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Effect of Cobalt Nitrate on the Electrical Properties of Kapton/Cobalt Oxide Composite by Ion Exchange Method

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Abstract
Kapton polyimide is widely recognized for its exceptional thermal stability and chemical resistance, making it ideal for demanding applications such as aerospace and industrial environments. However, its intrinsic electrical insulation limits its use in active electronic components. This study aims to enhance the electrical conductivity of Kapton without compromising its thermal and chemical integrity by incorporating cobalt oxide (Co_3O_4) through a surface-selective ion exchange method. Kapton films were first activated via alkaline hydrolysis and subsequently immersed in cobalt(II) nitrate solutions at concentrations of 0.25 M, 0.50 M, and 0.75 M, followed by thermal treatment to convert the absorbed cobalt ions into Co_3O_4. Characterization techniques, including Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR), X-ray diffraction (XRD), scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDX), and 4-point probe (4PP) measurements, were employed to analyze chemical structure, crystallinity, surface morphology, elemental composition, and electrical properties. FTIR-ATR confirmed Co_3O_4 formation with a Co–O peak at 663 cm^{-1}, and analysis verified the formation of crystalline Co_3O_4 with a spinel structure, with increasing peak intensity observed from 0.25 M to 0.75 M, indicating improved crystallinity. EDS analysis showed cobalt content increasing with precursor concentration, specifically 28.24 wt.% at 0.25 M, 37.08 wt.% at 0.50 M, and 51.65 wt.% at 0.75 M, while electrical increased from 0.0218 S/cm (0.25 M) to 0.1082 S/cm (0.50 M), before slightly decreasing to 0.0778 S/cm at 0.75 M. These findings suggest that 0.50 M cobalt nitrate offers the optimal balance between conductivity enhancement and structural uniformity for Kapton-based flexible electronics.
Keywords: Kapton composite; electrical properties; conductive polymer; ion exchange method; cobalt nitrate

Introduction

Kapton polyimide is a high-performance polymer known for its exceptional thermal stability, chemical resistance, and mechanical strength, making it widely used in aerospace, electronics, and harsh industrial environments. However, due to insulating nature limits its applicability in active electronic components where electrical conductivity is crucial. This limitation arises from its lack of mobile charge carriers and delocalized π-electrons, resulting in high-volume resistivity (Benfridja et al., 2022). Consequently, efforts have been made to enhance Kapton's electrical properties without compromising its thermal and mechanical advantages (Benfridja et al., 2022; Drasovean et al., 2010; Mu et al., 2010). Among the most promising approaches is the incorporation of cobalt oxide (Co_3O_4), a mixed-valence transition metal oxide that offers semiconducting behavior and stable electrical conductivity. To achieve this, the ion exchange method has been employed, involving alkaline activation of the Kapton surface followed by cobalt ion incorporation and thermal treatment to form a conductive Co_3O_4 layer.

Despite the potential of this method, a critical gap remains in understanding how varying cobalt nitrate concentrations affect the resulting Kapton/cobalt oxide composite. Too little cobalt may result in

insufficient conductivity, while excessive doping can lead to particle agglomeration and structural instability. Therefore, this study aims to investigate the influence of cobalt salt concentration on the structural, morphological, and electrical properties of Kapton/Co_3O_4 composites. The research focuses on three concentrations 0.25 M, 0.50 M, and 0.75 M applied through an ion exchange process. Key objectives include preparing composite films at these concentrations, analyzing changes in Kapton's structure after surface activation, and evaluating the electrical behavior of the doped films. Techniques such as FTIR-ATR, XRD, SEM-EDX, and 4PP measurements are used for comprehensive characterization of chemical bonding, crystallinity, surface morphology, and conductivity.

By optimizing cobalt nitrate concentration, this research contributes to the development of conductive Kapton-based materials for use in flexible electronics, sensors, and energy devices. The findings have practical significance for industries seeking lightweight, durable, and conductive polymer composites. Moreover, this work aligns with Malaysia's MADANI framework and supports the United Nations Sustainable Development Goal 9, which promotes innovation and infrastructure through advanced material technologies. The insights gained from this study will support future design strategies for high-performance polymer-inorganic hybrids and promote more sustainable and efficient electronic systems.

