

Synthesis, Thermal Stability, and Electrochemical Behavior of Polypyrrole/Ag₂O Nanocomposites in Chloride Media

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Abstract

Original Research Article

In this study, polypyrrole (PPy) nanocomposites modified with silver(I) oxide (Ag₂O) nanoparticles were successfully synthesized and characterized to evaluate their electrochemical stability, thermal stability, and structural properties. The UV-Vis spectra confirmed the incorporation of Ag₂O nanoparticles into the PPy matrix, evidenced by a shift in the polaron band and the appearance of an absorption shoulder around 600 nm. FTIR analysis verified the integrity of the PPy backbone and confirmed the presence of Ag - O vibrations in the fingerprint region. SEM and TEM images demonstrated the dispersion of Ag₂O nanoparticles with crystalline size 75 to 87 nm within the polymer matrix. Thermogravimetric analysis (TGA) revealed that while the incorporation of Ag₂O slightly lowered the onset temperature of thermal decomposition due to a catalytic effect, it significantly increased the final residual mass at high temperatures due to the formation of metallic silver and carbonaceous char. Furthermore, electrochemical open circuit potential (OCP) measurements in a 0.05 M NaCl medium indicated that the nanocomposite exhibits enhanced electrochemical stability, as evidenced by its significantly lower potential drift rate ($\nabla E/\nabla t$) at equilibrium compared to PPy. These findings suggest that PPy/Ag₂O nanocomposites hold significant potential for applications requiring electrochemically stable coatings, though further kinetic electrochemical studies (as EIS, Tafel) are recommended for comprehensive corrosion rate quantification and protection efficiency assessment.

Key words: Electrochemical stability, Poly Pyrrole, Silver (I) Oxide Nanoparticles, Nanocomposites, Thermal stability.

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1. INTRODUCTION

Conductive polymers, especially polypyrrole (PPy), have received significant attention due to their unique electrical properties and potential applications in various fields, including sensors, actuators, and protective coating materials [1-5]. The incorporation of nanoparticles into conductive polymers has been shown to significantly enhance their properties. Previous studies have indicated that nanoparticles can improve the electrical conductivity and thermal stability of polymer nanocomposites [6,7]. Additionally, silver oxide exhibits antimicrobial properties, which could be beneficial for biomedical applications [8]. The current research aims to explore the structural, thermal, and electrochemical properties of polypyrrole modified with Ag₂O nanoparticles. By fabricating these nanocomposites through a controlled chemical process, we aim to achieve a uniform dispersion of Ag₂O within the PPy matrix. We hypothesize that the addition of Ag₂O will alter the thermal decomposition pathway and influence the

electrochemical stability of the coating in chloride containing environments. Characterization techniques such as FTIR, SEM, and TGA will be used to evaluate the structural integrity and thermal stability of the resulting materials. Also, we hypothesize that the addition of silver(I) oxide (Ag₂O) will not only improve the thermal properties but also enhance the electrochemical stability of PPy, making it a suitable candidate for protective coatings in various industrial applications. The results of this study will contribute to understanding how modifying conductive polymers with metal oxides can improve electrochemical stability and enhance performance in practical applications [9,10].

2. MATERIALS AND METHODS

The following materials were used in this study: Pyrrole monomer (99%), Silver nitrate (AgNO₃) (99.8%, Sigma-Aldrich), Sodium hydroxide (NaOH) (99%, Merck), Ferric chloride (FeCl₃) (98%, Sigma-Aldrich), HPC (Hydroxypropyl Cellulose). Deionized water and

ethanol were used for washing and preparing the solutions.

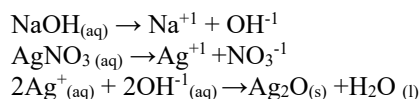
2.1. Synthesis of poly Pyrrole

A 25 mL aliquot of deionized water was placed in a round-bottomed flask, followed by the addition of 8.5 mL of pyrrole monomer. The mixture was homogenized via magnetic stirring for 10 minutes. A 50 mL aqueous solution of 2.5 M ferric chloride (FeCl₃), acting as an oxidant, was introduced dropwise under continuous stirring. Immediate formation of black precipitates signaled the onset of poly pyrrole polymerization. The reaction proceeded for 24 hours at room temperature to ensure complete monomer conversion. The resultant precipitate was vacuum-filtered and subjected to sequential washing cycles with deionized water and ethanol to eliminate residual monomers and oxidative by-products. The purified polypyrrole was dried in a vacuum oven at 50 °C for 24 hours, and the final product was weighed to determine the reaction yield [9,10].

