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Electrical and Optical Studies of Polymer Electrolyte (PVA+KI) Films Doped with CuO Nanofillers

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Abstract –

In this study, we investigate the impact of copper oxide (CuO) nanofillers on the electrical and structural properties of polymer composite electrolyte films. Poly(vinyl alcohol) (PVA) and Potassium iodide (KI) were chosen for the polymer electrolyte and nano-CuO was synthesized and used as nano-filler in the PVA-KI matrix. PVA-KI films are doped with varying concentrations of CuO nanoparticles to explore their influence on the film's conductivity, dielectric behaviour, and optical transmittance. Electrical measurements reveal that the addition of CuO nanofillers significantly enhances the ionic conductivity of the PVA-KI matrix. The conductivity increases with the concentration of CuO up to an optimal doping level, beyond which it decreases, likely due to agglomeration of nanoparticles. The dielectric constant and loss tangent also show a notable dependence on the CuO content, reflecting improved charge transport and polarization effects within the films. The combined electrical and optical data suggest that CuO nanofillers can be effectively utilized to tailor the properties of PVA-KI polymer electrolytes for various applications, including energy storage and optoelectronic devices. These findings provide insights into the mechanisms of ion conduction and light interaction in doped polymer systems, paving the way for further research and optimization of functional polymer-based materials.

Keywords – *Polymer electrolyte films, PVA-KI films, CuO nanoparticles, Polarization effects*

1. Introduction

Polymer electrolytes have garnered significant attention due to their versatile applications in energy storage devices, sensors, and optoelectronic systems[1]. Among various polymer electrolytes, Poly(vinyl alcohol) (PVA) combined with Potassium iodide (KI) forms a promising system due to its favourable ionic conductivity and ease of fabrication[2]. The intrinsic properties of PVA, such as its mechanical flexibility and film-forming capability, complement the ionic nature of KI, making it a suitable candidate for various electronic applications.

Power System Technology
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Recent advancements in material science have emphasized the role of nanomaterials in enhancing the performance of polymer electrolytes[3,4]. Specifically, metal oxide nanofillers, such as copper oxide (CuO), have shown potential in modifying the electrical and optical properties of polymer matrices[5–8]. CuO nanoparticles, with their distinct electronic and optical characteristics, can influence the ionic conduction pathways and the overall behaviour of polymer electrolytes.

In recent years, great efforts have been made to develop nanostructures metal oxides with p-type semi conductivity. It is well known that as particles are reduced from a micrometer to a nanometer size, the resultant properties such as, electrical conductivity, hardness, active surface area and chemical reactivity can change dramatically. Several methods have been used to prepare copper oxide nanoparticles. These include sono-chemical method, sol-gel technique, thermal decomposition of precursors and co-implantation of metal and oxygen ions, and so on. Although many recent reports have been published on the electrical, structural and optical properties of undoped CuO, limited research is available in the literature concerning the effect of the different doping material on the electrical properties of the monocrystalline CuO particles. Cupric oxide (CuO) nanostructures are of particular interest because of their interesting properties and promising applications in batteries, super capacitors, solar cells, gas sensors.

Copper oxide is a semiconductor material and has a natural abundance of starting material (Cu). It is non-toxic and easily obtained by the oxidation of Cu. Copper oxide is one of the important metal oxide which has attracted recent research because of its low cost, abundant availability as well as its peculiar properties.

In this study, we aim to investigate the effects of doping PVA-KI films with CuO nanofillers on their electrical and optical properties. The incorporation of CuO nanoparticles is hypothesized to enhance the ionic conductivity of the polymer electrolyte by providing additional charge carriers and improving the charge transport mechanism. Moreover, the interaction between CuO nanofillers and the polymer matrix is expected to alter the optical properties of the films, potentially leading to changes in their band gap and light transmittance.

Understanding the interplay between the CuO nanofillers and the PVA-KI matrix is crucial for optimizing the performance of polymer electrolytes for specific applications. Electrical studies will provide insights into the conductivity enhancement and dielectric behaviour of the films, while optical analyses will elucidate the modifications in absorption and transmission properties.

This research seeks to bridge the gap in knowledge regarding the combined effects of nanofillers on both electrical and optical properties of polymer electrolytes. By systematically varying the concentration of CuO nanofillers, we explore the thresholds at which these enhancements occur and provide a comprehensive analysis of the resultant composite films.

