



STRUCTURAL AND MORPHOLOGY PROPERTIES OF POLYPYRROLE / TiO₂ - CuO NANOCOMPOSITE FOR SUPERCAPACITOR APPLICATIONS

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ABSTRACT

Conductive polymers have recently attracted considerable attention for supercapacitor applications. Among these polymers, polypyrrole is the most commonly used due to its unique electrical conductivity, photovoltaic properties, redox properties and excellent environmental stability. Nanocomposites of polypyrrole (ppy) and titanium dioxide (TiO₂) doped with copper oxide (CuO) were prepared, with titanium dioxide/copper oxide added at concentrations of 10 per cent, 30 per cent and 50 per cent using a simple and inexpensive chemical oxidation polymerisation process. The structural and physical properties of these nanocomposites were characterised and evaluated using Fourier-transform infrared spectroscopy (FTIR), ultraviolet-visible spectroscopy (UV-Vis), field-emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and X-ray energy dispersive spectroscopy (EDS). Cyclic voltammetry (CV) was also employed. The FTIR spectrum confirmed the formation of a polymeric nanocomposite, as the characteristic bands of polypyrrole shifted to a region with a lower wavenumber. The UV-Vis spectrum revealed that all absorption peaks of the composite had shifted to a higher wavelength compared to polypyrrole. This shift in the absorption peaks is attributed to the strong interaction between the nanoparticles and the polymer chain. Scanning electron microscope (SEM) images showed that the nanoparticles were spherical in shape and uniformly distributed within the polypyrrole. X-ray diffraction results revealed that the crystallinity of the nanocomposite increases with increasing nanoparticle concentration. Furthermore, cyclic voltammetry (CV) data demonstrated the outstanding performance of the hybrid supercapacitor based on these composites, with high capacitance, good energy density and excellent power density.

Keywords: Polypyrrole (PPy); Transition Metal Oxides; XRD / FESEM; Cyclic Voltammetry (CV):Chemical Oxidative Polymerization; Supercapacitor.

1 INTRODUCTION

Super-capacitors or electrochemical capacitors have been developed due to their ability to store high amounts of energy using very fast and reversible reactions of oxidation and reduction or changes in the state of the surface or sub-surface layer of electrodes, and they have been modified for use in different portable electronic appliances [3-1]. Some characteristics that are possessed by electrochemical capacitors include rapid charge and discharge cycles, safety limits of operation, stability, and high energy density [4, 5]. Benefits of the solid-state supercapacitors include safety, low weight, and great flexibility as opposed to other types of capacitors that use liquids. Supercapacitors have been able to establish themselves among many other electrochemical energy storage systems due to their high energy density that allows them to store a lot of energy, as well as the fact that they can be cycled many times without losing their charge capacity (between 100 and 1,000 charge/discharge cycles), and practically infinite charge/discharge rate [9].

Polypyrrole (PPy) is among the most prominent conductive polymers in terms of wide-scale application; due to its several unique properties when compared to other conductive polymers, polypyrrole (PPy) is regarded as a superior candidate for the fabrication of supercapacitors. Some of the significant properties of polypyrrole include mechanical strength, specific capacitance, high electrical conductivity and biocompatibility [12-14]. Nevertheless, there are some limitations associated with the use of conductive polymers, including volumetric contraction upon discharge and poor cyclic stability; thus, the combination of polypyrrole with inorganic transition metal oxides is used to form nanocomposites [15, 16]. Due to rapid redox kinetics and good capacitance properties, transition metal oxides have been commonly used in order to enhance the cyclic stability of polypyrrole [17, 18].

2 MATERIALS AND METHODS

2.1 Materials

Table 1: Substances and Compounds Used

Sample	Ratio	mass
$TiO_2 - CuO$	50%	1.157 g
PPy / $TiO_2 - CuO$	30%	1.18 g
PPy	-	0.5 g

2.2 Methods

The first solution was prepared as follows: a 250 ml beaker was filled with 50 ml of 1 molar acid; the required amount of pyrrole, approximately 0.9313 g, was then added to the acid whilst stirring for at least one hour; the additive, the $TiCO_2 - CuO$ was added in the required proportion, approximately 30 per cent, equivalent to 0.186 g, and the mixture was stirred for a further 20 minutes.