2.2. Synthesis of Silver(I) Oxide (Ag₂O) Nanoparticles

0.02M sodium hydroxide (NaOH) solution was prepared by dissolving 0,05 g of NaOH granules in 100 mL deionized water. A separate 100 mL of 0.01 M AgNO₃ was prepared using deionized water, then NaOH solution was added dropwise to the AgNO₃ solution with continuous stirring. The addition rate was carefully controlled to ensure uniform nanoparticle formation. The

color change to brownish black indicated the successful formation of silver(I) oxide nanoparticles. The mixture was continuously stirred for 2 hours to ensure complete precipitation and stabilize. The collected nanoparticles were washed several times with deionized water and ethanol to remove any impurities or unreacted chemicals [11]. Finally, the purified nanoparticles were dried using a vacuum oven at 50 °C for 24 h.



2.3 Preparation of Poly pyrrole/Silver Oxide Nanocomposites

The poly pyrrole/silver (I) oxide (PPy/Ag₂O) nanocomposite was synthesized via *in-situ* oxidative polymerization. First, 5.4 g of Iron (III) chloride (FeCl₃) in 100 mL of deionized water under continuous magnetic stirring to form a homogeneous solution. Subsequently, 0.4 g of hydroxypropyl cellulose (HPC), acting as a stabilizer, and 0.2 g of silver (I) oxide (Ag₂ONPs) nanoparticles were dispersed into the solution, followed by 30 minutes of vigorous stirring to ensure uniform mixing. Freshly distilled pyrrole monomer (1 mL) was then injected into the reaction mixture, initiating oxidative polymerization at room temperature for 5 hours. The resulting dark precipitate was isolated via vacuum filtration, washed sequentially with deionized water and ethanol to remove impurities, and dried at 60°C for 24 hours [5,12]. A schematic representation of the synthesis process is provided in Figure 1.

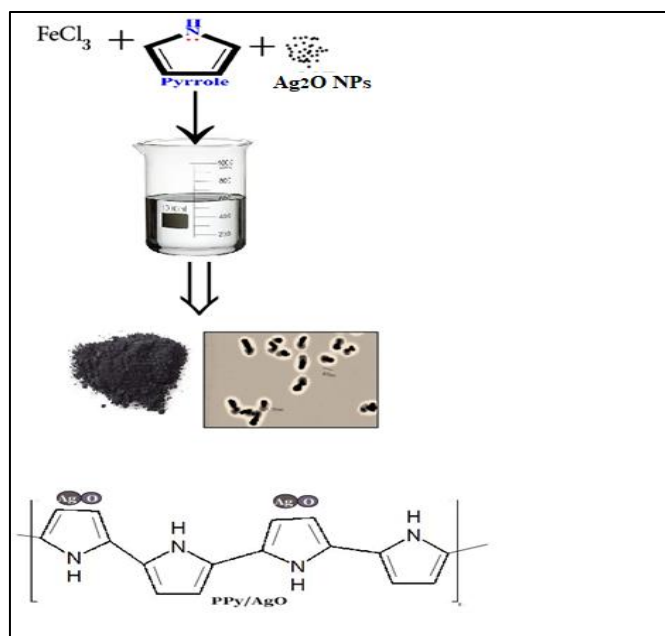


Figure 1: Synthesis of PPy/Ag₂O by oxidative method

3. RESULTS AND DISCUSSION

3.1. UV-Vis Spectroscopy

Figure 2 Shows the absorption spectra of poly pyrrole (PPy) and PPy/Ag₂O nanocomposite. Optical

absorption studies of the poly pyrrole/silver (I) oxide (PPy/Ag₂O) nanocomposite and prepared PPy demonstrated enhanced optical properties due to the addition of silver (I) oxide. The prepared PPy composite

exhibited strong absorption band 420 nm is associated with the $\pi-\pi^*$ transition of the conjugated polymer backbone, weak band at 580 nm this band is attributed to the formation of polaron states, which are generated due to partial oxidation of PPy, and weak absorption at 850 nm, this absorption in the near infrared region

corresponds to bipolaron transitions or free charge carrier absorption, indicating a highly doped conducting state of PPy. The broadness of this band suggests delocalized charge transport along the polymer backbone [3,13].

