the structural and optical performance of cobalt oxide thin films. The findings contribute to the optimization of thin-film fabrication processes, demonstrate the effectiveness of UV-Vis spectroscopy for optical characterization, and support the development of improved materials for optoelectronic, photovoltaic, sensor, and solar energy conversion applications.

Cobalt oxide thin films have garnered significant attention due to their potential applications in various fields, including solar cells (Shinde et al., 2018), electrochromic devices (Granqvist, 2014), and catalysts (Wang et al., 2019). The optical properties of these films play a crucial role in determining their performance in these applications.

Annealing is a critical step in the fabrication of cobalt oxide thin films, as it can significantly impact their structural, electrical, and optical properties. Previous studies have shown that annealing can improve the crystallinity and grain size of cobalt oxide thin films, leading to enhanced optical properties.

Spectrometric techniques, such as UV-Vis spectroscopy, spectroscopic ellipsometry and Fourier transform infrared spectroscopy are powerful tools for characterizing the optical properties of thin films (Tompkins & Irene, 2005). These techniques can provide valuable insights into the optical constants, such as refractive index and extinction coefficient, of cobalt oxide thin films.

Literature Review

The spectrometric measurement of optical properties in cobalt oxide thin films has been extensively studied using various techniques, including spectroscopic ellipsometry, UV-Vis-NIR spectroscopy, and Raman spectroscopy. Factors such as annealing temperature, doping, and deposition methods significantly influence the optical characteristics, including band gap energies, refractive indices, and absorption coefficients. These studies provide valuable insights for optimizing cobalt oxide thin films for applications in photovoltaics, sensors, and electrochromic devices. This review summarizes key studies that have utilized various spectrometric methods to characterize these materials.

Gallant et al. (2006) explored the optical and physical properties of cobalt oxide films electrogenerated in bicarbonate aqueous media. They utilized spectroscopic ellipsometry and UV-Vis-NIR reflectance to determine film thicknesses ranging from 31 to 290 nm, with refractive indices between 1.8 and 2.2 at 632.8 nm. The study confirmed a bilayered structure comprising a Co_3O_4 outer layer and a CoO inner layer, with the amorphous nature corroborated by Raman spectroscopy and X-ray diffraction analysis.

Similarly, a study published in Solar Energy Materials and Solar Cells investigated cobalt oxide thin films deposited by the sol–gel method. Spectroscopic ellipsometry, along with atomic force microscopy and X-ray photoelectron spectroscopy, was employed to analyze the optical properties as a function of dipping time. The optical constants (n and k) were reported in the 200–800 nm range, and the films exhibited a multilayer stack with voids, with thickness and void/Co_3O_4 percentage ratios strongly dependent on immersion time (Barrera-Calva et al.,2006)

Chansaengsri et al. (2016) examined the effect of annealing temperature on the structural and optical properties of cobalt oxide thin films prepared by electrostatic spray deposition. Using UV-Vis

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spectroscopy, they observed that higher annealing temperatures led to a decrease in optical band gap values, attributed to changes in crystalline size and defect sites within the films.

In another study, Zribi et al. (2023) investigated the impact of tin (Sn) doping on the physical properties of Co_3O_4 thin films. Employing UV-Vis-NIR spectroscopy and spectroscopic ellipsometry, they found that Sn doping significantly altered optical transitions, dielectric functions, and band gap energies. Doped samples exhibited higher absorption coefficients ($>10^5$ cm^{-1}) over a broad energy range, with a fundamental band gap transition around 0.77 eV.

A study published in Materials Research Innovations focused on the preparation and characterization of dip-coated cobalt oxide thin films. By varying the withdrawal speed during the sol–gel dip coating process, the researchers analyzed the optical properties using UV-Vis spectrophotometry. The films exhibited polycrystalline Co_3O_4 phases, with optical constants (n and k) evaluated in the 300–900 nm range. The study also assessed magnetic properties, noting saturation magnetization values between 16.26 and 114.30 emu/cm³.

