International Journal of Research Studies on Environment, Earth, and Allied Sciences (IJRSEAS)
| *Volume-3 Issue-2* | *April-2026* | *ISSN: 3066-0637* | *Website:* https://eesrd.us

Manuscript ID:
IJRSEAS-2026-030239

OPEN ACCESS

Quick Response Code:

Website: https://eesrd.us

DOI: 10.5281/zenodo.20840110

DOI Link:
https://doi.org/10.5281/zenodo.20840110

Volume: 3

Issue: 2

Pp. 232-236

Month: April

Year: 2026

E-ISSN: 3066-0637

Submitted: 14 Mar. 2026

Revised: 25 Mar. 2026

Accepted: 15 Apr. 2026

Published: 30 Apr. 2026

Address for correspondence:
Nilesh S. Kanhe
Department of Physics, Arts, Commerce and Science College Satral, (SPPU, Pune) Maharashtra, India
Email: nilesh264284@gmail.com

How to cite this article:
Kanhe, N. S., Bhalekar, V. P., & Nawale, A. B. (2026). Dielectric behavior of electrodeposited cobalt oxide thin films. International Journal of Research Studies on Environment, Earth, and Allied Sciences, 3(2), 232–236. https://doi.org/10.5281/zenodo.20840110

Dielectric behavior of electrodeposited cobalt oxide thin films

Nilesh S. Kanhe[1], Vikram P. Bhalekar[2], Ashok B. Nawale[3]
[1]Department of Physics, Arts, Commerce and Science College Satral, (SPPU, Pune) Maharashtra, India
[2]Department of Physics, Arts, Science and Commerce College Rahata, (SPPU, Pune) Maharashtra, India
[3]Department of Physics, Government Vidarbha Institute of Science and Humanities, Amravati, Maharashtra, India

Abstract

Face-centered cubic (FCC) Co_3O_4 cobalt oxide thin films were deposited on copper substrates using the electrodeposition technique. The films exhibit a flake-like surface morphology, resulting in an increased effective surface area. The dielectric properties of the films, prepared at an applied potential of 1.9 V for 60 min, were investigated over a frequency range of 100 Hz to 5 MHz at room temperature. The results show that the dielectric response depends on film thickness, with dipole relaxation time decreasing as the thickness increases. These findings suggest that the synthesized films are promising for applications that require controllable dielectric properties.

Keywords: *electrodeposition, dielectric, cobalt oxide and thin films*

Introduction

Materials in bulk form possess three dimensions: length, width, and thickness, which define their physical characteristics. When one dimension, typically thickness, is reduced to a very small scale, the material is referred to as a thin film. Thin films generally have thicknesses ranging from a few nanometers to several micrometers and their physical and chemical properties can differ significantly from those of bulk materials due to size effects and surface phenomena [1,2]. Thin films can be fabricated using a variety of physical and chemical deposition techniques. Physical methods, such as thermal evaporation, sputtering, electron beam evaporation, pulsed laser deposition and cathodic arc deposition, generally produce films with high uniformity and reproducibility, but often involve higher costs and complex instrumentation [2,3]. In contrast, chemical methods, including chemical solution deposition and electrodeposition, are more economical and suitable for large-scale production [4–6]. Among these techniques, electrodeposition has gained considerable attention due to its simplicity, low cost and scalability. This method allows for the deposition of uniform coatings on both flat and irregular surfaces. In addition, it provides flexibility in controlling film composition, thickness and morphology by adjusting deposition parameters. As a result, electrodeposition is widely used for preparing a variety of materials, including metals, semiconductors, polymers, and functional oxides [7,8].

Cobalt readily forms several oxides, including cobalt monoxide (CoO), cobalt sesquioxide (Co_2O_3) and cobalt oxide (Co_3O_4) [9]. Among these, Co_3O_4 is thermodynamically stable and exhibits a face-centered cubic (FCC) structure. It is commonly formed through the thermal transformation of Co_2O_3 at elevated temperatures [10]. Cobalt oxides have attracted significant research interest due to their wide range of applications, including corrosion resistance, magnetic devices, sensors, electrochromic systems and energy storage devices such as supercapacitors [8,11–13]. In particular, Co_3O_4 is considered a promising electrode material because of its good electrical conductivity, chemical stability and high surface activity. Nanostructured cobalt oxide, especially with porous or flake-like morphology, offers a large surface area and enhanced electrochemical performance, making it suitable for advanced applications [8,14,15].

