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Spray Pyrolysis-Derived Cobalt Oxide Thin Films: Synthesis and Optical Property Evaluation

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Abstract

This study presents a simple and cost-effective approach for synthesizing cobalt oxide (Co_3O_4) thin films on glass substrates using a homemade spray pyrolysis technique. The investigation centers on how thermal treatment affects the optical properties and band gap of Co_3O_4 thin films, synthesized with varying precursor concentrations and layer thicknesses. As-synthesized Co_3O_4 thin films stand out for its remarkable catalytic activity, thermal stability, and optical characteristics attributed to variable oxidation states of Co, strong Co-O bonds, and a narrow band gap. X-ray diffraction (XRD) analysis confirmed the formation of crystalline Co_3O_4, with crystallite size increasing from 12.52 nm (non-annealed) to 16.23 nm after annealing. Optical transmittance data were used to determine the band gap energies, revealing that non-annealed films possessed higher band gap values. This indicates a consistent decrease in band gap energy with increasing annealing temperature. Overall, the spray pyrolysis method offers practical advantages for thin film fabrication, including operational simplicity, scalability, and low production cost.

Keywords*: Cobalt oxide, transmittance, X-ray diffraction, UV-visible spectroscopy, optical bandgap*

1. Introduction

The global push for cleaner energy technologies and advanced sensing systems has intensified research into materials that are efficient, stable, and economically viable. As the demand grows for devices that support sustainable energy conversion and environmental monitoring, the development of multifunctional materials compatible with scalable fabrication techniques becomes increasingly important. Recent environmental studies have also highlighted the significance of cost-effective chemical analysis and characterization techniques for understanding material properties and pollution-related impacts (Karki et al., 2025). In this context, thin film materials have emerged as a critical component in devices such as supercapacitors (Lokhande et al., 2011), solar cells (Ali et al., 2016), gas sensors (Kumarage & Comini, 2021), and electrochromic systems (Yaseen et al., 2024) due to their tunable properties and diverse functionalities.

Among the various classes of functional materials, transition metal oxides (TMOs) have gained significant attention owing to their wide band gaps, high dielectric constants, variable oxidation states, and excellent chemical and thermal stability (Bhandari et al., 2024). These characteristics make TMOs highly suitable for a broad spectrum of applications in optoelectronics, energy storage, catalysis (Kandel et al., 2023), sensing (Farhan et al., 2020) and as electrocatalyst for water-splitting (Kandel et al., 2022). A variety of deposition techniques including RF magnetron sputtering (Dvořáková et al., 2019), atomic layer deposition (Huang et al., 2015) sol–gel processing (Armelao et al., 2001), chemical vapor deposition (Barreca et al., 2001), co-precipitation (G et al., 2018), and spray pyrolysis (Abbas et al., 2017) have been explored to fabricate TMO thin films with tailored properties. Notably, spray pyrolysis has emerged as a particularly attractive method due to its simplicity, low cost, and ability to deposit uniform films over large or non-planar substrates without requiring vacuum conditions.

Within the TMO family, cobalt oxide (Co_3O_4) stands out as a multifunctional and chemically stable material exhibiting a spinel structure composed of Co^{2+} and Co^{3+} ions occupying tetrahedral and octahedral sites, respectively (Zahan & Podder et al., 2019). This mixed valence nature endows Co_3O_4 with unique redox properties and intrinsic p-type semiconducting behavior (Kandalkar et al., 2007). It exhibits two direct optical transitions with band gaps around 1.6 eV and 2.3 eV (Barnasas et al., 2021), making it highly suitable for optoelectronic applications. Additionally, Co_3O_4 demonstrates excellent performance in oxygen evolution and reduction reactions (OER/ORR)(Kandel et al., 2024), gas sensing (ethanol, CO_2, NO_2) (Bagul et al., 2021), electrochromic devices (El Bachiri et al., 2019), and supercapacitors where it has been considered a viable low-cost alternative to noble metal-based materials like RuO_2 (Shinde et al., 2006). Previous studies have highlighted that post-deposition annealing can significantly influence the morphology (Assaker et al., 2024), crystallinity (El Sayed et al., 2016), and optical behavior (Louardi et al., 2017) of Co_3O_4 thin films, leading to enhanced performance in target applications.