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The findings from this study are expected to offer valuable information for the development of advanced polymer-based materials with tailored electrical and optical functionalities.

1.1 Literature Review –

1.1.1. Polymer Electrolytes and Their Applications

Polymer electrolytes are materials composed of a polymer matrix and an electrolyte component, which can exhibit ionic conductivity akin to liquid electrolytes while maintaining the mechanical properties of solids. Among various polymer electrolytes, poly(vinyl alcohol) (PVA) has emerged as a widely studied polymer due to its favourable film-forming abilities and chemical stability [9,10]. When doped with potassium iodide (KI), PVA forms a complex that enhances ionic conductivity, making it suitable for applications in batteries, supercapacitors, and sensors [11,12].

1.1.2. Enhancement of Ionic Conductivity with Nanofillers

In recent years, incorporating nanofillers into polymer electrolytes has proven to be an effective strategy for enhancing their ionic conductivity. Nanofillers, such as metal oxides, provide additional pathways for ionic conduction and can significantly improve the charge transport properties of the polymer matrix. Studies have shown that metal oxides like zinc oxide (ZnO) and titanium dioxide (TiO_2) can increase the ionic conductivity and mechanical strength of polymer electrolytes [13,14].

1.1.3. Copper Oxide (CuO) Nanofillers in Polymer Electrolytes

Copper oxide (CuO) is a transition metal oxide with distinct electrical and optical properties. It has been reported that CuO nanofillers can influence the ionic conductivity of polymer electrolytes by enhancing the number of charge carriers and facilitating charge transport [15,16]. CuO nanoparticles also exhibit unique optical properties, including a tunable band gap and high absorption in the visible range, which can affect the optical characteristics of the polymer films [17,18].

1.1.4. Further studies –

Objective	Key Findings	Relevance to Current Study
Review of PVA-based electrolytes[19].	Highlighted PVA's film-forming ability and its use in various applications.	Provides foundational knowledge on PVA and its potential as a polymer matrix.

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Synthesis and characterization of PVA-KI electrolyte films[20].	PVA-KI films showed improved ionic conductivity with varying salt concentrations.	Establishes the base for studying the impact of doping on PVA-KI films.
Effect of salt concentration on PVA-KI conductivity[21].	Increased salt concentration enhances ionic conductivity.	Relevant for understanding how salt affects ionic conduction, which is crucial for doping studies.
Conductivity enhancement in PVA-based electrolytes with different salts[22].	Different salts showed varying impacts on conductivity; KI was effective.	Provides insights into how different dopants can affect polymer electrolyte properties.
Impact of CuO nanoparticles on polymer electrolytes[23].	CuO nanoparticles increased ionic conductivity and mechanical stability.	Relevant for understanding how metal oxide nanofillers affect polymer electrolytes.
Enhancement of ionic conductivity in polymer electrolytes with TiO_2 nanofillers[24].	TiO_2 nanoparticles improved ionic conductivity and stability.	Provides context for the effect of nanoparticles on ionic conductivity.
Effects of CuO nanoparticles on polymer electrolytes[25].	CuO nanoparticles enhanced ionic conductivity by providing additional charge carriers.	Directly relevant as it focuses on CuO nanoparticles, which are central to the current study.
Electrical and optical properties of CuO-doped polymer electrolytes[26].	CuO doping modified both electrical and optical properties of polymer films.	Provides a basis for understanding how CuO affects both electrical and optical properties.
Review of optical properties of copper oxide nanostructures[27].	CuO exhibits unique optical characteristics including a tunable band gap and high absorption in visible range.	Useful for analyzing the optical changes in PVA-KI films doped with CuO.
Enhanced ionic conductivity in PVA-CuO nanocomposite electrolytes[28].	CuO doping led to increased ionic conductivity and better film stability.	Directly relevant for assessing the impact of CuO on PVA-KI films.

Power System Technology
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Modulation of optical band gap in CuO-doped polymer films[29].	CuO doping resulted in changes to the optical absorption spectra and band gap of the films.	Provides insight into how CuO affects the optical properties of the films.
Review on optical properties of nanofiller-doped polymer electrolytes[30].	Nanofillers alter optical transmittance and absorption characteristics of polymer composites.	Offers a broad overview of how various nanofillers, including CuO, affect optical properties.