Lakehal et al. (2018) investigated manganese-doped Co_3O_4 thin films prepared via a sol–gel dip-coating process. Using UV-Vis spectroscopy, they observed that increasing Mn concentration led to decreased transmittance and optical band gaps, suggesting the introduction of impurity energy levels. The films maintained a cubic spinel structure, as confirmed by X-ray diffraction and FTIR analyses.

Hashim et al. (2024) conducted a study on the optical properties of cobalt oxide using ultraviolet-visible spectroscopy. They prepared samples with varying molarities via the sol–gel technique and analyzed absorption, reflection, refraction, transmission, absorption coefficient, and energy gap. The energy gap was determined to be 3.782 eV at 368 nm, aligning with expected values for conductor oxides. The study highlighted the potential applications of cobalt oxide in optical reagents and solar cells.

Ilenikhena and Okeke (2005) compared cobalt oxide (CoO) and copper oxide (CuO) thin films deposited by an improved electroless chemical bath technique. They measured optical properties such as absorbance, transmittance, refractive index, and band gap using a single-beam spectrophotometer in the 200–900 nm range. CoO thin films exhibited band gaps ranging from 2.36 to 2.75 eV, suggesting suitability for applications in antireflection coatings and solar thermal control coatings..

Materials and Methods

The materials to be used in carrying out this research includes;
i.Cobalt chloride $CoCl_2$ salt
ii.Triethanolamine (TEA) (stabilizer)
iii.Methanol (solvent)
iv.De-ionize water
v.Electronic weighing
balance
vi.Magnetic stirrer
vii.Soda lime glass slide substrate
(SLG)
viii.Beakers (100 mL)
ix.Syringe and needle (mL and 5 mL)
x.Oven
xi.Furnace
xii.Vacuum spin coater (VTC-100)
xiii.UV- Vis Spectrophotometer, JENWAY, (Cole palmer Ltd , UK)

3.1.1 The spin Coater

The spin coater is a tool that uses centrifugal force to apply thin, homogeneous films to flat substrates. It is frequently used to produce thin films of several materials, including oxides, polymers, and photoresists, in the microfabrication, material science, and semiconductor sectors. Three primary steps are involved in the spin coater's operation: dispense the solution, spin the substrate, and dry or cure the film (Singh & Patidar, 2018).

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Plate 3.1: The VTC-100 vacuum Spin Coater

3.1.2.Ultraviolet visible spectrophotometer

The Ultraviolet-Visible (UV-Vis) Spectrophotometer is an analytical instrument used to measure the absorbance or transmittance of a sample in the ultraviolet (UV) and visible regions of the electromagnetic spectrum, typically in the range of 200–800 nm. It is widely used for both qualitative and quantitative analysis in chemistry, material science, biology, and other fields.

The UV-Vis spectrophotometer works on the principle that different compounds absorb light at different wavelengths. The instrument measures the amount of light absorbed by the sample as a function of wavelength, which can be used to determine various properties of the sample. The spectrophotometer has two light sources namely: Deuterium lamp: For the UV range (200–400 nm) and Tungsten-halogen lamp: For the visible range (400–800 nm)

Monochromator: The light from the source is passed through a monochromator, which separates the light into different wavelengths and directs a specific wavelength toward the sample (Skoog et al., 2017).

Sample Holder: The sample is placed in a cuvette, usually made of quartz (for UV measurements) or glass (for visible range). The cuvette contains either a liquid solution or solid material.

Detector: After passing through the sample, the remaining light reaches a detector, which measures the intensity of light that passes through (transmittance).

Plate 3.1: Ultraviolet visible spectrophotometer (Jenway . UK) 2 Method

3.2.1.Synthesis of Cobalt oxide Thin Film Preparation of precursor solutions

The precursor solutions will be prepared using analytical grade reagents of Cobalt chloride ($CoCl_2$) , methanol and TEA. 0.4 M of $CoCl_2$ will be prepared by dissolving 2.077g of cobalt chloride dissolved in 40 mL of methanol and then stirred thoroughly by magnetic stirrer at speed of 1000 rpm at a temperature of 50 °C for 10 minutes to obtain a homogenous solution. This solution will be mixed with 2 mL TEA thoroughly by a magnetic stirrer embedded in a constant temperature bathe at speed of 1000 rpm at a temperature of 50 °C for 10 minutes.