Herein, we investigate a cost-effective and scalable strategy for tuning the structural and optical properties of Co_3O_4 thin films using a homemade spray pyrolysis technique. Unlike vacuum-based techniques which require expensive equipment, complex setups, and controlled environments, spray pyrolysis facilitates thin film deposition through a very simple and relatively cost-effective processing method (especially with regard to equipment costs). It does not require high-quality substrates or chemicals and has immense potential regarding deposition of dense,porous multi-layered films (Perednis & Gauckler et al., 2005).Among all chemical methods solution spraying technique (SP) is most popular today because of its applicability to produce a variety of conducting and semiconducting materials (Kate et al., 2022). By systematically varying the precursor concentration and post-deposition annealing temperature, we aim to correlate synthesis parameters with key material attributes such as crystallite size, optical transmittance, and band gap energy. This work not only demonstrates the viability of low-cost fabrication methods for high-quality oxide films but also contributes valuable insights toward optimizing Co_3O_4 thin films for use in energy storage, sensing, and optoelectronic devices. Our findings lay the groundwork for further advancements in functional oxide thin film technology through accessible and scalable fabrication techniques.

2. Experimental

Cobalt chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) was used as the precursor material and dissolved in deionized water to prepare solutions of 0.1 M and 0.025 M concentrations. Glass slides (25.4 × 76.2 mm) were cleaned using alcohol-soaked cotton, followed by sonication for 15 minutes and drying in a hot-air oven at 70°C. A homemade spray pyrolysis setup, consisting of a nebulizer and a household heater, was employed to deposit the films. The system enabled uniform spraying of the precursor solution onto the glass substrates, with synchronized horizontal and vertical movements allowing precise control over film thickness by varying the number of deposition layers. Thin films were prepared using 2 and 4 layers for each concentration. To study the effect of thermal treatment, half of the deposited films were annealed at 500°C in a muffle furnace. This method permitted the study of the influence of precursor concentration, layer thickness, and annealing on the optical and structural properties of the ensuing Co_3O_4 thin films.

3. Materials and Methods

Cobalt chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) was used as the precursor material and dissolved in deionized water to prepare solutions of 0.1 M and 0.025 M concentrations. Glass slides (25.4 × 76.2 mm) were cleaned using alcohol-soaked cotton, followed by sonication for 15 minutes and drying in a hot-air oven at 70°C. A homemade spray pyrolysis setup, consisting

