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RESEARCH PAPER

Synthesis and Study Implications of Including Cobalt Oxide Nanoparticles on Electrical and Thermal Properties of PVA/PAA Blend Films

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ABSTRACT

Pure PVA/PAA polymer films, manufactured by solution casting technique, reinforced with cobalt oxide nanoparticles (CoO NPs) calcined at temperatures (800 ᵒC), at different weight percentages (pure, 1, 3, 5, 7 and 9) wt%. XRD analysis of the as-synthesized nanomaterial powder indicated that cobalt oxide nanoparticles were obtained at the calcination temperature (800 °C). Heat treatment promotes continued crystallization, leading to an expansion in the dimensions of nanoparticles. The electrical properties revealed a substantial enhancement in both the (ε'), (tanδ) and σ (a.c.) of the reinformed polymer blends. The thermal properties of the films reinforced with cobalt oxide nanoparticles show an increase in the thermal conductivity (K) of the prepared films with increasing reinforcement ratios.

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INTRODUCTION

Novel polymeric nanocomposites are a class of materials known for their distinct combination of nanostructures and polymeric composites. Their unique characteristics do not stem from their nature, but from the relationship between them [1-4]. The widespread use of polymeric materials has led to the development and production of polymeric compounds. The significance of polymers is intrinsically related to their perception as economical and easy-to-manufacture materials [5,6]. Nanoparticles have a higher surface area to volume, making them more intereactive. the It is atoms of the material on the surface that determined the reaction of that substance, as these atoms directly come into contact with another substance [7]. Nanotechnology has entered many fields, including display screens, stain-resistant clothing manufacturing, health and automotive. Emphasis has been placed on organic/inorganic composite nanomaterials with diverse structures in recent years. Composite materials resulting from the combination of organic and inorganic materials will have the advantages of both inorganic components (such as high thermal stability, stiffness, strength, high refractive index, and rigidity) and organic polymers (such as insulation, softness, and flexibility), creating wide-ranging uses in several fields. [8,9]. Polymers buffer applications include printed circuit boards, wire sheathing materials, corrosion-protective electronic devices, and cable sheathing materials. In the microelectronics manufacturing industry, polymers are used in the photolithography process [10]. Polyvinyl alcohol (PVA) is an important polymer due to its unique physical and chemical properties. This polymer

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can be manufactured in powder, film, and fiber forms. Polyvinyl acetate (PVA) is a semi-crystalline polymer composed of hydrogen bonds and hydroxyl (OH) bonds. PVA that are soluble in water and is characterized by good flexibility and high transparency. Industrially, it is used in adhesives and sizing materials, and in medical substances such as drug and membranes delivery systems. It is known as a medical substance because of its agree with the body [11]. Polymer compounds are characterized by their light weight, high stiffness and strength, and high corrosion resistance. In recent years, much research has been conducted on composite materials reinforced with natural fibers in various fields. [12,13]. In our research, we aim to study the electrical and thermal properties of nanocomposites (PVA/PAA:CoO).

MATERIALS AND METHODS

Preparation Of (CoONPs)

The synthesis of CoO NPs or cobalt oxide nanoparticles, was performed using a chemical precipitation method [14]. The desired concentration of cobalt nitrate was $Co(NO_3)_2.6H_2O$, with a molecular weight of 291.04 g/mol. 29.103 g of cobalt nitrate, $Co(NO_3)_2.6H_2O$, was dissolved in 100 ml of (distilled water). Use a magnetic stirrer to stir the mixture at a temperature of 50 °C until a homogeneous mixture was obtained. A 25% ammonium hydroxide (NH_4OH) solution was gradually added, and a precipitate was formed. The precipitate was cleaned. It was oven-dried after being repeatedly rinsed with distilled water at 200 °C. The resulting precipitate was placed in a crucible and calcined at 800 °C to obtain cobalt oxide nanoparticles (CoO NPs).