Substrate cleaning

In order to remove dirt from soda lime glass (SLG) substrate used for the film deposition, it was washed with detergent, rinsed with methanol, deionized water and dried in the oven at 100 ^{o}C for 10 minutes.

3.2.2.Deposition of Cobalt oxide thin films

1.The precursor solutions were be sucked into the syringe and then spin coated onto glass substrates at a speed of 2000 rpm for 30 seconds each.
2.After spin coating, the substrates were dried in an oven at 150 °C for 10 min to evaporate the residue solvents in the film and the same procedure was repeated 10 times to get film with a desired thickness.
3.The coated films were annealed in a muffle furnace at 250, 300, 350 and 400 °C for 1 hour

3.2.3 Measurement of optical properties of fabricated Cobalt oxide thin films.

The optical properties of the annealed cobalt oxide thin films such as absorbance and transmittance were measured using a spectrometric technique known as UV-Vis spectroscopy. This was carried out

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by UV- Vis Spectrophotometer, JENWAY, (Cole palmer Ltd, UK) at Instrumentation Laboratory, Chemistry Department Joseph Sarwuan University, Makurdi, Benue State.

To measure the absorbance of a thin film using the Jenway spectrophotometer, the following procedure was followed:

i.The film surface was cleaned with a soft cloth to remove any dust particles or fingerprints that could interfere with the measurement.

ii.The spectrophotometer was turned on and allowed to warm up for approximately 20 minutes. This warm-up period ensures the stability of the light source and accurate measurement readings.

iii.The desired wavelength range for the measurement, typically between 200 nm and 1000 nm, was set on the spectrophotometer.

iv.The instrument was set to operate in "Absorbance" mode, which is suitable for measuring the amount of light absorbed by the thin film.

v.Before measuring the absorbance of the thin film, a baseline correction was performed. This was achieved by placing a clean, transparent substrate (identical to the one used for the thin film) into the sample holder of the spectrophotometer. This blank sample is used to set the baseline (zero absorbance), eliminating the effect of the substrate's inherent absorbance on the measurement. After setting the baseline, the glass substrate is removed from the sample holder.

vi.The thin film sample was then carefully placed in the sample holder of the spectrophotometer, ensuring that the film side is directly facing the light path.

vii.The sample compartment is securely closed to prevent any external light from affecting the readings.

viii.The spectrophotometer is then used to measure the absorbance of the thin film over the selected wavelength range. As the light passes through the film, the instrument records the absorbance values at each wavelength, providing a detailed absorbance spectrum of the thin film.

x.Once the measurement was completed, the absorbance data was saved and exported for further analysis.

xi.After data collection, the thin film sample was carefully removed, and the sample holder is cleaned. Finally, the spectrophotometer is properly shut down

Results and Discussion

4.1.Presentation of Results

The results of the spectrometric measurements of optical properties carried out on the annealed cobalt oxide thin films prepared by spin coating are presented in figures 4.1, 4.2, 4.3, 4.4 and table 4.1.