Materials and methods

3.1. Experimental Materials

A commercial polyimide film (Kapton) with thickness of 0.04 mm was used in this study. The films were clean with acetone and ethanol purchased from R&M Chemicals, Malaysia. Cobalt nitrate ($Co(NO_3)_2$ (>99.0%) with analytical quality was purchased from HmbG Chemicals, Germany. Potassium hydroxide (KOH) was obtained from Systerm, Malaysia and used without further purification

3.2. Preparation of Kapton/Co_3O_4 Composite Film

Kapton with a thickness of about 0.04 mm was cut into small pieces measuring 10 mm × 10 mm. The films were then cleaned ultrasonically with acetone and absolute ethanol for 10 minutes to remove any residual organic impurities from the surface of the film and were allowed to dry naturally. For this experiment, the smooth and reflective side of the Kapton film was selected as the test surface.

3.3. Hydrolysis Reaction

The Kapton film was immersed in a 5 M KOH solution at room temperature. After 5 minutes, it was rinsed thoroughly with a large amount of water to remove any residual lye from the surface. This reaction broke down the polyimide structure of Kapton due to the presence of hydroxide ions, which cleaved the imide linkages in the polymer chain. This process resulted in the decomposition of the polymer into its monomeric components, such as diamines and tetracarboxylic acids. The structural changes of the hydrolyzed Kapton film were characterized using FTIR-ATR.

3.4. Film Composite

The hydrolyzed Kapton film was immersed in a 0.25 M $Co(NO_3)_2{\cdot}6H_2O$ solution for 120 minutes, followed by thorough washing with a large amount of deionized water to remove any residual metal salt solution from the surface. The film was then left to dry naturally in air. Subsequently, the film underwent heat treatment in a high temperature-controlled tube furnace, where the temperature was increased from room temperature to 135 °C and held for 1 hour. It was then further heated to 350 °C at a rate of 2 °C/min. After 2 hours at 350 °C, the film was cooled to room temperature, resulting in the formation of a Kapton/Co_3O_4 nanocomposite film on the Kapton substrate. The samples were characterized using XRD, SEM-EDX, and 4PP measurements to examine the structural changes, morphology, and electrical properties of the composite film. These steps were repeated using 0.5 M and 0.75 M $Co(NO_3)_2{\cdot}6H_2O$ solutions to study the effect of precursor concentration on the properties of the resulting nanocomposite films. The parameters tested summarized in Table 1.

Table 1: Parameters of Kapton composite films prepared under different conditions.

Sample	KOH treatment conditions		Ion-exchange conditions in $Co(NO_3)_2 \cdot 6H_2O$		Thermal treatment time at 350 ºC/h
	Concentration (M)	Time (min)	Concentration (M)	Time (min)	
Kapton	5	5	0.25 0.50 0.75	120	2

Results and discussion

4.1. Fourier Transform Infrared Spectroscopy-Attenuated Total Reflectance (FTIR-ATR)

FTIR-ATR spectroscopy was performed to evaluate the structural and chemical changes in Kapton films following surface activation with KOH and subsequent treatment with cobalt salt solutions of varying concentrations of 0.25 M, 0.50 M, and 0.75 M. The spectra of pure Kapton, KOH-activated Kapton, and the Kapton/cobalt oxide composites are presented in Figure 1.

FTIR-ATR analysis reveals progressive structural modifications in Kapton films following alkaline activation and cobalt doping. Pure Kapton exhibited characteristic absorption bands at 1775 cm^{-1}, 1709 cm^{-1}, and 1372 cm^{-1}, confirming the integrity of its imide carbonyl groups and polyimide backbone. Upon alkaline treatment, the intensity of carbonyl peaks diminished, and new bands appeared at 1643 cm^{-1} and 1548 cm^{-1}, indicating imide ring cleavage and the formation of amide structures. This confirmed successful surface activation through hydrolysis. Following cobalt ion exchange at 0.25 M, additional peaks near 1363 cm^{-1} and 718 cm^{-1} emerged, suggesting initial Co–O interactions. At higher concentrations 0.50 M and 0.75 M, more pronounced changes are observed, with distinct Co–O vibrational bands appearing at 661 cm^{-1} and 663 cm^{-1} respectively, confirming the formation of cobalt oxide (Co_3O_4) nanoparticles (Budiyanto et al., 2023). These spectral changes, supported by the visible dark green colour of the films (Mu et al., 2010), validate the effective incorporation and crystallization of cobalt oxide on the Kapton surface, with increased cobalt concentration enhancing dopant integration and oxide phase formation.