The second solution (oxidising agent) is prepared as follows: another beaker is taken and ferric chloride ($FeCl_3$) is added to it in sufficient concentration; it is then dissolved in 46 ml of distilled water and 4 ml of acid, after which the solution is stirred for 20 minutes

Mixing and filtration: The first solution is added to the second solution dropwise and gradually, as usual, and the mixture is left for several hours (or left until the following day for the reaction to complete). Finally, the precipitate is filtered and washed thoroughly with distilled water.

3 RESULT AND DISCUSSION

3.1 XRD Analysis

3.1.1 Pure polypyrrole (PPy) sample

Figure (1) shows an X-ray diffraction pattern attributable to the amorphous nature of the pure polypyrrole chain; this broad peak provides information on the interchain distance of the polymer chains resulting from the random repetition of polypyrrole rings during the polymerisation process, aided by the oxidising agent $FaCl_3$. The X-ray diffraction pattern of the samples indicates the coexistence of the tetragonal phase of titanium oxide and the monoclinic phase of copper oxide, which is consistent with GCDs cards Nos. 1272-21 and 0937 -45 respectively. As for the pure polythiophene in the samples examined, a distinct, broad, amorphous peak was observed, centred at an angle of approximately 25 degrees; this is a characteristic of the pi-conjugated polymer of the thiophene series [19].