Creating Nanocomposites of PVA/PAA:CoO

Pure blend of PVA and PAA polymer and polymers Samples of nanocomposite were made using a straightforward technique “solution casting”. The blend film PVA/PAA has been prepared by mixture PVA (70%) and PAA (30%). To prepared PVA/PAA:CoO nanocomposites films with varying percentages of weight (pure, 1, 3, 5, 7, and 9), by added 0.5 g of cobalt oxide nanoparticale to 30 ml of distilled water [15]. Pure blend of PVA and PAA polymer and polymers Samples of nanocomposite were made using a straightforward technique “solution casting”. The blend film PVA/PAA has been prepared by mixture PVA (70%) and PAA (30%). To prepared PVA/PAA:CoO nanocomposites films with varying percentages of weight (pure, 1, 3, 5, 7, and 9), by added 0.5 g of cobalt oxide nanoparticale to 30 ml of distilled water [15]. Electrical insulation tests were performed on the prepared pure polymer films and composite films using an Agilent Impedance Analyzer 4294A, an LCR meter manufactured by Tawan CompanyThe Lee disc, manufactured by George & Griffin, was used to measure the thermal conductivity of pure and composite polymer films. Use a three-disc (A, B, C) li-disc and an electric heater connected to an electric circuit to calculate the thermal conductivity (K). Both devices are located at the College of Science, University of Diyala, Iraq.

The crystal size of the samples is is computed using the highest XRD peak utilizing Scherrer’s equation as shown in Eq. 1 [16].

$$D = \frac{0.9\,\lambda}{\beta\cos\theta} \tag{1}$$

to calculate the dielectric constant (ε′) using the Eq. 2 [17].

$$\varepsilon'_r = C' d_{dis}/\varepsilon_o A \tag{2}$$

The dielectric loss () is given by the Eq. 3 [18].

$$\varepsilon''_r = \tan\delta * \varepsilon'_r \tag{3}$$

using the Eq. 4 [19]. The AC conductivity (σa.c) was calculated.

$$\sigma_{a.c} = \omega\,\varepsilon_o\,\varepsilon''_r \tag{4}$$

the thermal conductivity (k) is determined using the Eq. 5 [20].

$$k[(T_B-T_A)/ds]=e[T_A+r/2(d_A+ds/4)T_A+dsT_B/2r] \tag{5}$$

RESULTS AND DISCUSSION

Analysis of Structures

X-ray Diffraction (XRD)

XRD test has been used to done to investigate the structure type (phase) and the crystalline size of prepared CoO NPs using the chemical precipitation method. Fig. 1 presents the obtained XRD patterns of prepared CoO NPs.

After calcination of CoO nanoparticles at temperatures (800 °C), several characteristic peaks were observed at (2θ = 19.001°, 31.27°, 36.84°, 44.81°, 59.35°, and 65.23°) of (111), (200), (311), (400), (511), and (440). The detected characteristic peaks confirmed the formation of cubic structure of CoO nanoparticles with stereogenic group (Fd3m No. 227) and lattice parameters (a = 8.1290 Aº, b = 8.1290 A°, c = 8.1290 A°) and (α = β = γ = 90°) which are consistent with the usual data (ICDD). (00-043-1003) [14].

Electrical Properties

Dielectric Constant (ε')

Calculate the insulation constant (ε') values using Eq. 2, prepared pure polymeric films (PVA/PAA) supported with cobalt oxide nanoparticles (CoO NPs) with different weight percentages (pure, 1, 3, 5.7 and 9), calcined at a temperature (800 °C), The results showed that increasing the frequency values offset by a reduction in values (ε›) for all the prepared strengthened and pure films [21, 22].

Most polymeric materials have a decreasing value of the dielectric constant with increasing frequency [23,24].

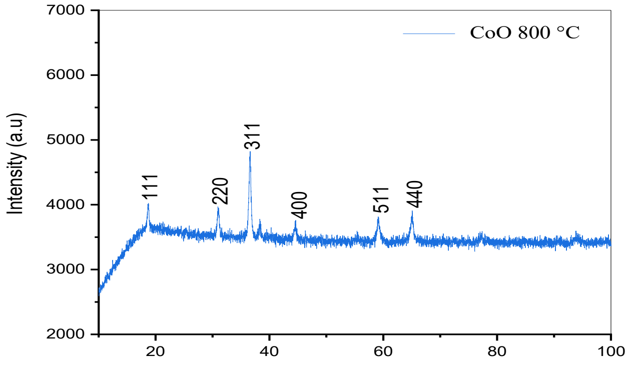

Fig. 1. XRD patterns of prepared CoO NPs 800 °C, Miller coefficients (440).