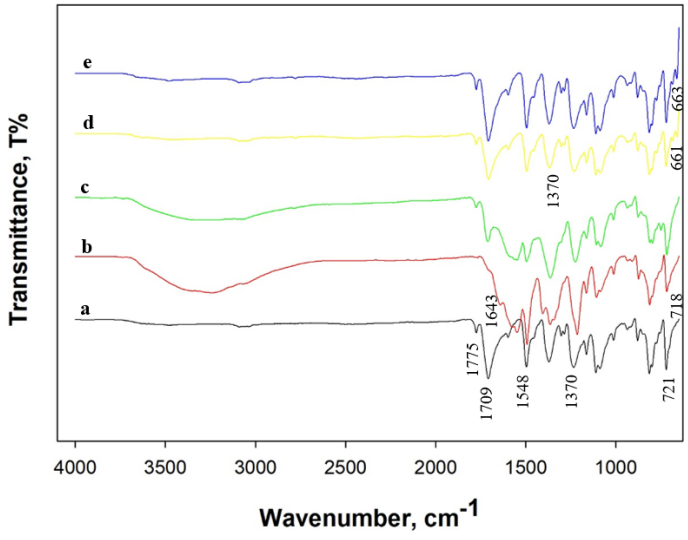

Figure 1 FTIR-ATR spectra of the Kapton polyimide film: (a) pure Kapton, (b) surface activation, (c) 0.25 M (d) 0.50 M, and (e) 0.75 M

4.2. X-ray diffraction (XRD)

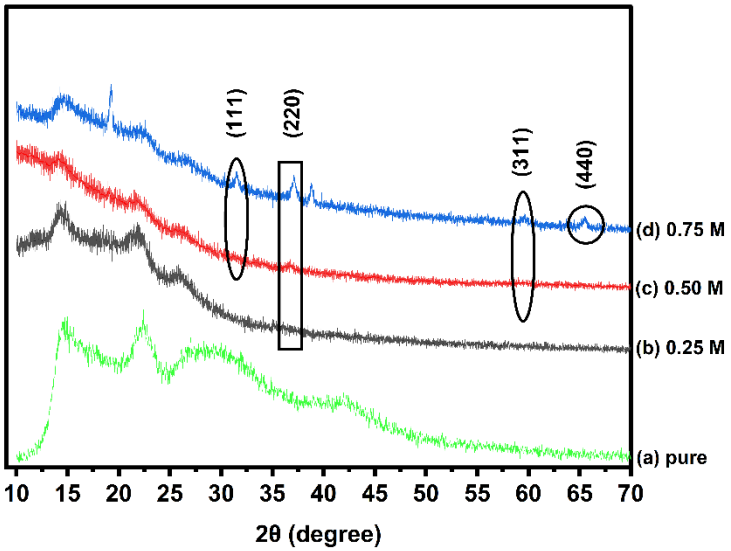

Figure 2 XRD patterns for the Kapton/Co_3O_4 composite films obtained in different concentrations

XRD analysis confirmed the formation and crystallinity of Co_3O_4 in Kapton-based composite films treated with varying concentrations of cobalt nitrate the resulting diffraction patterns are presented in Figure 2. While pure Kapton exhibited a broad amorphous hump around $2\theta \approx 20°$ (Iqbal et al., 2023), the doped samples displayed sharp diffraction peaks at $2\theta \approx 31.5°$, 37.0°, 44.9°, 59.3°, and 65.2°, corresponding to the (111), (220), (400), (311), and (440) planes of cubic spinel Co_3O_4. Increasing cobalt precursor concentration resulted in enhanced peak intensity and sharpness, indicating improved crystallinity and greater cobalt oxide deposition (Iqbal et al., 2023). The 0.25 M sample showed weak, broad peaks suggestive of fine-grained crystallites, while the 0.50 M and 0.75 M samples demonstrated progressively more defined peaks, confirming the formation of larger and more ordered crystallites. Despite this surface crystallization, the underlying Kapton retained its amorphous nature. These results affirm that higher cobalt concentrations promote efficient nucleation and Co_3O_4 growth, which are expected to improve the electrical and morphological properties of the composites.