4.1.Results

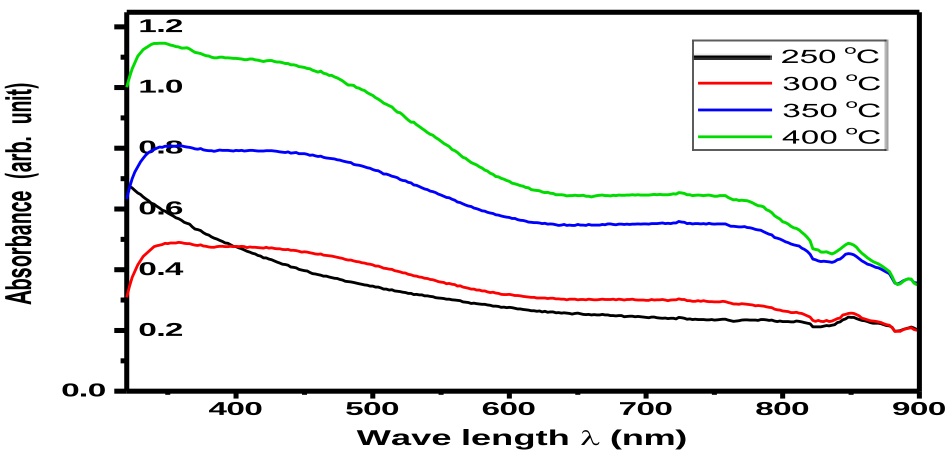

Figure 4.1: Absorbance spectra of annealed cobalt oxide thin films

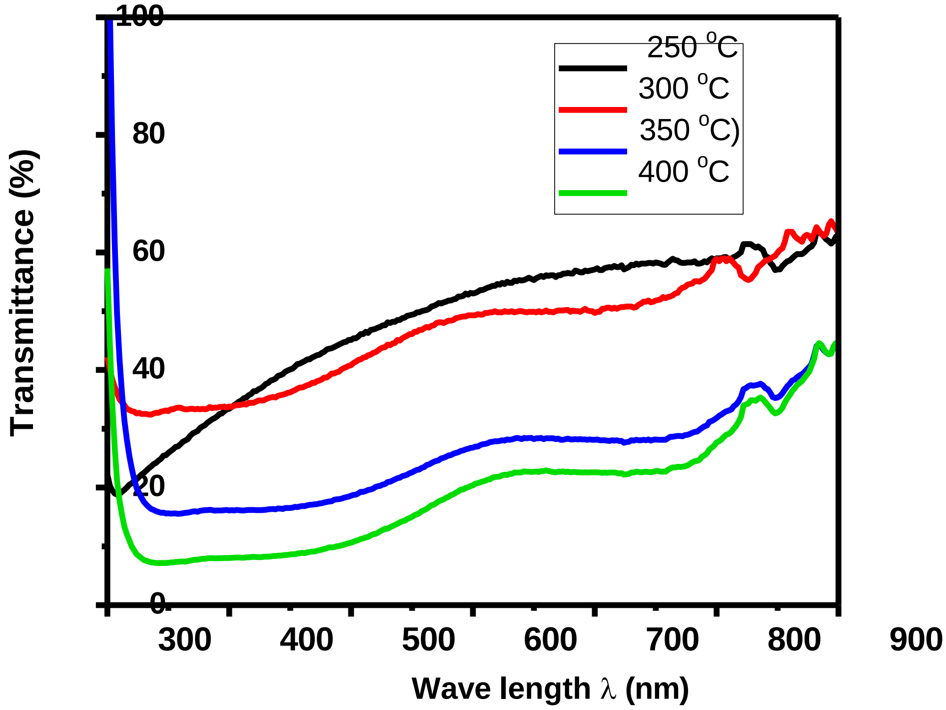

Figure 4.2: Transmittance spectra of annealed cobalt oxide thin films

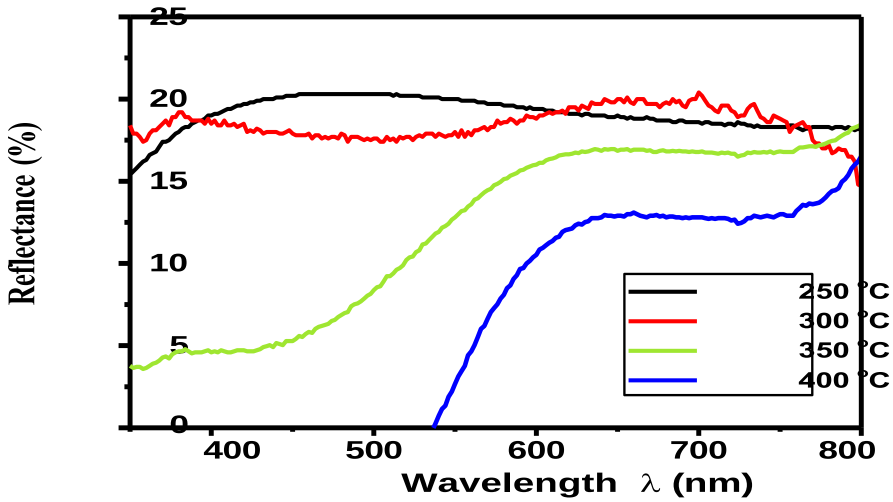

Figure 4.3: Reflectance spectra of annealed cobalt oxide thin films

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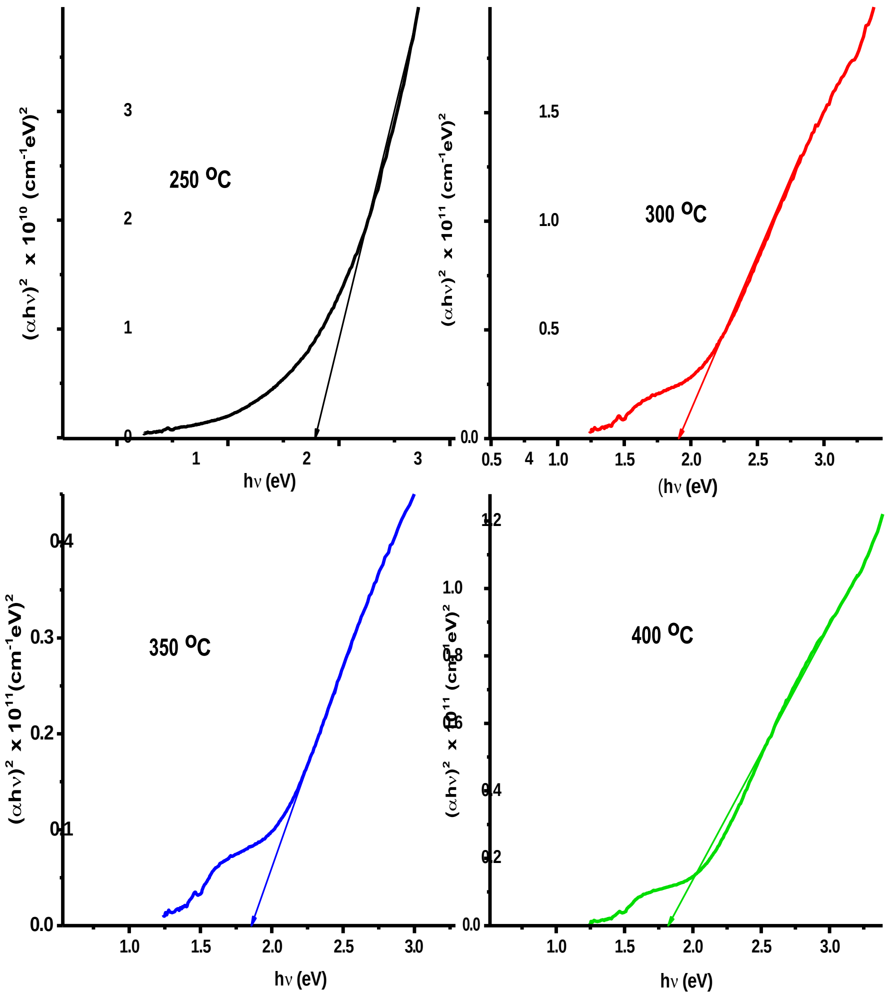

Figure 4.4: Tauc's plot for annealed cobalt oxide thin films

Table 4.1 Optical Properties of Annealed Cobalt oxide thin Film

Temp °C	optical band gap E_g (eV)	Refractive index η	Optical dielectric Constant ε_{opt}
250	2.75	2.467	5.09
300	1.89	2.789	6.78
350	1.83	2.816	6.93
400	1.8	2.828	7.00

4.2. Discussion

4.2.1. Optical Absorption, Transmittance, and Reflectance

Absorption is a fundamental optical property that determines how much light a material can absorb at different wavelengths. For cobalt oxide thin films, the absorption spectra (Figure 4.1) demonstrate that increasing the annealing temperature generally leads to an increase in absorption intensity.