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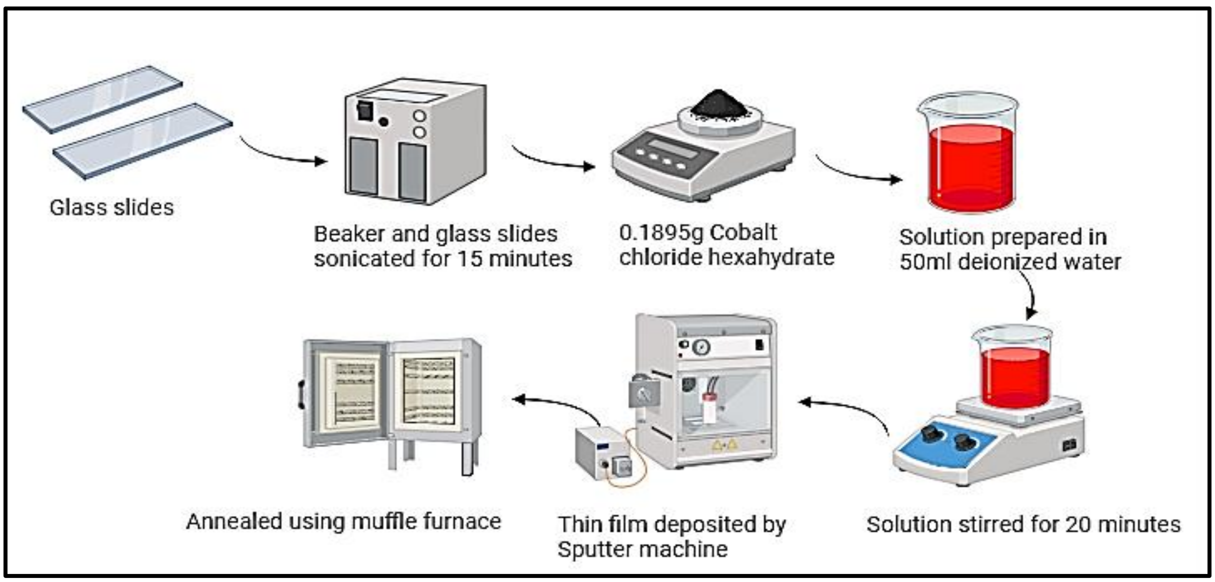
3. Physicochemical Characterization

The optical characterization of the thin films was carried out using a UV-Vis spectrophotometer (HR4000CG UV-NIR, Ocean Optics) at the Physics Research Laboratory, Amrit Campus. X-ray diffraction (XRD) was employed to determine the crystallinity and crystallite size of the films using Scherrer's equation. The crystal structure of the nanoparticles was analyzed using powder XRD with CuKα radiation (λ = 1.54 Å) over a 2θ range of 5° to 90°. XRD measurements were conducted at the National Academy of Science and Technology (NAST).

4. Results and Discussion

Cobalt oxide thin films were synthesized by first preparing glass substrates through alcohol cleaning and 15-minute sonication to ensure surface purity for uniform film deposition. A precursor solution was made by dissolving $CoCl_2 \cdot 6H_2O$ in deionized water.



Scheme 1: Synthesis of thin film of Co_3O_4.

Stirring promoted complete ion dissociation, producing free Co^{2+} ions in solution. The solution was sprayed onto heated glass substrates via spray pyrolysis, where thermal energy decomposed the cobalt salt, initiating oxidation reactions. Subsequent annealing in a muffle furnace facilitated further oxidation and crystal growth, yielding phase-pure Co_3O_4 with enhanced optical and electronic properties ideal for catalytic and semiconducting applications (**Scheme 1**).

To confirm the successful synthesis and phase formation of the Co_3O_4 thin films, X-ray diffraction (XRD) analysis was carried out. The diffraction patterns for four-layered Co_3O_4 films, both non-annealed and annealed, are presented in **Figure 1(a)** and **Figure 1(b),** respectively. The analysis was conducted using Cu Kα radiation with a wavelength of 1.54 Å. The XRD pattern of the non-annealed film displayed broad and low-intensity peaks, indicating poor crystallinity and the presence of partially amorphous phases. In contrast, the annealed film at 500°C exhibited well-defined and sharp diffraction peaks, characteristic of a crystalline cubic spinel structure of Co_3O_4 (**Figure 3(b**).