Different methods have been employed to fabricate cobalt oxide thin films on a variety of substrates, each offering its own set of benefits. In this work, cobalt oxide films are deposited onto copper substrates using the electrodeposition technique, which is a simple and economical approach. The resulting films are then characterized to evaluate their structural, surface morphological and dielectric properties.

Experimental

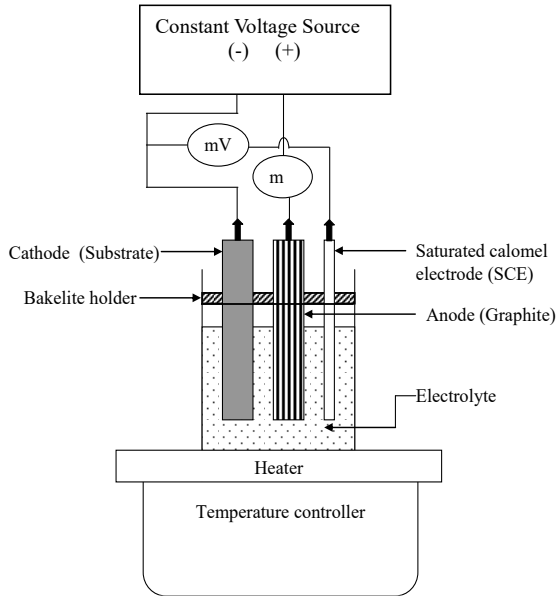

Figure 1: Electrodeposition setup adapted from reference [16]

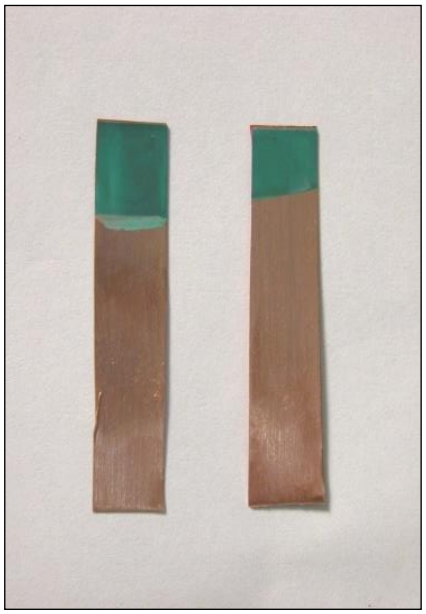

Figure 1: Photograph of as synthesized cobalt oxide thin films.

Thin films of Cobalt oxide were deposited on copper substrates using a potentiostatic cathodic electrodeposition method as shown in Figure 1. The deposition was carried out using a regulated DC power supply (0–30 V) with a 0.1 M $CoCl_2$ aqueous solution as the electrolyte. A copper strip served as the working electrode while a graphite as a counter electrode. The electrolyte was prepared using double-distilled water, stirred until fully dissolved and maintained at a pH of 5–6. All experiments were performed at room temperature under controlled conditions [5,14,17,18].

Prior to deposition, the copper substrates were mechanically polished to remove surface oxides and irregularities, followed by cleaning with water and acetone, and finally rinsed with double-distilled water. This ensured a smooth and clean surface for uniform film growth [19,20]. The electrodes (5×1 cm^2) were immersed in approximately 50 mL of electrolyte, with an effective deposition area of about 1×1 cm^2. The applied voltage and deposition time were optimized to obtain uniform films with desired thickness and morphology. During deposition, electrochemical reactions at the cathode led to the formation of cobalt oxide layers.

The as-prepared films were examined using X-ray diffraction (XRD) to investigate their crystal structure, while scanning electron microscopy (SEM) was employed to study surface morphology. Energy-dispersive X-ray spectroscopy (EDS) was used to determine the elemental composition. In addition, dielectric properties were evaluated at room temperature across a range of frequencies.

Results and discussion

Thin films of cobalt oxide were successfully deposited on Cu substrates using a chloride-based electrolyte via a potentiostatic electrodeposition method. Similar electrodeposition approaches for cobalt-based oxide thin films have been widely reported in recent literature [21,22].