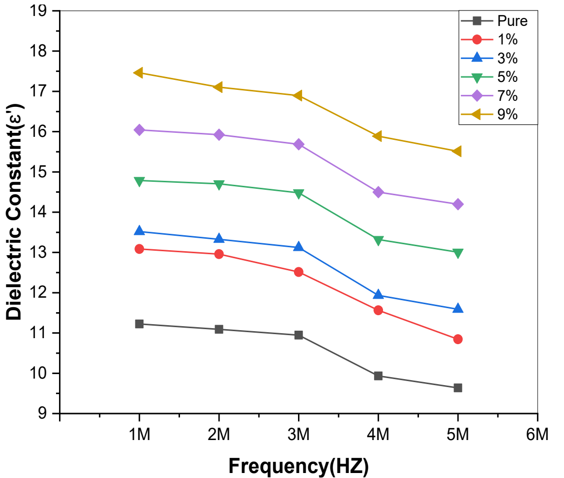

Fig. 2. Change of the dielectric constant as a function of frequency for pure (PVA/PAA) films supported with cobalt oxide nanoparticles (CoO NPs) calcined at (800 °C).

Table 1. Calculated XRD parameter of synthesized CoO NPs 800 °C.

Sample	2θ (°) Standard	2θ (°) Practical	FWHM (deg)	D (nm)	(hkl)
CoO NPs at 800 C°	36.83	36.53	0.4149	20.16	(311)

Also, the results showed an increase in the constant values of the electrical insulation of all prepared films (PVA/PAA) at every frequency by adding cobalt oxide nanoparticles (CoO NPs) to the mixture. As the percentage of nanomaterial increases, The dielectric constant values increase., as the membrane (PVA/PAA: 9wt% CoO NPs at 800 °C) achieves the higher value than dielectric constant. Electrical insulation, due to crystalline defects formed due to the presence of nanoparticles, This leads to the formation of multiple interfaces and this increases the (ε’) [25,26].

Dissipation Factor (tanδ)

Knowing and studying the dielectric loss coefficient (tanδ) is one of the parameters that are directly related to the applications of composite materials from the polymer. As (tanδ) is defined, a scale of the ratio of energy loss that passes in the insulating material to the total energy that passes through the dielectric. The insulation loss

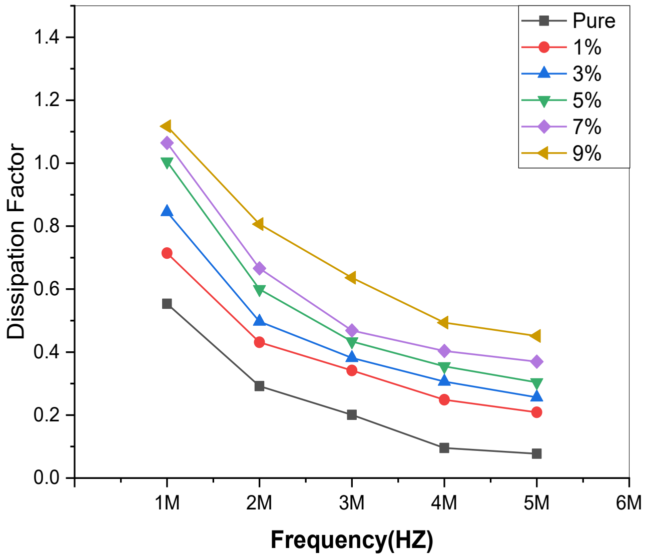

Fig. 3. Frequency (tanδ) shift of pure PVA/PAA films supported by cobalt oxide nanoparticles (CoONPs) annealed at 800 °C.

Table 2. (ε›) values for films prepared for different frequencies.

	Dielectric Constant (ε’) at 800 °C					
Frequency (MHZ)	(PVA/PAA): pure	(PVA/PAA): 1 wt% CoONPs	(PVA/PAA): 3 wt% CoONPs	(PVA/PAA): 5 wt% CoONPs	(PVA/PAA): 7 wt% CoONPs	(PVA/PAA): 9 wt% CoONPs
1	10.96	12.55	13.12	14.76	16.04	17.47
2	11.07	13.05	13.33	14.74	15.94	17.13
3	11.26	13.35	13.53	14.51	15.67	16.90
4	9.64	10.82	11.60	13.01	12.24	15.51
5	9.96	11.60	1196	13.33	14.51	15.90

Table 3. Dielectric loss factor values for membranes prepared at different frequencies.