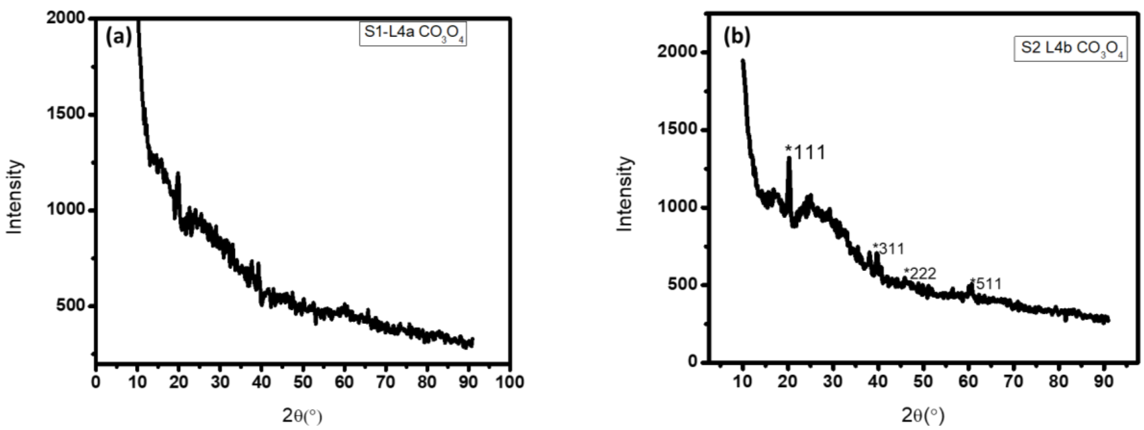


Figure 1. XRD spectra for **(a)** non- annealed and **(b)** annealed cobalt oxide thin film.

The prominent diffraction peaks observed at 2θ ≈ 19.55°, 37.4°, 47.43°, and 59.94° correspond to the (111), (311), (222), and (511) planes, respectively, consistent with standard Co_3O_4 spinel phase as reported in the literature (Patil et al., 1996). The appearance of these well-resolved peaks confirms the formation of a crystalline Co_3O_4 phase with random orientation, which is typical for spray-pyrolyzed thin films. Crystallite sizes were estimated using the Scherrer equation, revealing an average size of 12.52 nm for the non-annealed film and 16.23 nm for the annealed film. On annealing at 500 °C, typical XRD patterns for Co_3O_4 of noticeable sharpness emerge. Annealing Co_3O_4 thin films enhances their crystallinity by allowing atoms to reorganize into a more ordered spinel structure. This leads to sharper and more intense XRD peaks due to increased crystallite size and reduced defects. Annealing also helps in removing lattice strain and converting other cobalt oxide phases into pure Co_3O_4, improving phase purity and structural order (Louardi et al., 2017).Thus, the increase in crystallite size upon annealing suggests that higher thermal energy promotes grain growth, improves crystal orientation, and enhances overall crystallinity(Bekzhanov et al., 2022). These findings underscore the importance of post-deposition thermal treatment in achieving high-quality Co_3O_4 thin films suitable for optoelectronic and catalytic applications.

The optical behavior of the synthesized Co_3O_4 thin films was investigated using UV-Vis-NIR spectroscopy (Ocean Optics spectrometer) across the wavelength range of 200 to 1000 nm. Transmittance data were recorded, and corresponding spectra were plotted with wavelength (nm) on the X-axis and transmittance (%) on the Y-axis (**Figure 2**). The optical transmittance curves provided valuable insights into the light absorption features of the films. To further evaluate the electronic structure, the optical band gap energies were determined using Tauc plots, which relate the absorption coefficient (α) to photon energy (hν) for direct permitted transitions via the equation $(\alpha h\nu)^2 \propto (h\nu - E_g)$. The band gap (Eg) energy was assessed by extrapolating the linear portion of the plot to the energy axis where $(\alpha h\nu)^2 = 0$ (Pejova et al., 2001). Interestingly, Co_3O_4 thin films exhibit dual band gap behavior, comprising both direct and indirect transitions (Bashir et al., 2018). The direct band gap corresponds to a straightforward transition between the valence and conduction bands without a change in momentum, while the indirect band gap involves phonon participation and additional intermediate states. This dual nature is a result of the complex electronic interactions between Co^{2+} and Co^{3+} ions within the spinel lattice structure. According to previous studies, the two distinct transitions can be attributed to charge transfer processes: the first band gap arises from the $O^{2-} \rightarrow Co^{2+}$ transition, and the second from the $O^{2-} \rightarrow Co^{3+}$ transition (Basyooni et al., 2020).