4.3. Scanning electron microscopy (SEM-EDX)

SEM was used to examine the surface morphology of Kapton/Co_3O_4 composite films prepared with varying cobalt nitrate concentrations 0.25 M, 0.50 M, and 0.75 M. Although high-magnification imaging was conducted, distinct cobalt oxide particle features are not clearly visible, likely due to the smooth Kapton surface, fine particle size, and uniform cobalt oxide coverage. The Co_3O_4 layer and the insulating nature of Kapton further limited contrast, rendering SEM based morphological comparisons inconclusive. To confirm cobalt incorporation, EDX was employed, revealing increasing cobalt content with higher precursor concentrations. These results validated successful cobalt oxide deposition, despite the limited surface detail observed in SEM images.

SEM was employed to investigate the surface morphology of Kapton/Co_3O_4 composite films and to assess the influence of cobalt nitrate concentration on particle distribution and coverage. The micrograph of pure Kapton Figure 3 reveals a relatively smooth and homogeneous surface, typical of an untreated polyimide film consistent with its amorphous and non-porous polymeric nature. This morphology is typical for polyimide films, which do not possess inherent surface roughness or particulate features in their pristine state.

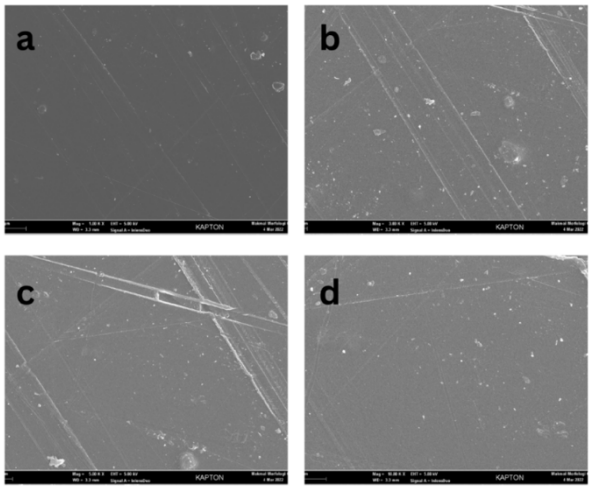

Figure 3 SEM micrographs of pure Kapton films at various magnifications (a) 1.0 K, (b) 3.0 K, (c) 5.0 K and (d) 10.0 K

At a cobalt nitrate concentration of 0.25 M, the SEM image from Figure 4 does not show distinct or well-defined cobalt oxide nanoparticles. The low particle density suggests limited cobalt incorporation, possibly due to low ion availability or insufficient nucleation (Liu et al., 2015). From Table 2, the 0.25 M sample showed the lowest cobalt content at 28.24 wt.%, with 25.30 wt.% oxygen and 46.35 wt.% carbon. The high carbon content is attributed to the Kapton substrate, indicating that cobalt oxide deposition is sparse and that a large portion of the polymer surface remained exposed. This result is consistent with limited nucleation of cobalt oxide at low precursor concentrations.

As the cobalt nitrate concentration increases to 0.50 M, the surface morphology shown in Figure 5 difference surface compared to the 0.25 M sample. These features suggest enhanced adhesion and growth of crystalline cobalt oxide on the Kapton substrate, indicating more effective doping at this intermediate concentration (Budiyanto et al., 2023; Liu et al., 2015). Corresponding elemental analysis in Table 2 shows that the cobalt content increases significantly to 31.93 wt.%, while the carbon content decreases to 36.82 wt.%, implying greater Co_3O_4 deposition and reduced surface visibility of the underlying polymer. Additionally, the oxygen content rises to 30.56 wt.%, further supporting the presence of metal oxide species (Mu et al., 2010). These compositional changes confirm that 0.50 M cobalt nitrate enables more efficient nucleation and growth of cobalt oxide, resulting in improved surface coverage and a denser, more conductive composite layer.