	(tanδ) at 800 °C					
Frequency (MHZ)	(PVA/PAA): pure	(PVA/PAA): 1 wt% CoONPs	(PVA/PAA): 3 wt% CoONPs	(PVA/PAA): 5 wt% CoONPs	(PVA/PAA): 7 wt% CoONPs	(PVA/PAA): 9 wt% CoONPs
1	0.69358	0.81456	0.84502	1.00527	1.06439	1.11717
2	0.3419	0.37141	0.40713	0.43967	0.56599	0.70631
3	0.3367	0.36179	0.38172	0.43384	0.46839	0.63636
4	0.0457	0.27893	0.3065	0.39502	0.4536	0.46353
5	0.03707	0.24882	0.29631	0.3337	0.36966	0.45125

coefficient is proportional to the dissipated power within a range between (1 MHz) and (5 MHz) directly. The insulation loss coefficient values for the prepared films were calculated using Eq. 3.

In the results, it was observed that the values of (tanδ) for the grafted and pure films decreased with increasing weight percentages, Table 3 shows these values. This is attributed to the absorption of the energy of the dipoles within the structure of the prepared membranes in order to overcome the obstruction caused by the materials surrounding the dipoles, reduces the mobility of the diodes, so the charge carriers decrease with increasing frequency. So, dipoles will require higher energy for relaxation to occur, and therefore lower dielectric loss factor values (tanδ) [27]. Finally, it was found that reinforcement with CoONPs leads to an increase in the tanδ values for all prepared PVA/PAA films at each frequency [28,29].

A.C Electrical Conductivity ($\sigma_{a.c}$)

The alternating current electrical conductivity (σa.c) was calculated for all membranes as a function of the electric field frequency ranging

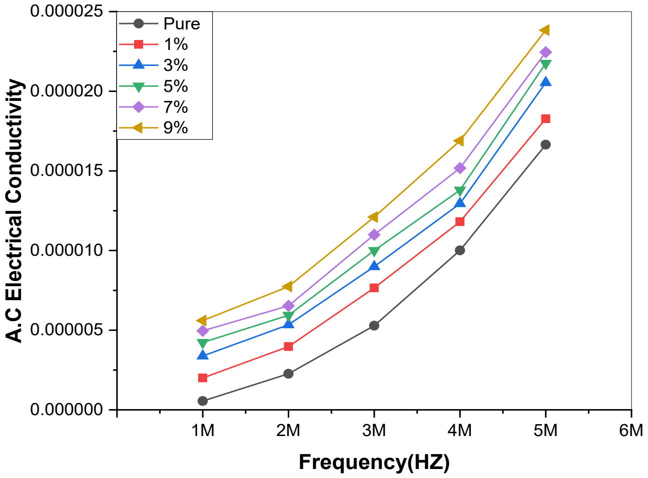

Fig. 4. Change in AC electrical conductivity as a function of frequency for pure PVA/PAA films supported by CoO NPs.

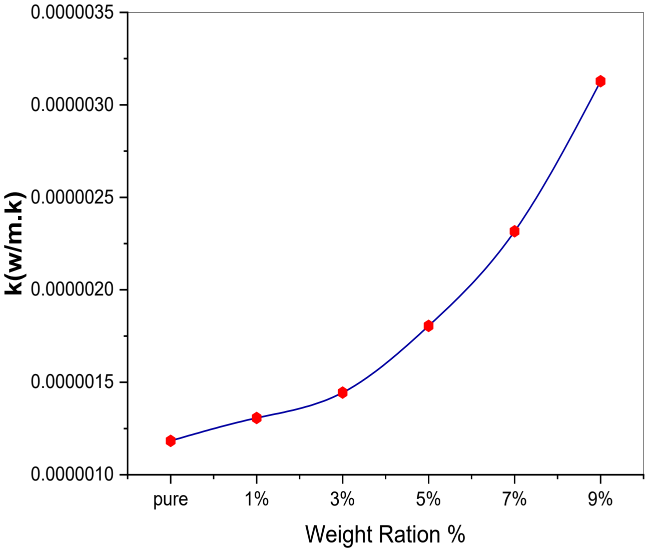

Fig. 5. Thermal conductivity coefficient of the prepared (PVA/PAA) films before and after reinforcement with cobalt oxide nanoparticles (CoO NPs).