Table 1: Typical electrodeposition parameters and corresponding observations

Ob. No.	Applied Voltage in Volts	Deposition time in Minutes	Observations
1.	1.9	15	Uniform films
2.	1.9	30	Uniform films (thickness increases than above)
3.	1.9	45	Uniform films (thickness increases than above)
4.	1.9	60	Uniform films (thickness increases than above)
5.	1.9	85	Cracks are observed on the film.
6.	1.9	25 + 25	Double depositions: Uniform and thick film

The $CoCl_2$ solution used in the process exhibited a pH of 5–6, and the electrochemical cell showed a half-cell potential of approximately 0.8 V. The deposition conditions, such as applied voltage and deposition duration, were carefully adjusted to achieve uniform coatings. The films obtained after deposition showed a green color, as illustrated in Figure 1, which is consistent with cobalt oxide/hydroxide-related phases reported in previous studies [21,23,24]. At an applied voltage of 1.9 V and a deposition time of 25 min, uniform film formation was observed. Increasing deposition time led to a gradual increase in film thickness. However, prolonged deposition resulted in reduced adhesion and occasional cracking, which may be attributed to ion depletion in the electrolyte and stress

accumulation in the growing film[22,24]. Therefore, at an applied voltage of 1.9 V and a deposition time of 25 min, film was double deposited. Ambient oxygen during deposition also promotes the formation of cobalt hydroxide along with cobalt oxide phases, as reported in similar systems[23].

The electrodeposition process involves dissociation of cobalt chloride in aqueous solution:

$$CoCl_2 \rightarrow Co^{2+} + 2Cl^- \quad (1)$$

Under an applied electric field, Co^{2+} ions migrate toward the cathode, where they undergo electrochemical reactions leading to cobalt oxide/hydroxide formation. Hydrogen evolution occurs simultaneously at the cathode, while chloride ions migrate toward the anode. Such reaction pathways for cobalt oxide formation via aqueous electrodeposition have been widely reported in literature[21,22]:

$$2CoCl_2 + 3H_2O \rightarrow Co_2O_3 + 3H_2\uparrow + 4Cl^- \quad (2)$$

However, due to the presence of dissolved oxygen and hydroxyl species, mixed oxide-hydroxide phases are commonly formed [23].

1. X-ray Diffraction (XRD) Analysis

Figure 2 (a-b) presents the XRD patterns of electrodeposited cobalt oxide films in both the as-deposited and annealed states. The as-deposited film exhibits diffraction peaks corresponding to Co_2O_3 along with hydroxide-related peaks, consistent with previous reports on electrodeposited cobalt oxide systems[10,23,24].

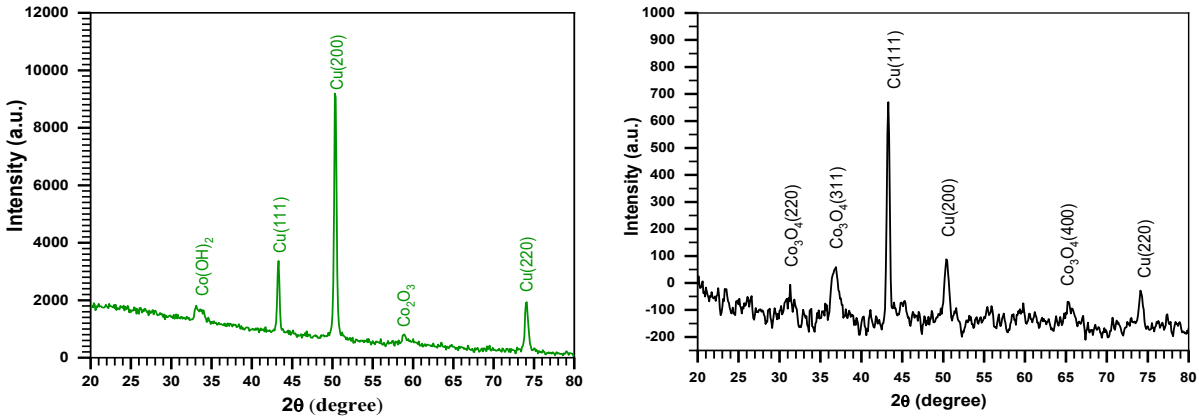

Figure 2: XRD patterns of cobalt oxide films: (a) before annealing, (b) after annealing at 200 °C for 3 hr.