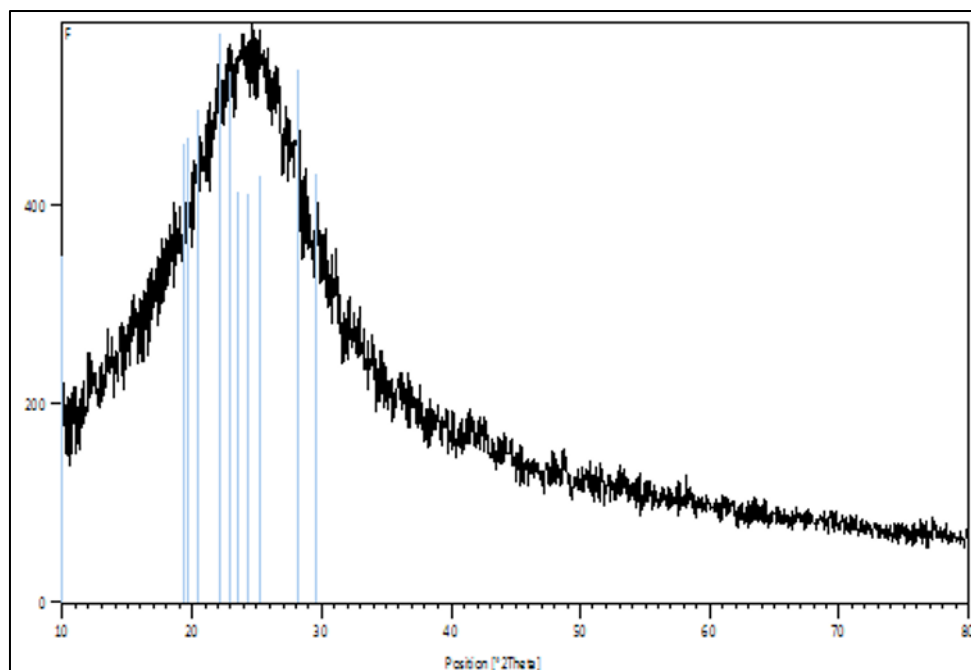


Figure 1: X-ray diffraction analysis of polypyrrole

3.2 Sample of the $\text{TiO}_2\text{-CuO}$ oxide compound

Figure (2) shows an X-ray diffraction pattern exhibiting distinct, sharp and well-defined peaks at various angles, which provides information on the high and distinctive crystallinity of the oxide compound. The first peak exhibits a sharp peak pattern characterised by high intensity at an angle of 25° , which can be attributed to the (101) crystal plane corresponding to the anatase form of titanium dioxide; whilst the other peak appears as a sharp, double peak at two angles, approximately 35.5° and 38.5° ; these angles can be attributed to the (111) and (110) crystal planes associated with the cuprate phase of copper oxide. It can be concluded that the observed patterns confirm the presence of two distinct pure crystalline phases of titanium oxide and copper oxide, one alongside the other, forming impure phases; this indicates that the mixture has stabilised as a binary oxide with a 50 per cent addition, This is consistent with the study [20].

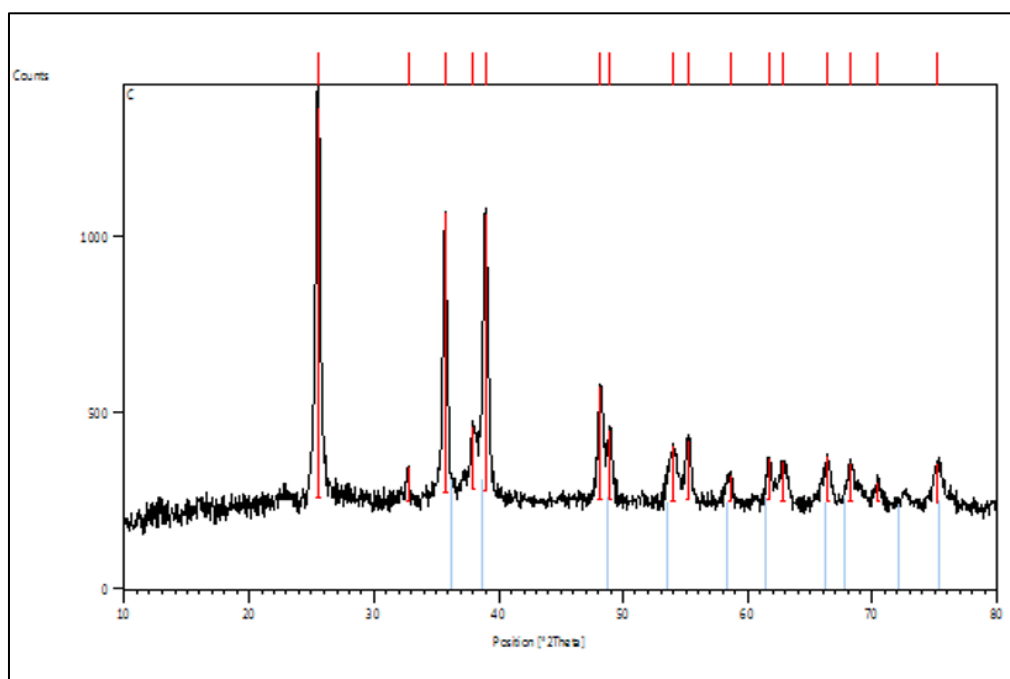


Figure 2: X-ray diffraction analysis of the $\text{TiO}_2\text{-CuO}$ oxide compound

3.2.1 Sample of the PPy/TiO₂-CuO hybrid nanocomposite

Figure (3) shows the X-ray diffraction pattern of the prepared sample; the results of this analysis indicate a distinct superposition of the properties of the amorphous PPy polymer and the crystalline oxides TiO₂ -CuO, with a sharp peak appearing at an angle of approximately 25°, which is attributable to titanium oxide, albeit with reduced intensity. There is also a clear, broad peak for the polymer in the region between 20° and 26°, as well as a reduction in the intensity of the copper oxide peaks. It can be concluded from the analysis that the reduction and alteration in the sharpness of the peaks are due to the coating of the oxide particles with a layer of amorphous pyrrole polymer during the reaction and polymerisation process, which confirms the successful synthesis of a composite material. The final hybrid composite, which was prepared by mixing titanium and copper oxides with polypyrrole, retained the crystalline peaks of the oxides, albeit with indistinct features; this demonstrates the successful formation of the composite without altering the underlying crystalline networks. This corresponds to the source [21]