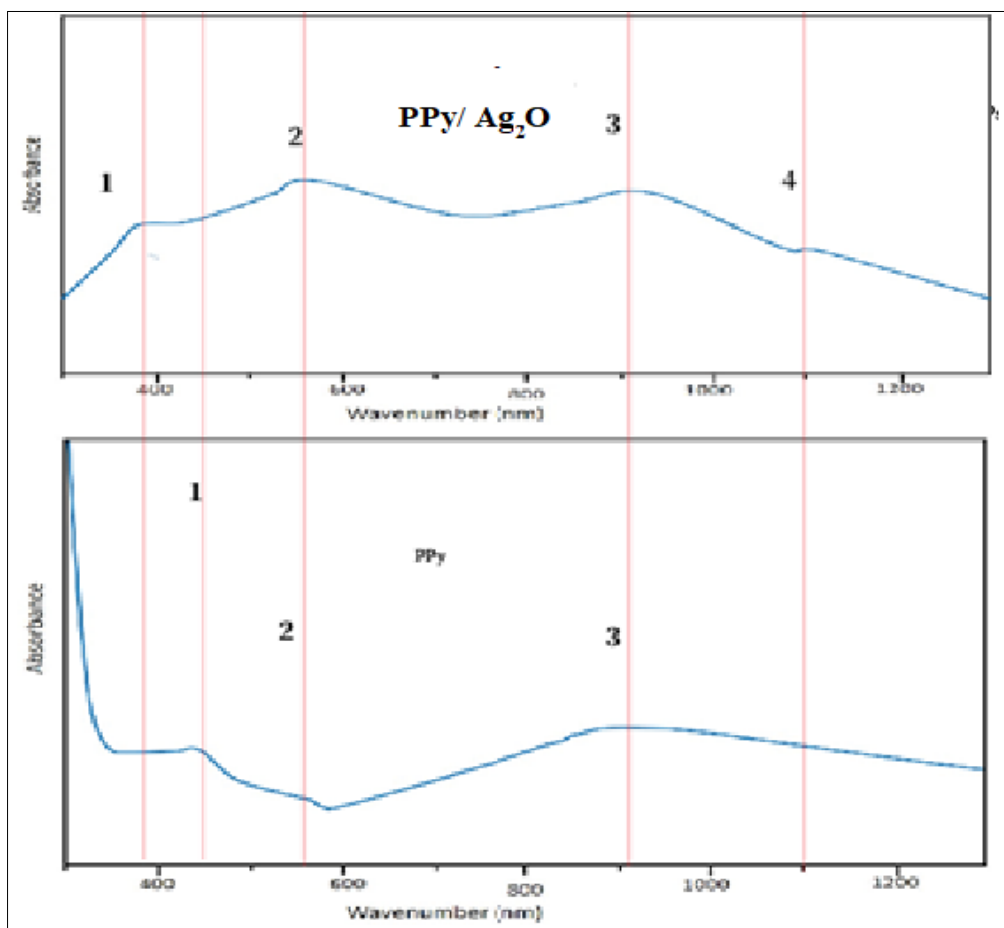


Figure 2: UV-Vis absorption spectra of poly pyrrole (PPy) and PPy/Ag₂O nanocomposite

In contrast, the PPy/Ag₂O nanocomposite exhibited distinct absorption peaks, the moderate intensity peak at 420 nm, similar to PPy, this peak corresponds to the $\pi-\pi^*$ transition of the PPy backbone. However, slight changes in intensity and position suggest interaction between PPy chains and Ag₂O nanoparticles, strong absorption peak at 560 nm this absorption can be attributed to polaron related transitions, but the shift compared to PPy indicates altered electronic structure caused by formation, and significant intensity peak at 910 nm this broad absorption band indicates enhanced charge carrier transitions. The increased intensity suggests improved charge delocalization due to interaction between PPy and Ag₂O, as well as a broad absorption band spanning 1100 nm, this additional absorption band observed only in the nanocomposite is likely due to interfacial charge transfer interactions between PPy and Ag₂O nanoparticles. This new feature confirms successful incorporation of Ag₂O into the polymer matrix [3, 14,15].

3.2. Fourier transform infrared (FTIR) spectroscopy

Figure 3 shows the FTIR spectra of polypyrrole (PPy) and the PPy/silver (1) oxide (PPy/Ag₂O) nanocomposite in the range of 450 to 4000 cm^{-1} . The spectra provide information about the functional groups, bonding characteristics, and interactions between PPy and Ag₂O in the nanocomposite. The FTIR spectrum of PPy shows several characteristic absorption bands corresponding to the vibrational modes of the polypyrrole backbone. The broad peak at 3300 cm^{-1} corresponds to N-H stretching vibrations. The peaks at 1550 cm^{-1} correspond to C=C ring stretching vibrations of the conjugated pyrrole ring, indicating the presence of the polymerized PPy backbone, small peak at 1450 cm^{-1} assigned to C-N stretching vibration, characteristic of the pyrrole structure, while two bands at 1300 and 1050 cm^{-1} due to C-N stretching, and C-H out of plane bending confirm the integrity of the polymer structure.