The diffraction peaks observed at 2θ values of 31.27°, 36.84°, and 65.23° are indexed to the (220), (311), and (440) crystallographic planes, respectively. Comparable structural characteristics have also been documented in previous studies on cobalt oxide thin films prepared using similar methods [10,23,24]. Copper substrate peaks are also observed due to X-ray penetration. After annealing at 200 °C for 3 h, a clear phase transformation occurs. A noticeable colour transition from copper-blue to black was also observed in the film. The disappearance of hydroxide-related peaks and structural modification confirm the conversion of Co_2O_3 into the stable FCC Co_3O_4 phase as presented in Figure 2(b), in agreement with literature[23].

2. Surface Morphology and Elemental Analysis (SEM & EDS)

Surface morphology was examined using a scanning electron microscope (SEM, JEOL JSM 6360A). To prevent charging during imaging, the samples were coated with a thin layer of platinum. The SEM images indicate a porous structure with a flake-like surface morphology, as shown in Figure 3, which significantly increases the effective surface area. Similar flake-like structures in electrodeposited cobalt oxide films have been reported to enhance electrochemical performance and sensing activity [8]. EDS analysis confirms the presence of cobalt and oxygen elements, indicating successful formation of cobalt oxide. However, hydroxide phases cannot be directly distinguished using EDS due to its elemental limitation.

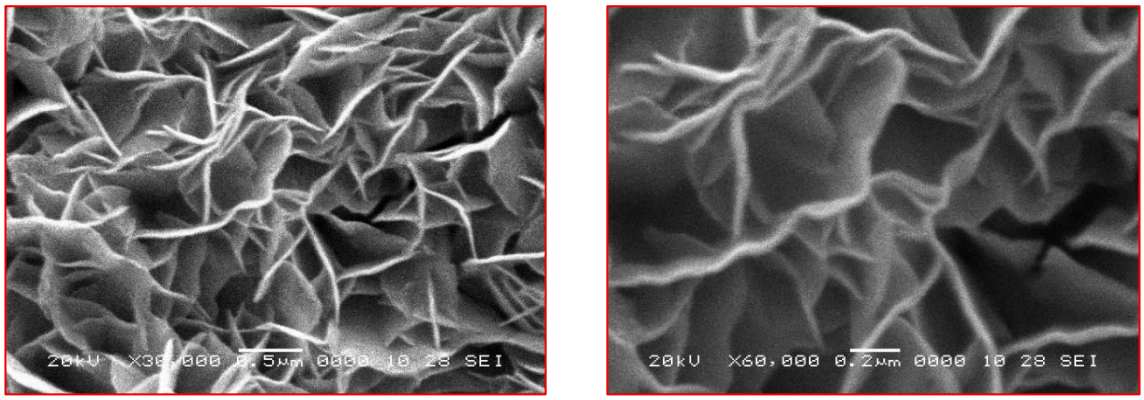

Figure 3: SEM images of cobalt oxide thin films at different magnifications.

3. Dielectric Properties

Dielectric properties were evaluated at room temperature using an LCR meter across a frequency range of 100 Hz to 5 MHz. The dielectric response of the cobalt oxide thin films is presented in Figure 4(a–c). Specifically, Figure 4 (a) illustrates the frequency-dependent behavior of films deposited for 15, 30, 45, and 60 minutes, respectively. The dielectric constant decreases with increasing frequency, which is attributed to the inability of dipoles and charge carriers to follow the alternating field at higher frequencies. Higher dielectric constant values observed in thicker films (45 min and 60 min deposition) indicate enhanced polarization effects due to increased grain boundary and interfacial polarization [25]. Figure 4 (b) shows the dielectric loss of cobalt oxide thin films. It clearly observed that film deposited near 30 minutes had minimum dielectric losses while in other films maximum losses due to poor adhesion or cracks. Therefore, we have doubly deposited the film for higher film thickness without any cracks at 1.9v and 25 minutes to avoid the minimum dielectric losses. Figure 4 (c) shows the ac resistivity of cobalt oxide thin films for different deposition time. This behavior is consistent with previously reported dielectric studies on metal oxide thin films [25]. Dielectric loss and AC resistivity also decrease with increasing frequency, which is typical behavior for semiconducting oxide materials and agrees well with literature reports[25].