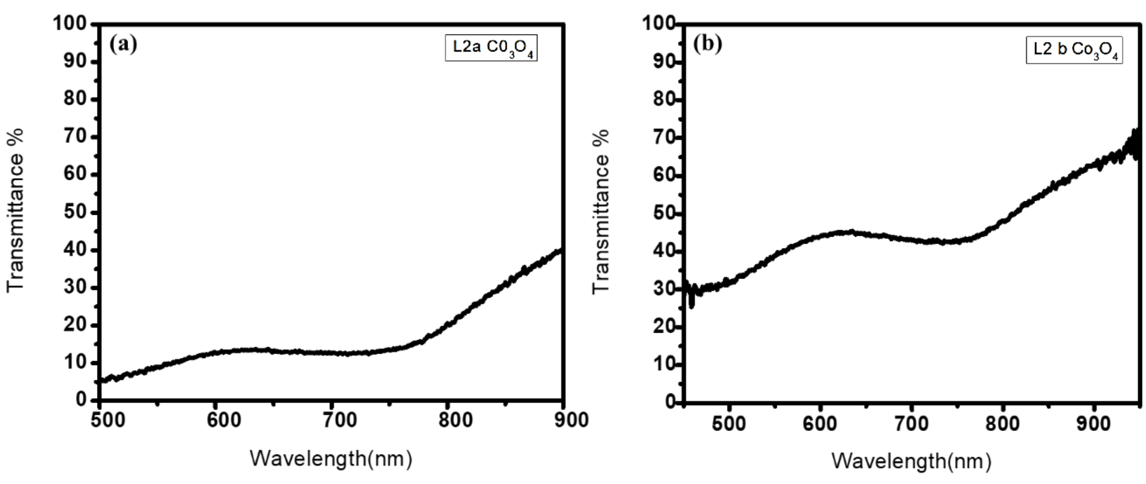


Figure 2. Transmittance curve of **(a)** non- annealed and **(b)** annealed 2 layered cobalt oxide thin film.

The influence of thermal annealing and film thickness (layer number) on the optical properties was systematically analyzed. Comparative evaluations of transmittance and band gap energy were made between annealed and non-annealed films with two-layer and four-layer coatings. The results presented in the following graphs reveal clear trends in how processing conditions impact optical transparency and electronic band structure. As shown in **Figure 2(a)**, the non-annealed two-layered film exhibited a transmittance peak in the range of 500–900 nm, which spans the visible to near-infrared region of the electromagnetic spectrum. The transmittance values for this sample were approximately 20–40%. However, after annealing at 500 °C, the transmittance increased significantly to 40–60%, as demonstrated in **Figure 2(b)**. The optical band gaps of the synthesized Co_3O_4 thin films were further analyzed using Tauc's plots, as shown in **Figure 3**. These plots were derived from the absorbance data to estimate the energy levels at which electronic transitions occur. As shown in **Figure 3 (a)**, the non-annealed two-layered film exhibited two distinct optical band gaps 1.43 eV (lower energy level) and 1.84 eV (higher energy level). After annealing at 500 °C, the corresponding band gaps slightly decreased from 1.84 to 1.64 eV (higher energy level), as seen in **Figure 3 (b)**.