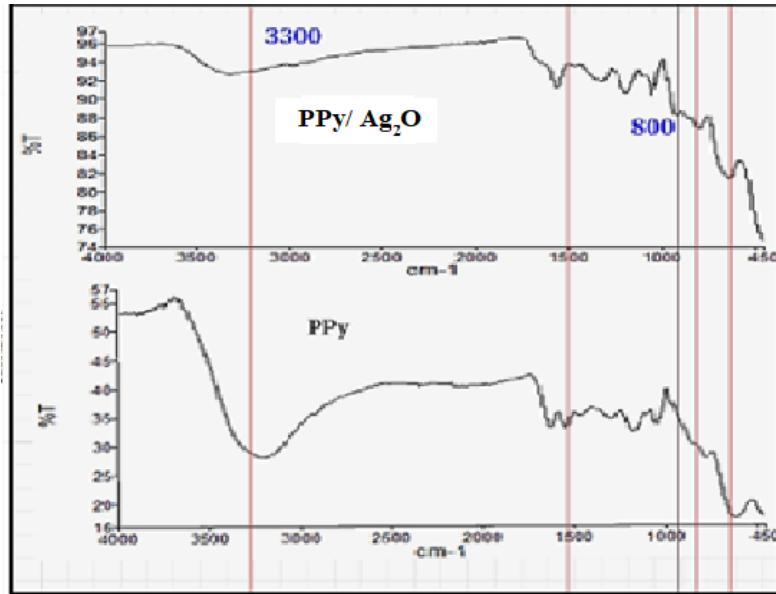


Figure 3: FTIR spectra of poly Pyrrole and poly Pyrrole /Silver (I) oxide nanocomposite

The PPy/Ag₂O composite spectrum displays significant alterations compared to the PPy spectrum, providing evidence for the incorporation of Ag₂O nanoparticles within the PPy matrix and their interaction with the polymer chains. Compared with the polypyrrole (PPy) spectrum, the broad peak corresponding to N–H stretching vibration was slightly shifted to 3400 cm⁻¹, suggesting interaction between PPy chains and Ag₂O nanoparticles, the peak corresponding to C=C ring stretching vibrations also shifted slightly to 1560 cm⁻¹, indicating changes in the electronic environment of the PPy backbone due to the incorporation of Ag₂O.

Additionally, new absorption bands appeared below 700 cm⁻¹, which are attributed to Ag–O stretching vibrations, confirming the presence of silver (I) oxide in the nanocomposite [5,16].

3.3. X-ray diffraction (XRD)

Figure 4 Shows the XRD pattern of Poly pyrrole and the PPy/Ag₂O nanocomposite. The PPy exhibits a broad, low-intensity halo centered in the range of 2θ = 18 to 25 degrees, which this broad peak indicates that the PPy has a predominantly amorphous structure or weakly crystalline structure. The lack of sharp peaks suggests that there is no significant long-range crystalline order.

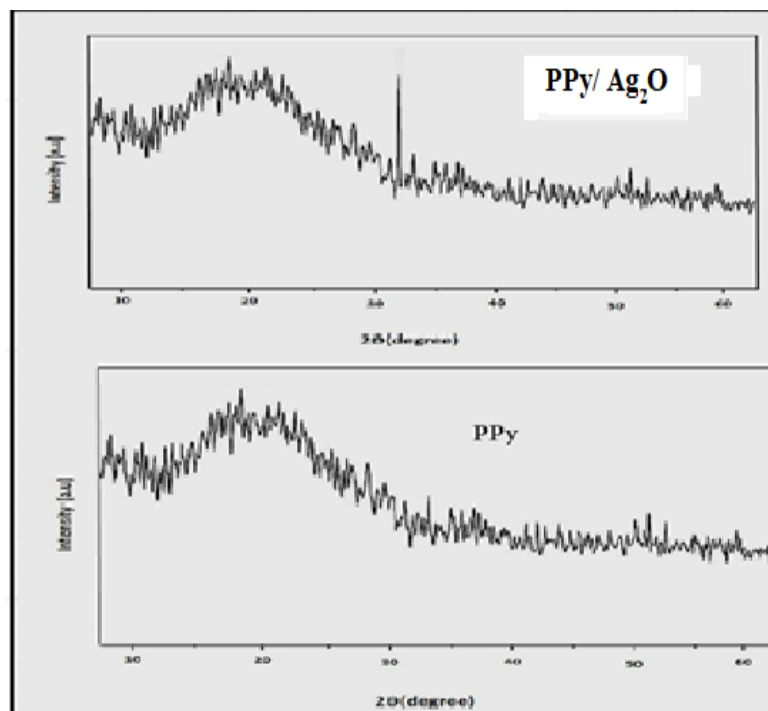


Figure 4: XRD patterns of PPy and PPy/Ag₂O nanocomposite.