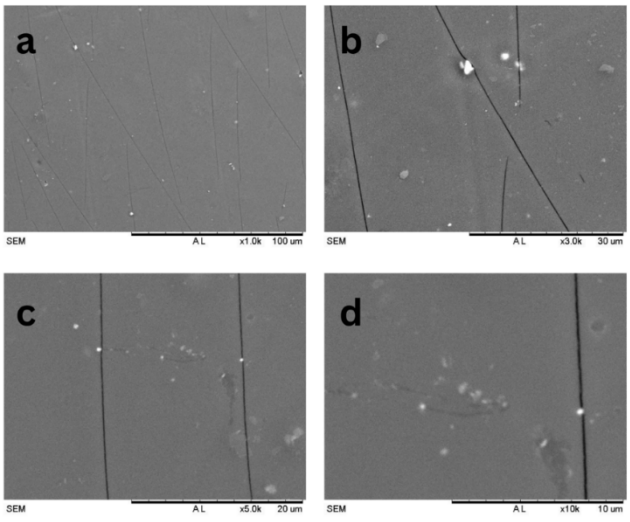

Figure 4 SEM micrographs of 0.25 M Kapton/Co_3O_4 nanocomposite films at various magnifications (a) 1.0 K, (b) 3.0 K, (c) 5.0 K and (d) 10.0 K

Figure 6 illustrates that at a cobalt nitrate concentration of 0.75 M, slight variations in brightness and surface texture are observed across the sample surface. Although the overall surface coverage appears higher than at lower concentrations, morphology is notably less uniform, which may introduce localized defects and compromise surface homogeneity. Such irregularities could adversely affect charge transport by creating discontinuities in the conductive pathway (Budiyanto et al., 2023). Additionally, due to the limited resolution or low contrast between the cobalt oxide and the polymer matrix, the nanoscale Co_3O_4 particles are not distinctly visible in the SEM image possibly due to their small size or overlapping with the Kapton background (Mu et al., 2010).

Table 2: EDX values for the composite films are treated with 0.25 M, 0.50 M, and 0.75 M cobalt nitrate precursor concentrations

Concentration	Weight %		
	Co	C	O
0.25 M	28.240	46.345	25.302
0.50 M	31.925	36.816	30.558
0.75 M	51.646	21.734	26.405

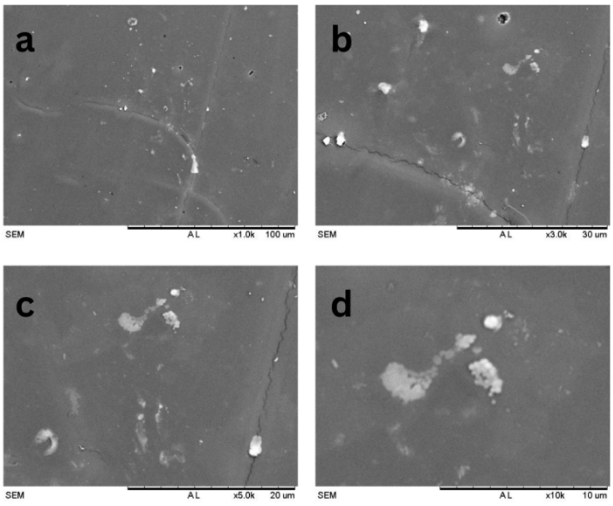

Figure 5 SEM micrographs of 0.50 M Kapton/Co_3O_4 nanocomposite films at various magnifications (a) 1.0 K, (b) 3.0 K, (c) 5.0 K and (d) 10.0 K