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Specifically, at 250°C, the absorption is around 0.85, increasing to 0.92 at 300°C, 0.95 at 350°C, and 0.98 at 400°C. This enhancement in absorption can be attributed to improved crystallinity, which increases the density of absorbing centers and reduces scattering effects, allowing more light to be absorbed (Ali et al., 2021; Singh & Gupta, 2019).
Transmission spectra (Figure 4.2) of the annealed cobalt oxide thin films exhibit a clear trend with annealing temperature. As the annealing temperature increases within the range of 250°C to 400°C, the transmission of light through the films tends to decrease slightly. This reduction in transmission is directly related to the increase in absorption, as more light is absorbed by the film at higher temperatures (Sharma et al., 2022; Kumar et al., 2020).
The reflection spectra (Figure 4.3) also respond to changes in annealing temperature. Typically, higher annealing temperatures (250°C to 400°C) result in a moderate decrease in reflection. The reflection decreases from 15% at 250°C to 12% at 400°C. This is because the enhanced crystallinity reduces surface scattering, allowing more light to be absorbed rather than reflected (Nguyen et al., 2018). Additionally, the reduction in surface imperfections at higher temperatures contributes to lower reflectance.
nm and 800 nm. As the annealing temperature increases from 250°C to 400°C, the extinction coefficient demonstrates a rising trend. Specifically, at 400 nm, the extinction coefficient is approximately 0.18 at 250°C, increasing to 0.24 at 400°C. At 600 nm, it rises from 0.12 at 250°C to
0.18 at 400°C, while at 800 nm, it grows from 0.08 at 250°C to 0.14 at 400°C. This increase can be attributed to enhanced absorption due to improved crystalline structure and increased density of absorbing centers within the film (Jain & Bhattacharya, 2021).

4.2.2.Optical Band gap, refractive index and optical dielectric constant

Table 4.1 shows the optical properties of cobalt oxide thin films annealed at different temperatures (250–400 °C) include variations in optical band gap energy, refractive index and optical dielectric constant.
It is observed that, the optical band gap decreased from 2.75 eV at 250°C to 1.80 eV at 400°C, indicating a redshift in the absorption edge with increasing annealing temperature. This suggests improved crystallinity and grain size, which typically reduce quantum confinement effects and lower the band gap. Such behaviour is consistent with previous studies. For example, Shinde et al. (2019) observed that the band gap of cobalt oxide thin films decreased with increased annealing temperature due to enhanced crystallinity and reduced defect density.
The refractive index increased from 2.467 at 250°C to 2.828 at 400°C. This trend suggests an increase in film density and a more compact microstructure as the annealing temperature rises. Higher refractive index values are typical of more crystalline and less porous films. According to Yadav *et al*. (2017), “a higher refractive index generally indicates improved film uniformity and reduced void fraction, which occur due to thermal annealing.”
The optical dielectric constant increased from 5.09 to 7.00 with annealing. This parameter reflects the film’s ability to polarize in response to an electric field and is related to both the refractive index and the band structure. This result aligns with findings by El-Trass *et al.* (2016), who noted that increased thermal treatment enhances the polarizability of the material due to improved structural order.

Conclussion and Recommendation

Spectrometric technique was successfully employed in measurement of the optical properties of annealed cobalt oxide thin films fabricated by sol-gel spin coating deposition method. The results demonstrated that thermal treatment plays a crucial role in tailoring their optical performance. With increasing annealing temperature, absorption intensity and absorption coefficient increased, indicating better light-harvesting capabilities. Transmittance and reflectance decreased, suggesting enhanced structural uniformity and reduced surface scattering. Optical band gap reduced significantly, from
2.75 eV to 1.80 eV, due to improved crystallinity and reduced quantum confinement. Refractive index and optical dielectric constant increased, pointing to greater film compactness and improved

optical response.

These improvements affirm that thermal annealing is a key factor in optimizing the optical properties of cobalt oxide thin films for practical applications in optoelectronics, sensors, and solar energy conversion.

5.2.Recommendations

In view of the findings of this study, the following recommendations were made:

i.Future studies should explore finer temperature intervals to determine the optimal annealing point for achieving desired optical and structural properties.

ii.Complementary techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and atomic force microscopy (AFM) be used to correlate optical behaviour with microstructural evolution.

iii.Given the favourable optical properties, further investigation into the integration of these films into optoelectronic devices such as photodetectors, solar cells, and smart windows is encouraged.

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