The XRD pattern of PPy/Ag₂O nanocomposite exhibits a broad halo similar to that of PPy, indicating the continued amorphous nature of the composite, but it is overlaid with distinct, sharp diffraction peak at 2θ approximately 32.8°. This sharp peak is superimposed on the broad amorphous background and is attributed to the crystalline silver(I) oxide nanoparticles. The position of this peak corresponds to the (111) crystallographic plane of cubic Ag₂O structure, according to the JCPDS (Joint Committee on Powder Diffraction Standards) card no. 41-1104. The XRD pattern does not show characteristic peaks for metallic silver (Ag⁰) at 2θ = 38.1°, 44.3°, 64.4°, and 77.4°, nor does it show peaks for silver (II) oxide (AgO). This confirms that the synthesized nanoparticles are indeed Ag₂O and that no reduction to metallic silver occurred during the polymerization process. These structural characteristics provide the foundation for understanding the enhanced corrosion protection and

modified thermal properties observed in the PPy/Ag₂O nanocomposite, as the crystalline nanoparticles are uniformly dispersed within the conductive polymer matrix, creating a multifunctional material with potential applications in protective coatings, sensors, and catalysis [17-19].

3.4. Scanning electron microscopy (SEM)

Figure 5 Shows the SEM image of Poly pyrrole and the PPy/Ag₂O nanocomposite. The surface morphology of PPy reveals a homogeneous and smooth surface morphology, typical of amorphous polypyrrole films. This uniformity indicates a well polymerized structure with minimal defects. In contrast, the PPy/Ag₂O nanocomposite exhibits a highly heterogeneous, granular surface with increased roughness, confirming the integration of inorganic particles into the polymer matrix.

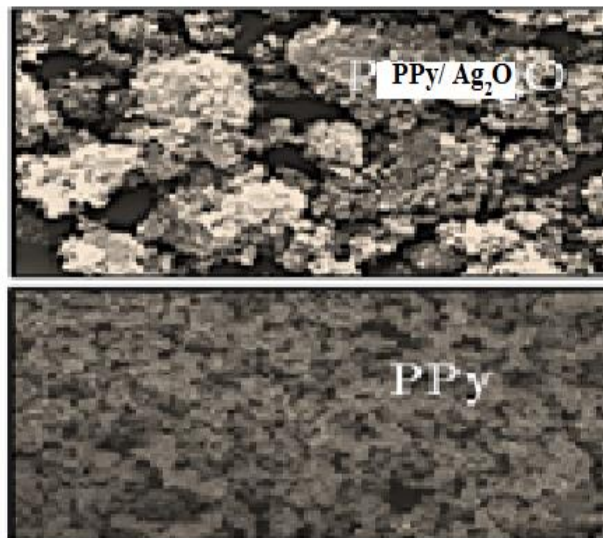


Figure 5: SEM images of PPy and PPy/Ag₂O nanocomposites.

3.5 Transmission electron microscopy (TEM)

Figure 6 Shows the TEM image of Poly pyrrole and the PPy/Ag₂O nanocomposite. The TEM image of PPy shows a dense amorphous structure without

distinctive features, which is consistent with the amorphous nature of polypyrrole.

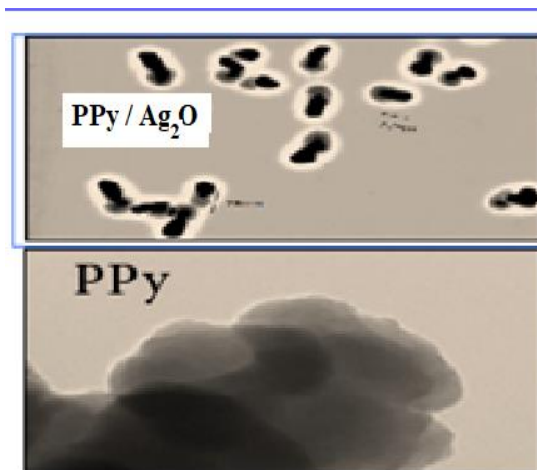


Figure 6: TEM images of PPy and PPy/Ag₂O nanocomposites.

The TEM micrograph of the PPy/Ag₂O nanocomposite exhibits a distinctly different morphology, providing direct visual evidence of the successful incorporation of silver (I) oxide nanoparticles. The image shows highly electron dense (dark) regions dispersed within a lighter, hazy background matrix. The intense darkness of these features arises from the high atomic number of silver ($Z = 47$), which scatters electrons much more strongly than the surrounding

organic polymer chains. These dark, irregular, and somewhat elongated clusters correspond to the Ag₂O nanoparticles embedded within the PPy matrix [5, 20].