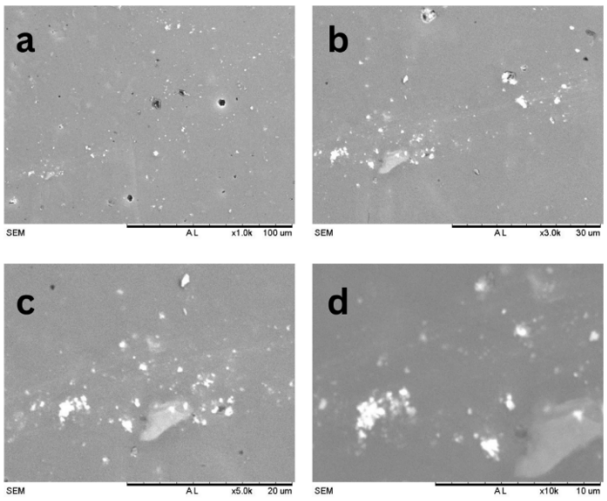

Figure 6 SEM micrographs of 0.75 M Kapton/Co_3O_4 nanocomposite films at various magnifications (a) 1.0 K, (b) 3.0 K, (c) 5.0 K and (d) 10.0 K

Elemental analysis of the 0.75 M sample reveals the highest cobalt content at 51.65 wt.%, accompanied by 26.41 wt.% oxygen and a markedly reduced carbon content of 21.73 wt.%. These results strongly suggest extensive surface coverage by cobalt oxide, likely forming a continuous or near-continuous layer over the Kapton substrate. The elevated cobalt levels and suppressed carbon signal are indicative of effective ion exchange and oxide formation, facilitated by the higher availability of precursor ions. This condition likely promotes both nucleation and lateral growth of cobalt oxide particles (W. Li, 2019; Mächler & Töpfer, 2017). However, the increased concentration may also lead to particle agglomeration or excessive film thickening, which could impact the mechanical flexibility and uniform electrical performance of the composite.

Although the SEM images provide limited morphological detail, the presence of cobalt oxide is strongly supported by FTIR-ATR, which confirms Co–O vibrational bands, and by XRD analysis, which clearly identifies crystalline Co_3O_4 phases. EDX further confirms cobalt incorporation, supporting the successful ion exchange and composite formation.

4.4. 4-Point Probe (4PP)

The electrical properties of the Kapton/Co_3O_4 composite films were systematically evaluated using the 4PP technique, which effectively minimizes contact resistance errors commonly encountered in two-point measurements. During testing, a constant current of 10 mA was applied to the outer probes, while the voltage drop was measured across the inner probes.

As summarized in Table 3, both the precursor concentration and film thickness significantly influenced the electrical behavior of the composite films. A reduction in thickness from 0.04 mm to 0.03 mm is observed at higher cobalt nitrate concentrations 0.50 M and 0.75 M, which is attributed to the densification of the cobalt oxide phase during thermal treatment. With increasing cobalt ion concentration, more Co^{2+} ions were incorporated into the hydrolyzed Kapton surface via ion exchange, promoting the nucleation and crystallization of Co_3O_4 nanoparticles. These nanoparticles formed a more compact and conductive surface layer upon thermal conversion, resulting in decreased film thickness and enhanced electrical performance. In contrast, pure Kapton remained at its original thickness due to the absence of chemical modification, ion exchange, or oxide formation. This contrast highlights the critical role of cobalt incorporation in driving structural densification and improved conductivity. These findings align with previous reports indicating that increased precursor concentrations promote more efficient oxide integration and surface compaction in polymer-based nanocomposites (Mu et al., 2010).

Table 3: Summarizes the average electrical conductivity and resistivity values for the composite films treated with 0.25 M, 0.50 M, and 0.75 M cobalt nitrate precursor concentrations