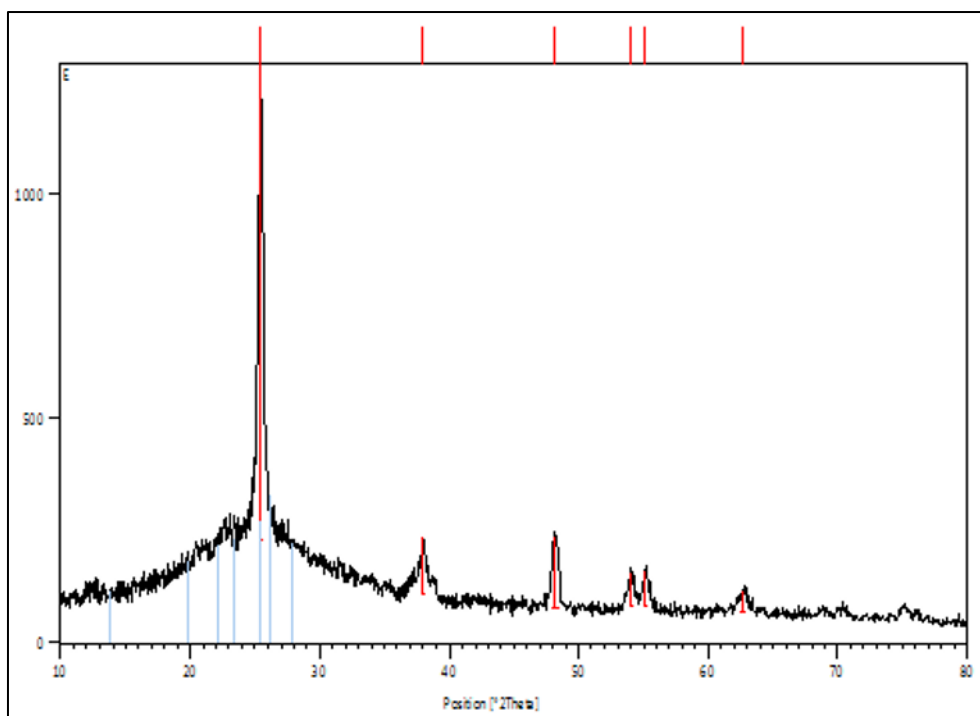


Figure 3: X-ray diffraction analysis of the PPy/TiO₂-CuO hybrid nanocomposite

3.3 SEM Analysis

3.3.1 Pure polypyrrole (PPy) sample

The SEM images shown in Figure (4) reveal agglomerated particles of pure polypyrrole, closer to the nanoscale, ranging in size from 266 –283 nanometres. These are the result of spontaneous oxidative polymerisation, which formed these agglomerates and polymer clusters with uniform symmetry. The polymer is characterised by the presence of interconnected, overlapping spherical agglomerates resembling coral, with no other sharp crystalline forms present. This confirms the purity of the polymer and its semi-crystalline structure .