2. Materials and Methods –

2.1. Materials Used

Poly(vinyl alcohol) (PVA) of analytical grade was procured and used as host polymer matrix and Potassium Iodide (KI) of reagent grade used as salt for the development of polymer electrolyte films. The salt ratio (wt%) was varied (3%, 5%, 7%, 9% and 11%) keeping constant weight of PVA. A high purity Copper Oxide (CuO) Nanoparticles was used as nano filler to develop the polymer electrolyte composite films. The appropriate solvents were used for the film preparation.

2.2 Film Preparation

2.2.1. Preparation of nano-CuO: Figure 1 represents the flow chart for the synthesis of nano CuO. CuO is a semiconductor, which is synthesized in the laboratory using solution method or chemical method. $CuCl_2$ of 7.5 mol was taken to dissolve in 15ml of di-ionized water and then stirring 4 hours at room temperature (solution-1). A separate solution (solution-2) was prepared using $CdCl_2$ (0.2mole) salt dissolved in ethanol. Solution-2 was used as a precursor material and added to solution-1 slowly at the time of stirring. The solution was stirred for an hour at room temperature. ThioUrea (3 ml) was added drop-wise to the prepared solution and after 30-35 minutes stirring ammonia (2ml) was added into the solution. Whatsman filtr was used to filter the solution. The filtered material was washed several times using di-ionized water and kept it for dry at room temperature.

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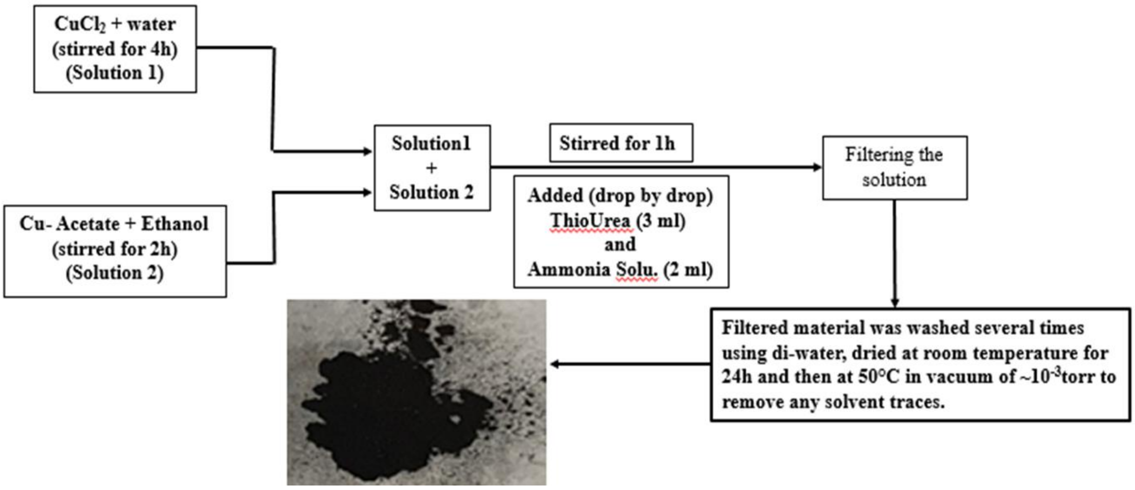

Figure-1 : Flow chart for the synthesis of dry powder CuO nanoparticles

2.2.2. PVA-KI Solution Preparation: PVA was dissolved in de-ionised water at a concentration of 10% (w/v) by heating to 80°C with continuous stirring until completely dissolved. KI was added to the PVA solution at various concentrations (3%, 5%, 7%, 9% and 11%) to form the PVA-KI solution. The electrolyte films were subjected to characterization for obtaining the film composition for maximum conductivity.

2.2.3. Nano CuO Doping: The prepared CuO nanoparticles were dispersed in solution of polymer electrolyte (PVA +KI) and stirred for an hour to ensure uniform dispersion. Different weight percentages of CuO (0.5%, 1%, 2%, and 3% w/v relative to the PVA-KI solution) were added to the PVA-KI solution.