Concentration	Thickness (mm)	Sheet Resistance (Ω/sq)	Resistivity (Ω·cm)	Conductivity (S/m)
0.25 M	0.04	1.009×10^9	40.36	0.0248
		1.061×10^9	42.44	0.0236
		1.125×10^9	45.00	0.0222
		1.222×10^9	48.88	0.0205
		1.380×10^9	55.20	0.0181
Average		1.160×10^9	46.18	0.0218
0.50 M	0.03	3.051×10^8	9.15	0.1093
		3.079×10^8	9.24	0.1083
		3.086×10^8	9.26	0.1080
		3.095×10^8	9.29	0.1076
		3.096×10^8	9.29	0.1076
Average		3.081×10^8	9.25	0.1082
0.75 M	0.03	4.017×10^8	12.05	0.0830
		4.024×10^8	12.07	0.0829
		4.444×10^8	13.33	0.0750
		4.454×10^8	13.36	0.0748

	4.549×10^{8}	13.65	0.0733
Average	4.298×10^{8}	12.89	0.0778

Electrical conductivity of the Kapton/Co_3O_4 composites showed a strong dependence on cobalt precursor concentration. As presented in Table 3, conductivity increased significantly from 0.0218 S/cm at 0.25 M to 0.1082 S/cm at 0.50 M, representing nearly a fivefold enhancement. This improvement is attributed to the increased Co_3O_4 content, which facilitates more efficient charge transport pathways within the polymer matrix. The trend is consistent with EDX analysis (Table 3), which confirms higher cobalt incorporation at increased precursor concentrations, contributing to the formation of conductive networks within the film.

However, further increasing the cobalt nitrate concentration to 0.75 M led to a decline in electrical performance, with conductivity dropping to 0.0778 S/cm and resistivity increasing to 12.89 Ω·cm. This non-linear behavior is likely due to particle agglomeration at higher Co_3O_4 loadings, as evidenced by SEM observations (Figure 6) and EDX results. Agglomerated domains can disrupt the uniformity of conductive pathways, introduce charge carrier scattering sites and localize resistive regions, thereby reducing overall conductivity (H. Li et al., 2023). Surface heterogeneity and potential variations in probe contact pressure during 4PP measurements may also contribute to measurement variability at higher loadings.

In addition, slight differences in film thickness across samples must be considered when interpreting conductivity values, since resistivity is directly proportional to thickness in 4PP analysis. Although reduced thickness at higher cobalt concentrations may amplify conductivity readings, the dominant factor influencing electrical performance remains the amount and distribution of Co_3O_4 within the Kapton matrix (Budiyanto et al., 2023).

Thus, the 4PP analysis confirms that cobalt oxide incorporation substantially improves the electrical conductivity of Kapton films. The optimal performance is achieved at a 0.50 M precursor concentration, where uniform Co_3O_4 dispersion and sufficient loading enabled efficient charge transport. These findings highlight the critical importance of tuning precursor concentration and film formation conditions to optimize the electrical properties of Kapton/Co_3O_4 composites for potential applications in flexible electronics, sensors, and conductive coatings.

Conclusion

This study investigates how varying cobalt salt concentrations influence the structural, and electrical properties of Kapton/cobalt oxide composite films. Using a simple and cost-effective method involving alkaline activation, cobalt nitrate immersion, and thermal treatment, composite films were successfully prepared at 0.25 M, 0.50 M, and 0.75 M concentrations. Structural analysis using FTIR-ATR confirmed that alkaline hydrolysis introduced reactive functional groups on the Kapton surface and Co_3O_4 formation peak at 663 cm^{-1} and 661 cm^{-1}, enabling cobalt oxide bonding. XRD results verified the formation of crystalline Co_3O_4 with improved crystallinity at higher precursor concentrations, while EDX analysis showed increasing cobalt content from 28.24 wt.% to 51.65 wt.%. Electrical measurements using the 4-point probe method revealed that conductivity improved significantly from 0.0218 S/cm at 0.25 M to 0.1082 S/cm at 0.50 M, before slightly decreasing to 0.0778 S/cm at 0.75 M, likely due to particle agglomeration affecting charge transport. Variations in film thickness also influenced resistivity but to a lesser extent. Overall, the findings demonstrate that precursor concentration plays a key role in shaping the material's performance, with 0.50 M identified as the optimal concentration for achieving a balance between cobalt oxide loading, structural uniformity, and electrical conductivity. This work provides valuable insight into the design of flexible, conductive composite films and offers a practical approach for tailoring their properties for use in electronics, sensors, and coatings.

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