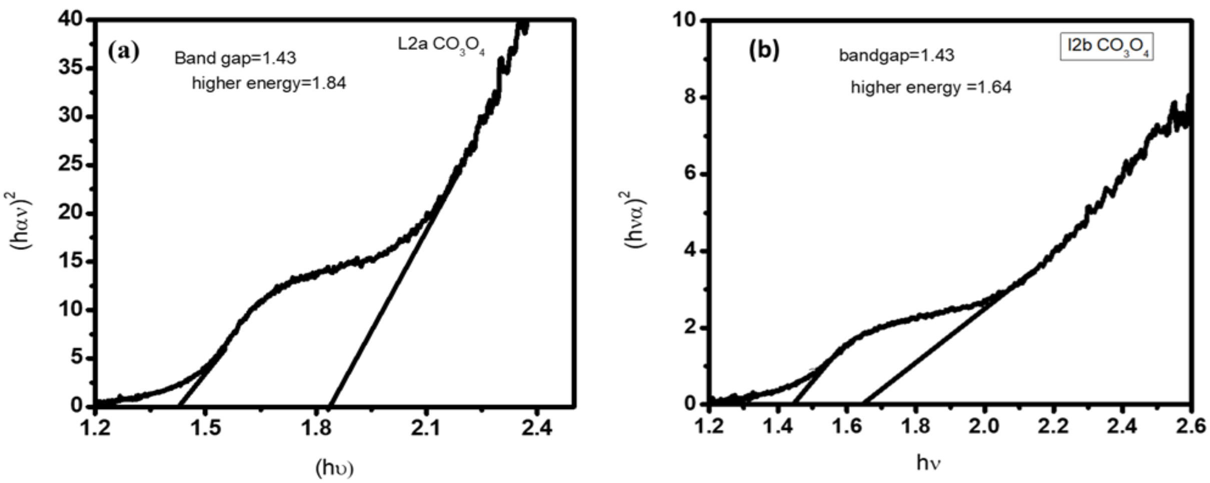


Figure 3. Tauc's plot for 2 layered **(a)** non-annealed and **(b)** annealed cobalt oxide thin film.

This trend aligns with previously reported values for Co_3O_4 thin films in the literature. Thin films typically contain a high density of defects, such as incomplete oxidation, interstitials, and oxygen vacancies, at lower annealing temperatures. Because of disordered electronic states, these defect states can increase the apparent optical band gap by introducing localized energy levels within the band gap. Many of these flaws are repaired as the annealing temperature rises, bringing the film closer to a more homogeneous and stoichiometric phase. This narrows the effective band gap by lowering the number of mid-gap states (Lin et al., 2024). In present case, the mean crystallite size increases from 12.52 nm to 16.23 nm after annealing at 500◦C. Moreover, it is understood that the amorphous phase is reduced with increasing annealing temperature, since more energy is supplied for crystallite growth, thus resulting in an improvement in crystallinity of Co_3O_4 films. Increased transmittance is also observed with higher annealing temperatures as a result of improved crystallinity and reduced defects. Thus, it is believed that both the increase in crystallite size and the reduction in amorphous phase cause decrease in band gap of annealed Co_3O_4 films (Amri et al., 2014). Smaller grains with more grain boundaries make up films at lower temperatures, which may serve as barriers and trapping locations.