3.6 Thermogravimetric Analysis (TGA)

Figure 7 Shows the thermal degradation profiles of PPy and PPy/Ag₂O nanocomposite over a temperature range of 0 °C to 400 °C.

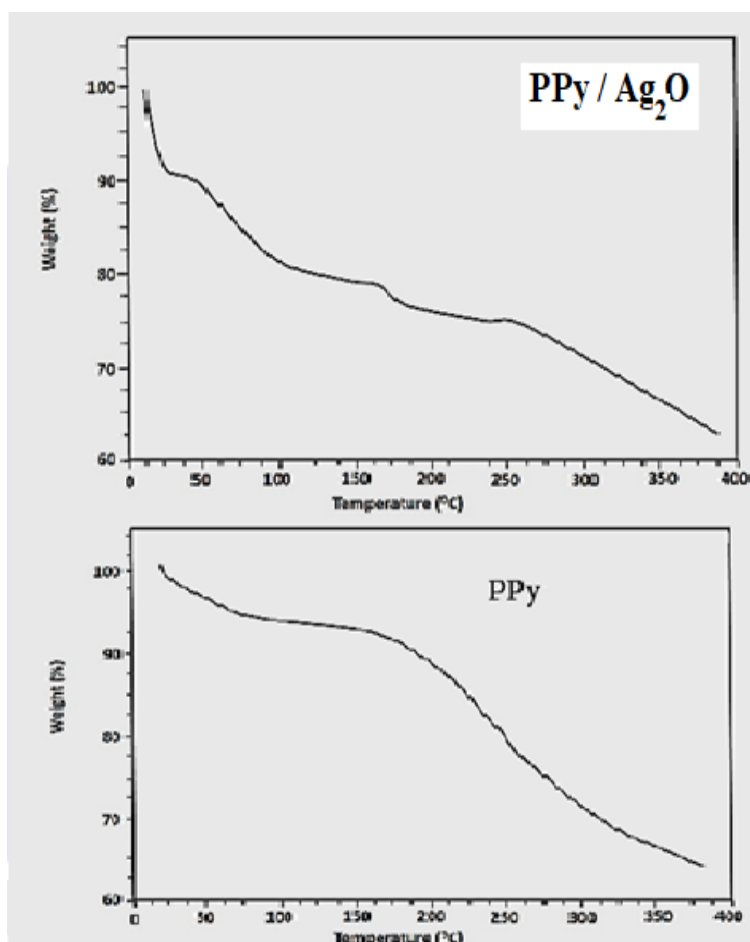


Figure 7: TGA curves of PPy and PPy/Ag₂O nanocomposites

The TGA curve for PPy exhibits a relatively smooth, continuous degradation profile. An initial, minor weight loss of approximately 5% is observed up to 100 °C, which is attributed to the evaporation of adsorbed moisture and residual solvent trapped within the polymer matrix. Following this, the polymer backbone remains relatively stable up to 200 °C. Beyond 200 °C a gradual and steady weight loss occurs corresponding to the removal of anion of the polymer chain (loss of chloride counter ions) and the subsequent thermal degradation of the polypyrrole backbone itself. At 400 °C, the residual weight is approximately 65%, representing the carbonaceous char formed from the incomplete combustion of the polymer. In contrast, the TGA curve for the PPy/Ag₂O nanocomposite reveals a more complex degradation pathway with distinct features not present in the pure polymer, The nanocomposite shows a

more pronounced initial weight loss between 0°C and 50°C compared to PPy. This indicates that the combination of hydrophilic Ag₂O nanoparticles and the porous structure of the nanocomposite (stabilized by HPC) lead to higher absorption of atmospheric moisture or trapped solvent during the synthesis process. A noticeable inflection in the weight loss curve can be seen at around 180°C. This feature is absent in the PPy curve and is likely attributable to two simultaneous phenomena, Ag₂O decomposition is thermally unstable and decomposes into metallic silver and oxygen gas at elevated temperatures. While bulk Ag₂O decomposes near 300 °C, nanoparticles often exhibit lower decomposition temperatures due to high surface energy and reduced thermal stability. The release of oxygen gas contributes to the observed weight loss step. Also, the hydroxypropyl cellulose (HPC) used as a stabilizer

during synthesis typically begins to degrade in the 200 to 300 °C range. The presence of this organic additive may contribute to the early weight loss observed in this region. At 400 °C, the residual weight of the PPy/Ag₂O nanocomposite (63%) is very similar to that of PPy (65%). This indicates that while the pathway of decomposition is altered by the presence of the inorganic nanoparticles (specifically the loss of oxygen from Ag₂O), the final char yield at this temperature is not significantly enhanced [21-23].