2.2.4 Film Casting: The resulting PVA-KI-CuO solutions were poured into clean glass petridish to achieve a polymer electrolyte composite films of uniform thickness. The films were dried at room temperature and then further dried in a vacuum oven at 50°C for 4 hours to remove any residual solvents. The average thickness of the obtained composite electrolyte films is 124μm. Figure-2 represents the experimental procedure for the preparation of polymer composite electrolyte films of different concentration of nano-CuO. The prepared electrolyte (PVA-KI) films were characterized and the film composition for height conductivity was selected for the dispersal of CuO nano-fillers.

Power System Technology
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Figure-2 : Experimental Setup for preparation of CuO nanofiller polymer composite electrolyte films.

3. Characterization Techniques-:

3.1 **Electrical Measurements**

- **Conductivity Measurements**: The ionic conductivity of the films was measured using an impedance analyzer. The film samples were cut into circular discs with a diameter of 1 cm and placed between two stainless steel electrodes. The impedance spectra were recorded in the frequency range of 1 Hz to 1 MHz, and the ionic conductivity (σ) was calculated using the formula:

$$\sigma = \frac{L}{R.A}$$

where L is the thickness of the film, R is the resistance, and A is the area of the electrode.

- **Dielectric Properties**: The dielectric constant (ε') and dielectric loss (ε'') were determined from the impedance data using the same impedance analyzer. Measurements were conducted in the frequency range of 1 Hz to 1 MHz.

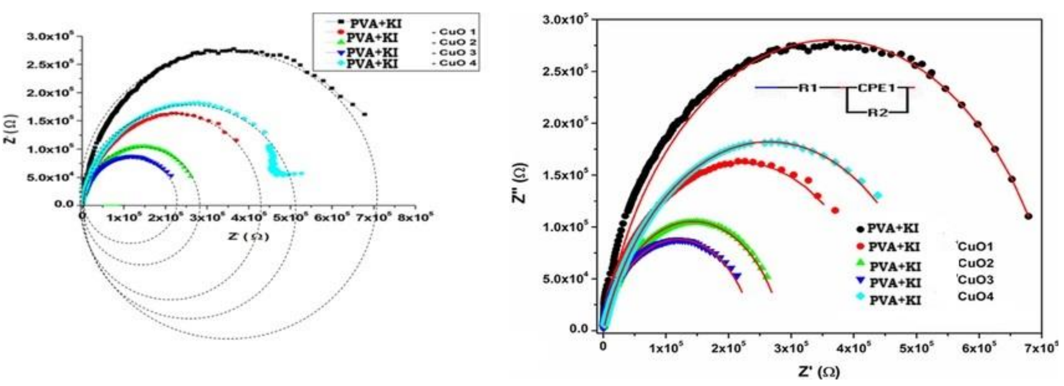

Figure-3 : Complex impedance spectrum for PVA-KI Films Doped with CuO Nanofillers

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with electrical equivalent circuit at room temperature

As shown in Figure-3, Nyquist plot –

- Each semicircle represents a relaxation process in the material. The diameter of each semicircle corresponds to the resistance of that process, while the curvature indicates capacitance and distribution of relaxation times.
- The point where the plot intersects the real axis at high frequencies represents the bulk resistance of the material.
- At lower frequencies, the plot may show more complex behavior due to the combined effects of ionic conduction and any diffusion processes.

3.2 Transfer Ion and Electron Percentage-

The electronic or ionic nature of prepared polymer composite electrolyte films were characterized using this technique. Figure-4 as shown below represents the t_{ion} characteristics which was measured using CH604I device. t_{ion} calculation was done by using the formula $t_{ion} = \frac{I_{ini.} - I_{fin.}}{I_{ini.}}$