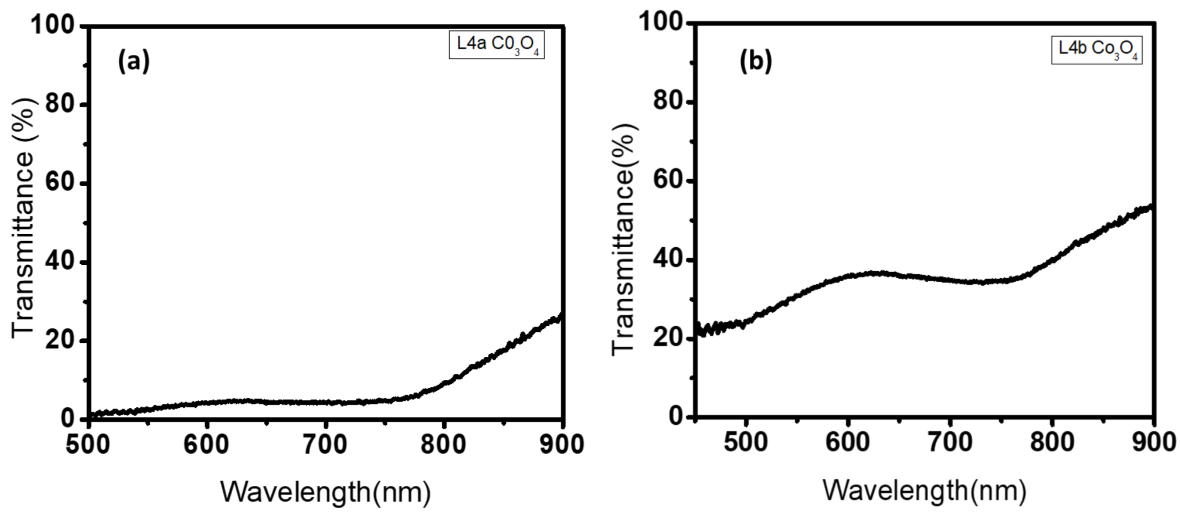


Figure 4. Transmittance curve of **(a)** non- annealed and **(b)** annealed 4 layered cobalt oxide thin film.

Grain boundaries have the ability to produce localized states and scatter charge carriers, which further widens the band gap. Grain growth during annealing lowers the density of the grain boundaries, sharpening the band edges and decreasing the band gap (Hwang et al., 2020). In addition to that the narrowing of band gap by means of charge transfer process ($O^{2-} \rightarrow Co^{3+}$ and $O^{2-} \rightarrow Co^{2+}$) into the Co_3O_4 can be accused for the observation of redshift of the optical absorption. The oxygen vacancy may be created due to the effect of temperature, thus to improve the absorbance and to decrease the optical band gap. The oxygen vacancy increase may indicate an increase of charge carrier in the film which is responsible to cause redshift (Zahan & Podder et al., 2019). Redshift is typically associated with enhanced crystallinity and reduced lattice disorder. Thermal treatment facilitates atomic rearrangement, resulting in improved crystal quality, more uniform energy band structures, and hence a narrower band gap (Duan et al., 2022). A similar pattern was observed for the four-layered Co_3O_4 thin films, with corresponding transmittance curves presented in **Figure 4(a)** and **(b)**. The non-annealed four-layered film showed a transmittance range of 0–20%, which increased substantially to 35–50% upon annealing. The transmittance peaks for both films were located in the 500–900 nm wavelength region, covering the visible (400–700 nm) and near-infrared (700–1400 nm) regions of the electromagnetic spectrum. This optical enhancement is attributed to the annealing-induced improvement in surface morphology and crystallinity, which minimizes scattering and increases transparency.