3.7 Electrochemical Measurements

Figure 8 Shows that the electrochemical analysis using open-circuit potential (OCP) measurements highlights significant differences in the electrochemical stability between poly pyrrole (PPy) and the PPy/Ag₂O composite in a 0.05 M NaCl medium over a period of 100 minutes, and the corresponding rate analysis graphs, with detailed numerical data summarized in Table 1.

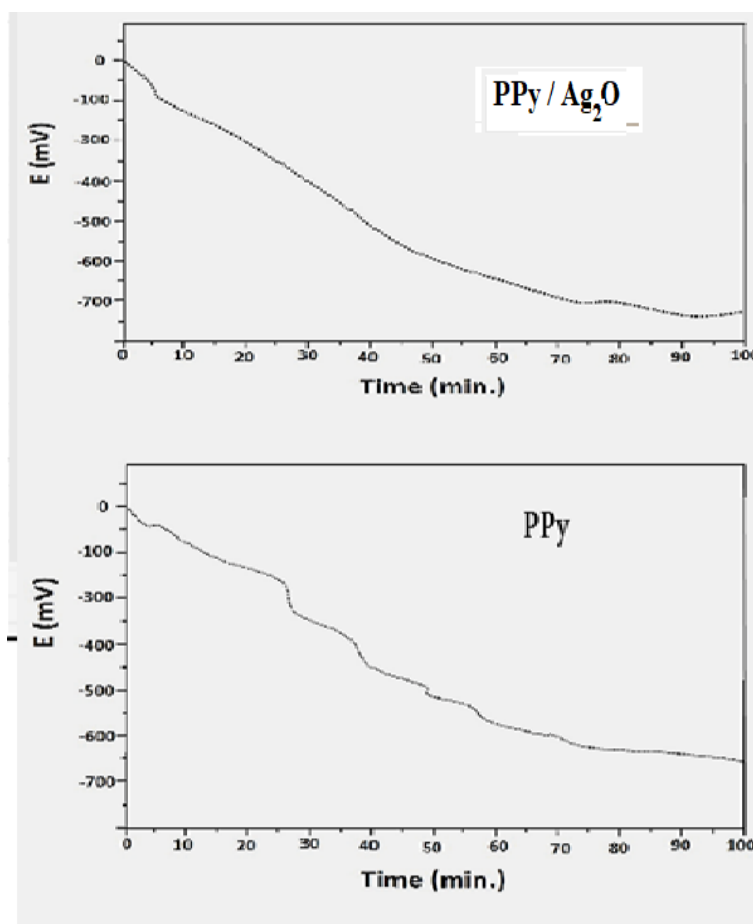


Figure 8: Open-circuit potential (OCP) versus time curves for PPy and PPy/Ag₂O nanocomposites in 0.05 mol/L NaCl solution

The PPy coating exhibited a rapid initial potential drop, reaching -310 mV within the first 30 minutes. The rate of change ($\Delta E/\Delta t$) was highly variable, peaking at -16 mV/min at 30 minutes (Table 1). This rapid initial fluctuation suggests that the PPy film is relatively porous, allowing the electrolyte (NaCl) to penetrate quickly and interact with the polymer backbone, resulting in less stable electrochemical behavior [9,10]. By the end of the experiment after 100 min, the potential stabilized at -650 mV, with a residual drift rate of -1 mV/min. While the potential stabilized, the relatively higher drift rate indicates that the interface was still undergoing slow changes, likely due to continued ion diffusion or polymer degradation, suggesting lower electrochemical stability. In contrast,

the PPy/Ag₂O nanocomposite exhibited a very rapid initial drop, reaching -170 mV within just 10 minutes, with a maximum rate of change of -20 mV/min. This initial rapid response is likely due to the high surface area and electrochemical activity of the Ag₂O nanoparticles, which may undergo rapid surface redox reactions or reduction upon contact with the electrolyte, followed by the establishment of a more stable electrochemical interface. After the initial rapid drop, the potential of the nanocomposite decreased much more gradually and smoothly than the PPy. By 100 minutes, the potential reached -725 mV, but crucially, the rate of change had dropped to a negligible -0.5 mV/min (Table 1), indicating superior electrochemical stability [3, 5].