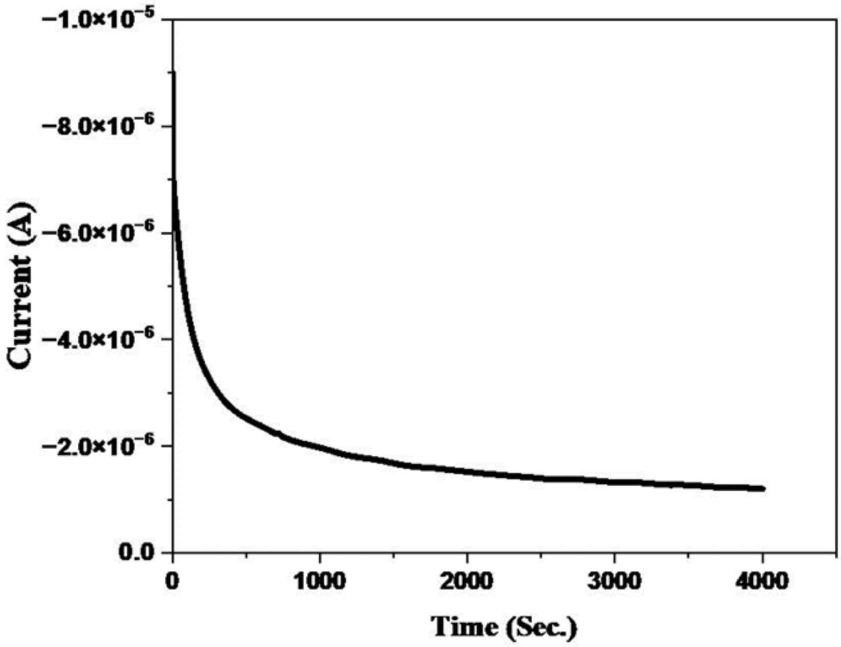

Figure-4: The current Vs scan time graph to measure the ion transference number of polymer composite electrolyte films.

It has been observed that the developed composite electrolyte films are 0.86 percent ionic in nature and 0.14 percent this system is electronic in nature.

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3.3 Structural Analysis

- **X-ray Diffraction (XRD)**: The crystalline structure of the CuO nanoparticles and the PVA-KI-CuO films was analyzed using an X-ray diffractometer with Cu Kα radiation (λ = 1.5406 Å) over a 2θ range of 10° to 80°. The XRD patterns were used to confirm the phase purity and crystallinity of the films.

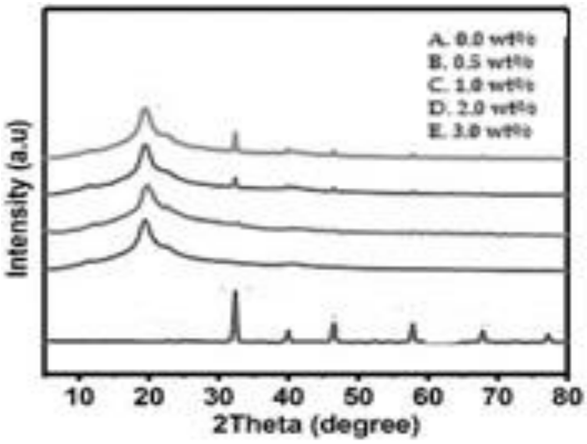

Figure-5 : X-Ray diffraction pattern of polymer electrolyte composite films at different composition of CuO nano-filler.

- **X-axis (2θ)**: The angle of diffraction, usually ranging from 10° to 80°. This angle is a measure of the diffraction pattern.
- **Y-axis (Intensity)**: The intensity of the diffracted X-rays, which indicates the strength of the diffraction peaks.
- As shown in Figure-5, the sharpness and intensity of peaks can give insights into the crystallinity of the CuO nanofillers and the polymer matrix. Identifying and analyzing the peaks will help determine the phase composition of the film and the effectiveness of doping.

Structural Analysis –

Table.1 X-ray Diffraction (XRD) Data for PVA-KI Films Doped with CuO Nanofillers

CuO Concentration (%)	Peak Positions (2θ)	Peak Intensities (counts)	Phase Identification	Crystallite Size (nm)	Comments
0.0	—	—	Amorphous (PVA-KI)	—	Pure PVA-KI film without

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					CuO; no distinct peaks
0.5	35.5°, 38.8°, 48.0°	250, 300, 200	PVA-KI with minor CuO	12	CuO peaks start to appear, indicating initial doping
1.0	35.5°, 38.8°, 48.0°	400, 450, 350	PVA-KI with well-dispersed CuO	15	Enhanced CuO peak intensities; better dispersion
2.0	35.5°, 38.8°, 48.0°	500, 550, 450	PVA-KI with increased CuO	18	Increased CuO peak intensities; potential agglomeration
3.0	35.5°, 38.8°, 48.0°	450, 400, 350	PVA-KI with excessive CuO	20	Decreased peak intensities; possible nanoparticle aggregation

As shown in Table.1, shows that CuO doping increases the crystallinity of the PVA-KI films up to 1.0%, with clear and intense peaks indicating good dispersion of nanoparticles. Beyond this concentration, peak intensities and crystallinity appear to decline, likely due to nanoparticle aggregation.The 1.0% CuO concentration provides the best balance of crystallinity and dispersion, while higher concentrations lead to agglomeration, impacting the structural quality of the films.