Table 4. AC electrical conductivity coefficient values for membranes prepared at different frequencies.

A.C Electrical Conductivity ($\sigma_{a.c}$)*10^{-5} (S/ m) at 800 °C						
Frequency (MHZ)	(PVA/PAA): pure	(PVA/PAA): 1 wt% CoONPs	(PVA/PAA): 3 wt% CoONPs	(PVA/PAA): 5 wt% CoONPs	(PVA/PAA): 7 wt% CoONPs	(PVA/PAA): 9 wt% CoONPs
1	0.0555155	0.202134	0.338451	0.42317	0.495834	0.559958
2	0.226054	0.397022	0.534048	0.593477	0.653102	0.773782
3	0.5284	0.765472	0.899361	1.00001	1.09955	1.21046
4	1.00092	1.18073	1.29428	1.37919	1.51752	1.68903
5	1.66477	1.82703	2.05454	2.17537	2.24541	2.38324

Table 5. Thermal conductivity coefficient (K) values for the prepared models.

Sample	K×10^{-6} (W/m.K)
PVA/PAA: pure	1.18
PVA/PAA: 1% CoONPs (800 °C)	1.30
PVA/PAA: 3% CoONPs (800 °C)	1.44
PVA/PAA: 5% CoONPs (800 °C)	1.81
PVA/PAA: 7% CoONPs (800 °C)	2.32
PVA/PAA: 9% CoONPs (800 °C)	3.14

from (1MHz) to (5MHz) and at temperature (25Cº), using Eq. 4 to know the polarization and electrical conductivity that occurs in the membrane structure. Explain the behavior (σa.c) of pure (PVA/PAA) films supported with cobalt oxide nanoparticles (CoO NPs) in different proportions as a function of frequency in Fig. 4.

The results showed that with increasing frequency values (σa.c) increases, Table 4 shows this increase. Increasing frequency values leads to increased electric polarization in the prepared films, resulting in rapid jumps of charge carriers between adjacent levels.

Also, the results showed that the addition of CoO NPs led to an increase in the σa.c values of all prepared PVA/PAA films at every frequency [30]. As the percentage of nanomaterial increases, the values of (σa.c) increase. Table 4 shows this increase. This increase is due to the addition of CoO NPs, which increases the number of electrons and thus increases the AC electrical conductivity values [31,32].

Thermal Characterizations

Thermal Conductivity (K)

The thermal conductivity (K) of polymers and polymer compounds is important in knowing the physical nature through which it is known how these polymers are suitable for the required thermal applications. K coefficients of the prepared samples were calculated using Lee's Disk Method, which is used to calculate the k coefficient of insulating materials using the relationship (5), [33].

The (K) coefficients of the prepared pure polymer films (PVA/PAA) reinforced with different weight percentages (pure, 1, 3, 5, 7 and 9) wt% with cobalt oxide nanoparticles (CoO NPs) fired at a temperature of (800 °C) are shown in Fig. 5.

The results showed that (K) increases with the addition of (CoO NPs) particles to the prepared polymer mixture (PVA/PAA), as it was observed that the (K) coefficient for the pure film (PVA/PAA) is equal to (1.18×10-6 W/m.K), By adding nanoparticles, the (K) coefficient began to increase with the increase in the reinforcement ratio, as shown in Table 5. The reason for the high values of the coefficient (K) is Due to the reinforcement of nanoparticles (CoO) to the polymeric material. Increase in thermal conductivity coefficient due to polymer filling with nanoparticles, These molecules therefore fill the structural voids formed inside the polymer during the molding process [34,35].

CONCLUSION

The chemical precipitation method has proven effective in synthesizing cobalt oxide nanoparticles, which are used at different weight ratios to reinforce the PVA/PAA polymer blend. The PVA/PAA: CoO NPs nanocomposite was manufactured using the solution casting method. X-ray diffraction data indicated the effect of calcination temperatures, accompanied by an improvement in crystallinity and crystallite sizeElectrical tests showed that ε′ and tanδ increased with the addition of doping ratios, while both decreased with increasing frequency. The (σA.C.) also increased with increasing doping percentages and frequency. Thermal tests also showed an increase in thermal conductivity (K) with increasing doping percentages.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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