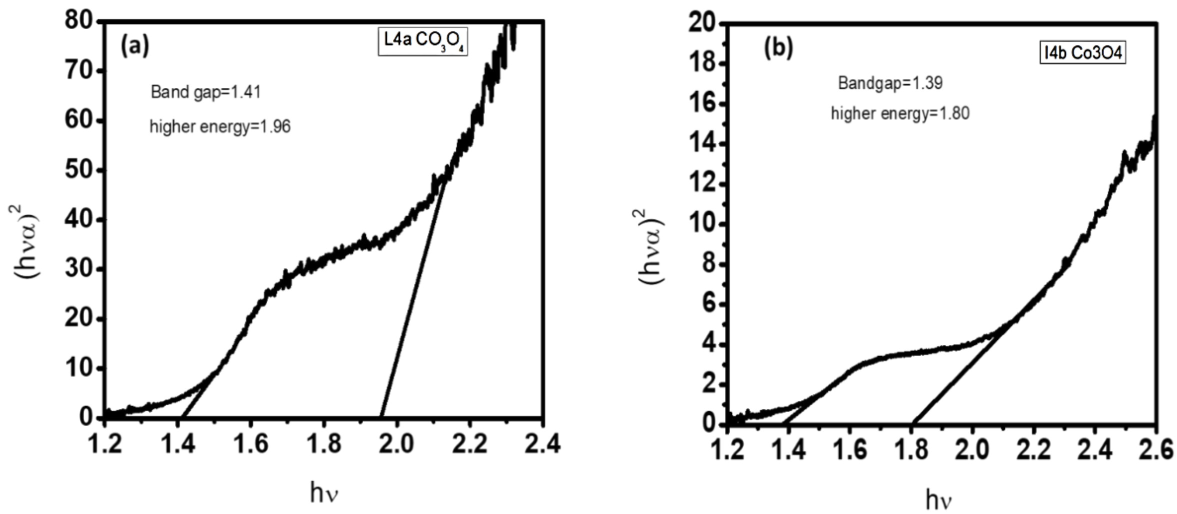


Figure 5. Tauc's plot for 4 layered **(a)** non-annealed and **(b)** annealed cobalt oxide thin film.

From **Figure 5 (a)** and **(b)**, the band gaps of the non-annealed four-layered film were observed to be 1.41–1.96 eV, while the annealed counterpart showed a reduced range of 1.39 – 1.85 eV. This further confirms that annealing leads to a decrease in band gap energy, consistent with the behavior observed in the two-layered films.

T**able 1:** Comparative study of optical properties of thin film.

Comparative study of thin films of 0.1M concentration				**0.025M**
S. N.	Samples	Transmittance (%)	Bandgap (eV)	Bandgap (eV)
1	L2a Co_3O_4	20-40	1.43-1.84	1.45-2.10
2	l2b Co_3O_4	40-60	1.43-1.64	1.42-1.95
3	L4a Co_3O_4	0-20	1.41-1.96	1.49-2.03
4	L4b Co_3O_4	35-50	1.39-1.8	1.42-1.82

To better understand the influence of precursor concentration and film thickness, a comparative study of optical properties was conducted for films prepared at 0.1 M and 0.025 M precursor concentrations. The results are summarized in Table 1. These results clearly demonstrate that annealing consistently enhances the optical transmittance and reduces the band gap energy in Co_3O_4 thin films, irrespective of layer thickness or precursor concentration. This behavior is mainly driven by improved crystal quality, grain connectivity, and reduced structural defects, all of which contribute to better optical performance. The ability to tune the band gap through simple thermal treatment makes Co_3O_4 a strong candidate for applications in solar cells, optoelectronics, and photodetectors.

5. Conclusions

This study successfully demonstrated the synthesis of cobalt oxide (Co_3O_4) thin films via a cost-effective homemade spray pyrolysis method, highlighting significant improvements in optical properties upon thermal annealing. The transmittance spectra exhibited characteristic double peaks within the visible to near-infrared region (500–900 nm), confirming the formation of optically active films. Notably, the optical band gap showed a decreasing trend with annealing for 2-layer films, consistent with earlier observations on annealing indicating enhanced crystallinity and reduced electronic disorder. Films prepared using a lower precursor concentration (0.025 M) showed slightly higher band gap values compared to those prepared with 0.1 M, revealing the influence of solution molarity on electronic structure. The improved transparency and narrowing of the band gap after annealing suggest better light absorption and electronic conductivity, making these films ideal candidates for applications in optoelectronics, solar energy conversion, and transparent electronic devices.

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CRediT authorship contribution statement

Muskan Ghimire: Methodology, Investigation, Formal analysis, Data curation, Writing-original draft preparation **Bishow Karki:** Conceptualization, Writing-original draft preparation, **Leela Pradhan Joshi:** Conceptualization, Review and Editing, **Lekhanath Kandel:** Formal analysis and review **Mani Ram Kandel:** Conceptualization, Resources, Writing-review and Editing, supervision **Sharmila Pradhan**: Conceptualization, Resources, Writing-review and Editing, Supervision, Project handling.

Conflicts of Interest: The authors declare no conflict of interest.

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