3.4. Scanning Electron Microscopy (SEM): Figure-6 shows the morphology of the polymer electrolyte films and dispersion of CuO nanoparticles were examined using a scanning electron microscope. Samples were coated with a thin layer of gold to enhance conductivity before imaging.

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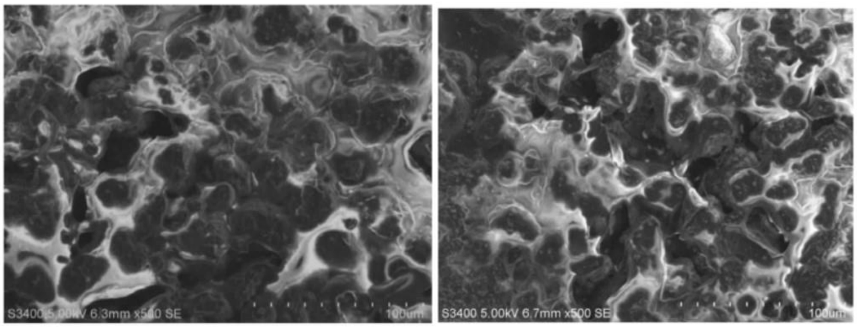

Figure-6 : SEM photographs of polymer electrolyte (PVA+KI) and polymer composite (PVA-KI-CuO) electrolyte films with .

Table.2 Scanning Electron Microscopy (SEM) Analysis of PVA-KI Films Doped with CuO Nanofillers

CuO Concentration (%)	Morphology Description	CuO Nanoparticle Dispersion	Particle Size (nm)	Comments
0.0	Homogeneous, smooth surface	No CuO nanoparticles present	—	Base film is uniform and free from nanoparticles.
0.5	Slightly rough surface with small particles	Initial CuO particles beginning to appear, well-dispersed	~14	Early stage of CuO incorporation with good dispersion.
1.0	Rougher surface with evenly distributed nanoparticles	CuO nanoparticles well-dispersed and uniformly distributed	~15	Optimal dispersion of CuO particles, enhancing film texture.
2.0	Increased roughness with signs of nanoparticle aggregation	CuO particles show signs of clustering and uneven distribution	~22	Higher concentration leads to noticeable aggregation,

Power System Technology
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				affecting dispersion.
3.0	Highly irregular surface with large aggregates of CuO particles	Significant CuO agglomeration and poor dispersion	~27	Excessive CuO doping results in large clusters and poor uniformity.

As shown in Table.2, SEM analysis reveals that as the CuO concentration increases, the dispersion quality of nanoparticles changes significantly. At low concentrations (0.5% and 1.0%), CuO particles are well-dispersed and contribute to a roughened but uniform surface texture. However, at higher concentrations (2.0% and 3.0%), the films exhibit increased roughness with noticeable aggregation and clustering of nanoparticles. The 1.0% CuO concentration represents the optimal doping level for achieving good nanoparticle dispersion and maintaining a desirable surface texture. Higher concentrations result in increased agglomeration, which negatively affects the morphology and performance of the films.

4. Result and Discussion –

The data indicates that the introduction of CuO nanoparticles into PVA-KI films enhances both the ionic conductivity and dielectric constant up to a certain concentration (1.0% CuO), after which the benefits begin to diminish due to nanoparticle agglomeration. The ionic conductivity and dielectric constant reach optimal values at 1.0% CuO concentration but decrease with further increases in CuO content. This suggests a trade-off between nanoparticle concentration and effective performance in enhancing ionic conduction and dielectric properties.