The most compelling evidence for the enhanced electrochemical stability of the nanocomposite is the stabilization rate. The PPy/Ag₂O composite achieved a drift rate of 0.5 mV/min at the end of the test, which is half that of the PPy (-1 mV/min). A lower drift rate $\Delta E/\Delta t$ about 0 indicates that the coating has reached a steady state where the flux of ions through the coating is minimized, suggesting improved barrier properties. This suggests that the Ag₂O nanoparticles effectively fill the micropores of the PPy matrix, creating a tortuous path that hinders the diffusion of aggressive chloride ions (Cl⁻) and water molecules to the metal interface. This barrier effect leads to a more stable electrochemical interface, as evidenced by the lower potential drift rate [10]. Also, it is important to note that the nanocomposite stabilized at a more negative potential (-725 mV) compared to PPy (-650 mV). In traditional corrosion science, a more

negative (active) potential often indicates a higher thermodynamic tendency for corrosion. However, for conductive polymer nanocomposites, this negative shift can also be attributed to the specific redox activity of the silver species (e.g., reduction of Ag⁺ to Ag) or the establishment of a stable electrochemical interface. The key observation is the enhanced stability of the potential over time, as indicated by the lower drift rate. The key indicator of electrochemical stability here is not the absolute value of the potential, but the stability of the potential over time. The fact that the nanocomposite potential stabilizes so effectively (low $\Delta E/\Delta t$) suggests it forms a robust, durable barrier that resists further degradation better than the pure polymer, indicating potential suitability for protective coating applications.

Table 1: Electrochemical Potential (E) and Rate of Change Over Time of PPy and PPy/Ag₂O nanocomposites in 0.05 M NaCl solution

Poly Pyrrole			
t (min)	E(Mv)	$\Delta E(Mv)$	$\Delta(mv/min)$
0	-20	-	-
10	-90	-70	-7
20	-150	-60	-6
30	-310	-160	-16
40	-420	-110	-11
50	-480	-60	-6
60	-550	-70	-7
70	-600	-50	-5
80	-630	-30	-3
90	-640	-10	-1
100	-650	-10	-1
Poly Pyrrole/Ag ₂ O			
t (min)	E(Mv)	$\Delta E(Mv)$	$\Delta(mv/min)$
0	0	-	-
5	-70	-70	-14
10	-170	-100	-20
20	-290	-120	-12
30	-400	-110	-11
40	-500	-100	-10
50	-590	-90	-9
60	-650	-60	-6
70	-690	-40	-4
80	-710	-20	-2
90	-720	-10	-1
100	-725	-5	-0.5

4. CONCLUSION

In this study, polypyrrole (PPy) nanocomposites modified with silver(I) oxide (Ag₂O) nanoparticles were successfully synthesized via an *in-situ* chemical oxidative polymerization method. Comprehensive structural and morphological characterizations confirmed the uniform integration of crystalline Ag₂O nanoparticles (75 to 87 nm) into the amorphous PPy matrix. UV-Vis and FTIR spectroscopy revealed significant interfacial interactions between the polymer backbone and the nanoparticles, evidenced by shifts in

the polaron band and the emergence of Ag–O vibrational modes. Furthermore, XRD analysis verified the phase purity of the cubic Ag₂O structure without any unintended reduction to metallic silver during polymerization. Thermogravimetric analysis (TGA) demonstrated that the incorporation of Ag₂O nanoparticles distinctly altered the thermal degradation pathway of the polymer. While the onset of thermal decomposition was slightly lowered due to the catalytic effect of the nanoparticles and the thermal breakdown of the Ag₂O/HPC components around 180 °C, the

nanocomposite maintained a high residual mass of approximately 63% at 400 °C. This indicates robust high-temperature char and metallic residue retention, ensuring structural integrity under moderate thermal stress. Most importantly, electrochemical evaluations in a 0.05 M NaCl medium revealed that the PPy/Ag₂O nanocomposite exhibits superior electrochemical stability compared to PPy, as evidenced by a significantly lower potential drift rate at equilibrium. Although both coatings exhibited a shift towards more negative potentials upon exposure to the electrolyte, the nanocomposite achieved a significantly lower potential drift rate ($\Delta E/\Delta t = -0.5$ mV/min) at equilibrium compared to PPy (-1 mV/min). This enhanced electrochemical stability is attributed to the Ag₂O nanoparticles effectively filling the micropores of the PPy matrix, thereby creating a tortuous diffusion path that restricts the penetration of chloride ions and water molecules to the underlying interface, resulting in a more stable electrochemical interface [5].

Overall, the PPy/Ag₂O nanocomposite presents a highly promising, multifunctional material for applications requiring electrochemically stable coatings with potential protective properties. While the OCP results indicate improved electrochemical stability, future studies will focus on comprehensive kinetic evaluations, including potentiodynamic polarization (Tafel plots) and electrochemical impedance spectroscopy (EIS), to fully quantify the corrosion protection efficiency and elucidate the exact electrochemical mechanisms at the coating-metal interface.

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