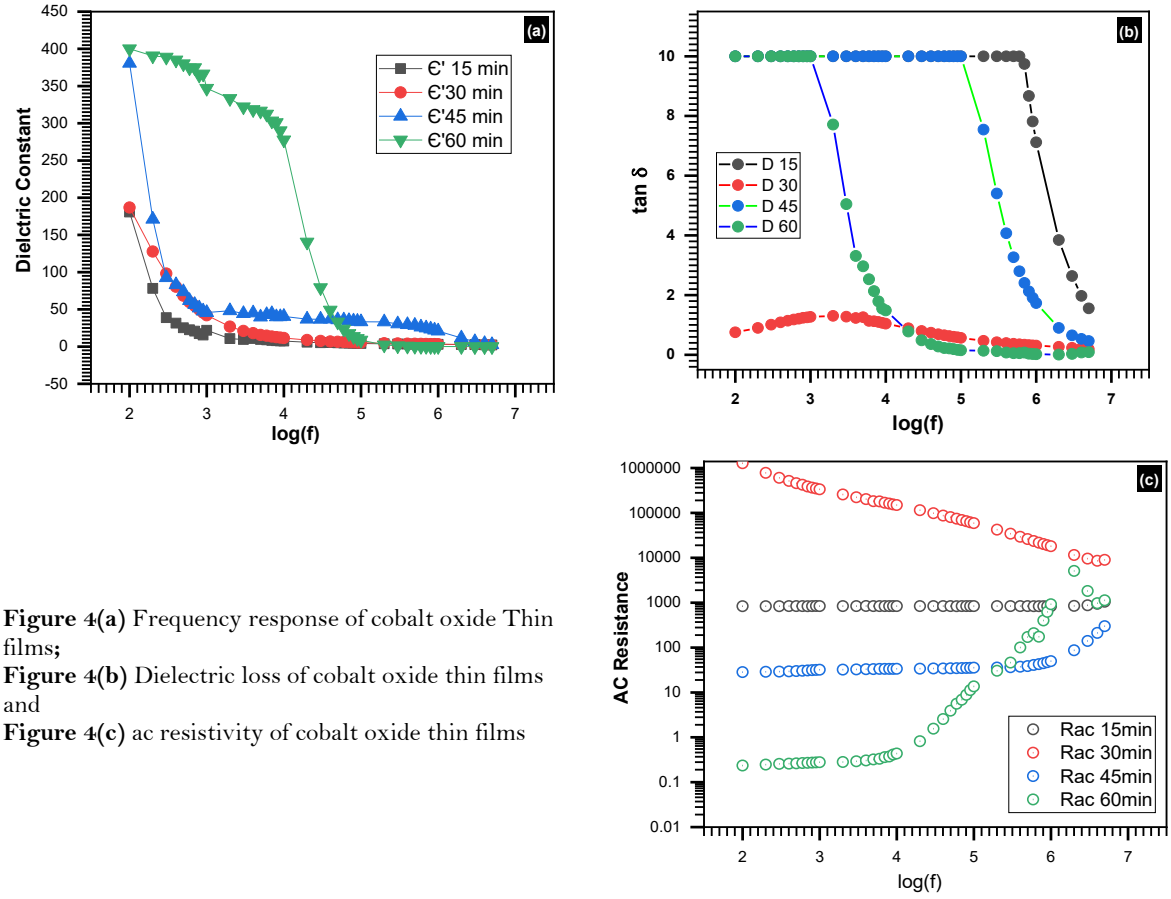

Figure 4(a) Frequency response of cobalt oxide Thin films;
Figure 4(b) Dielectric loss of cobalt oxide thin films and
Figure 4(c) ac resistivity of cobalt oxide thin films

Conclusions

Thin films of cobalt oxide were successfully prepared on copper substrates using an electrodeposition technique. The study shows that deposition conditions significantly affect film thickness, morphology and quality. Structural analysis indicates the presence of Co_2O_3 and hydroxide phases in as-deposited films, which transform into stable Co_3O_4 after annealing. Surface examination reveals a porous, flake-like structure that enhances the surface area. Dielectric measurements demonstrate a decrease in dielectric constant with increasing frequency, while thicker films exhibit improved dielectric response. These results confirm that the properties of cobalt oxide thin films can be controlled through deposition parameters for potential use in electronic and energy-related applications.

Acknowledgments

The authors sincerely acknowledge Prof. H. M. Pathan (SPPU Pune) and Prof. V. L. Mathe (SPPU Pune) for providing access to the experimental facilities used in this study.

Financial support and sponsorship

Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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