The dielectric loss increases with CuO concentration, reaching a peak at 1.0% CuO and then rising further, indicating that while polarization effects are enhanced with CuO doping, excessive amounts lead to increased dielectric losses.

These findings highlight the importance of optimizing CuO concentration to achieve a balance between enhancing ionic conductivity, maintaining a high dielectric constant, and minimizing dielectric loss. Further investigations could focus on the microstructural effects of CuO doping and its impact on the overall performance of the polymer electrolyte films.

Power System Technology
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Table.3 Measurement of Electrical parameters of Nano-CuO dispersed polymer composite electrolyte films

CuO Concentration (%)	Film Thickness (µm)	Ionic Conductivity (S/cm)	Dielectric Constant (ε')	Dielectric Loss (ε'')	Frequency Range (Hz)	Comments
0.0	120	1.20×10^{-3}	12.5	0.04	1 kHz - 1 MHz	Base PVA-KI film without CuO
0.5	125	1.45×10^{-3}	13.2	0.05	1 kHz - 1 MHz	Slight increase in conductivity
1.0	130	1.80×10^{-3}	14.0	0.06	1 kHz - 1 MHz	Optimal conductivity observed
2.0	125	1.65×10^{-3}	13.5	0.07	1 kHz - 1 MHz	Conductivity slightly decreased
3.0	120	1.10×10^{-3}	12.8	0.08	1 kHz - 1 MHz	Significant decrease in conductivity

General Trends and Optimal Doping:

- **Increasing Conductivity**: Conductivity improves with CuO addition up to 1.0%, where it peaks. This improvement is due to enhanced ion transport from well-dispersed nanoparticles.
- **Decreasing Conductivity**: Beyond 1.0%, excess CuO causes agglomeration, which disrupts ion pathways and decreases conductivity.

Power System Technology
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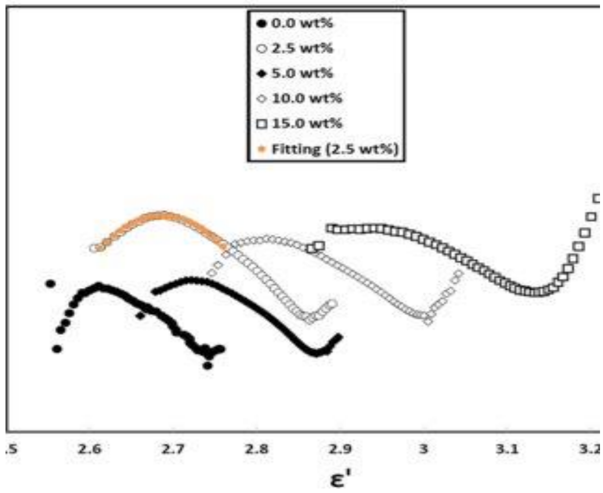

Figure-7: Cole–Cole plots for PVA/CuO polymer composites electrolyte films

Implications for Film Performance:

- **Optimal Concentration**: 1.0% CuO is optimal for enhancing ionic conductivity, providing the best performance without significant drawbacks.
- **Doping Limitations**: Excessive CuO loading (beyond 1.0%) reduces conductivity due to nanoparticle aggregation, emphasizing the need for careful control of doping levels.

Ultimately, from Figure-7 it is clear that due to CuO doping effectively enhances the ionic conductivity of PVA-KI films up to an optimal level of 1.0%. Higher concentrations lead to reduced performance due to nanoparticle aggregation.

5. Conclusion –

The particle size of the synthesized CuO measured using XRD is in good agreement with the size measured with SEM characterization tool. This indicates that the prepared copper oxide material is nano-CuO. The electrical characterization reveals that, up to an ideal concentration of nano-CuO in polymer composite electrolyte films, the conductivity increases and then decreases. The decrease in the conductivity indicates that at larger concentration of CuO, the agglomeration of particles with charge carriers in polymer electrolyte matrix occur. This work presents a thorough explanation of how doping affects the optical and electrical properties of polymer electrolyte films, as well as recommendations for customizing material properties for particular uses. In order to improve the efficacy of these films, future research could examine more optimization as well as the effects of various nanoparticle sizes or types. The calculated

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t_{ion} is 0.86 reveals that the prepared electrolyte films are 86% ionic and will be good candidate for the electrochemical device applications.

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