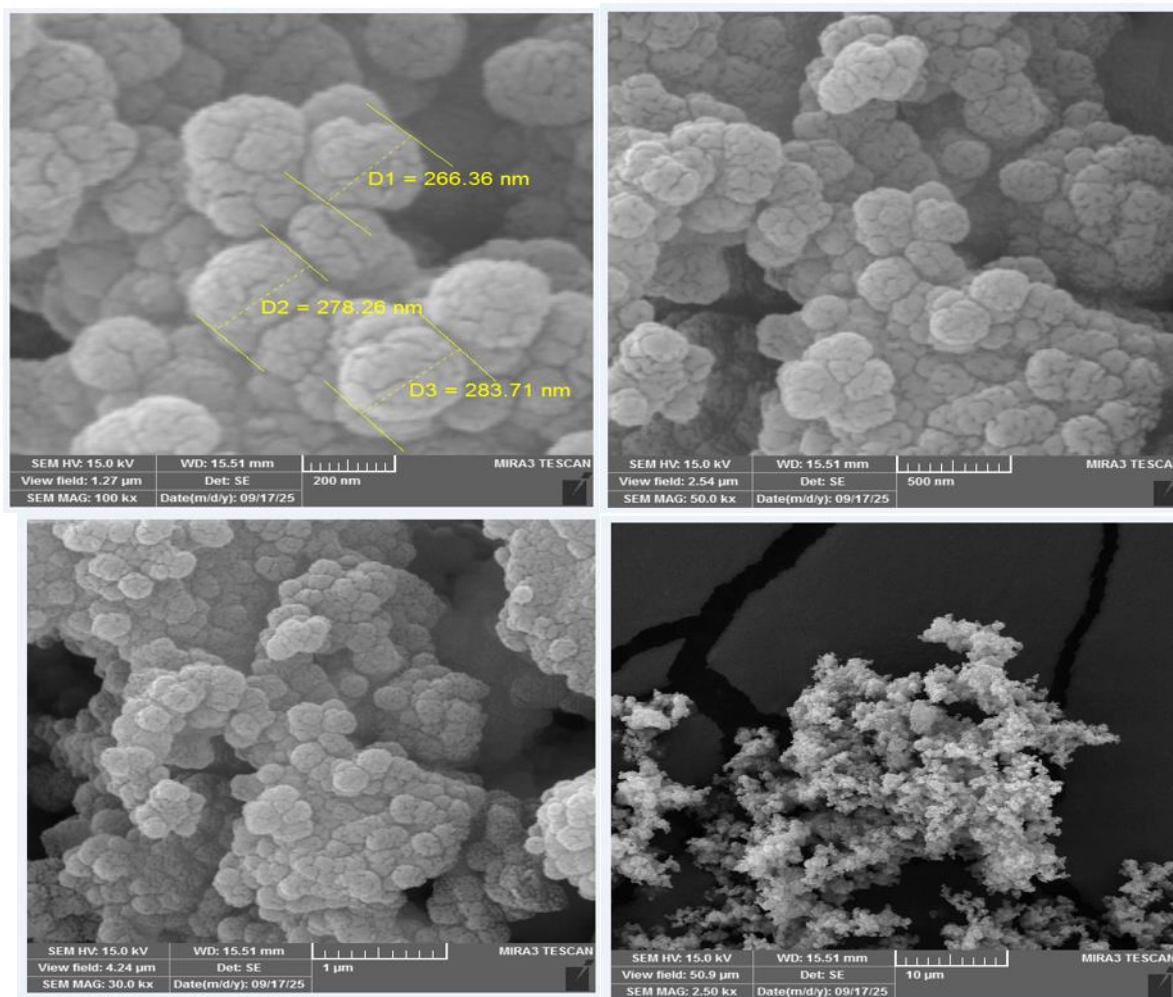


Figure 4: SEM of the Pure polypyrrole (PPy)

Analysis of SEM images revealed a transformation in the granular aggregates into a spherical morphology, which may resemble cauliflower, and a nano-encapsulation structure, as reported in the study [16].

3.4 Sample of the TiO_2 -CuO oxide composite

The scanning electron microscope images in Figure (5) clearly distinguish the material due to the presence of high surface porosity resulting from the distribution of spherical nanoparticles beneath and above the crystalline platelets, with clear agglomeration. It can be concluded from these images that this is a binary nanocomposite comprising spherical titanium oxide particles with nanoscale sizes ranging from 43 –55 nanometres and crystalline copper oxide platelets. This supports the interaction between the oxides and the surface area And this is entirely consistent with the source[19].

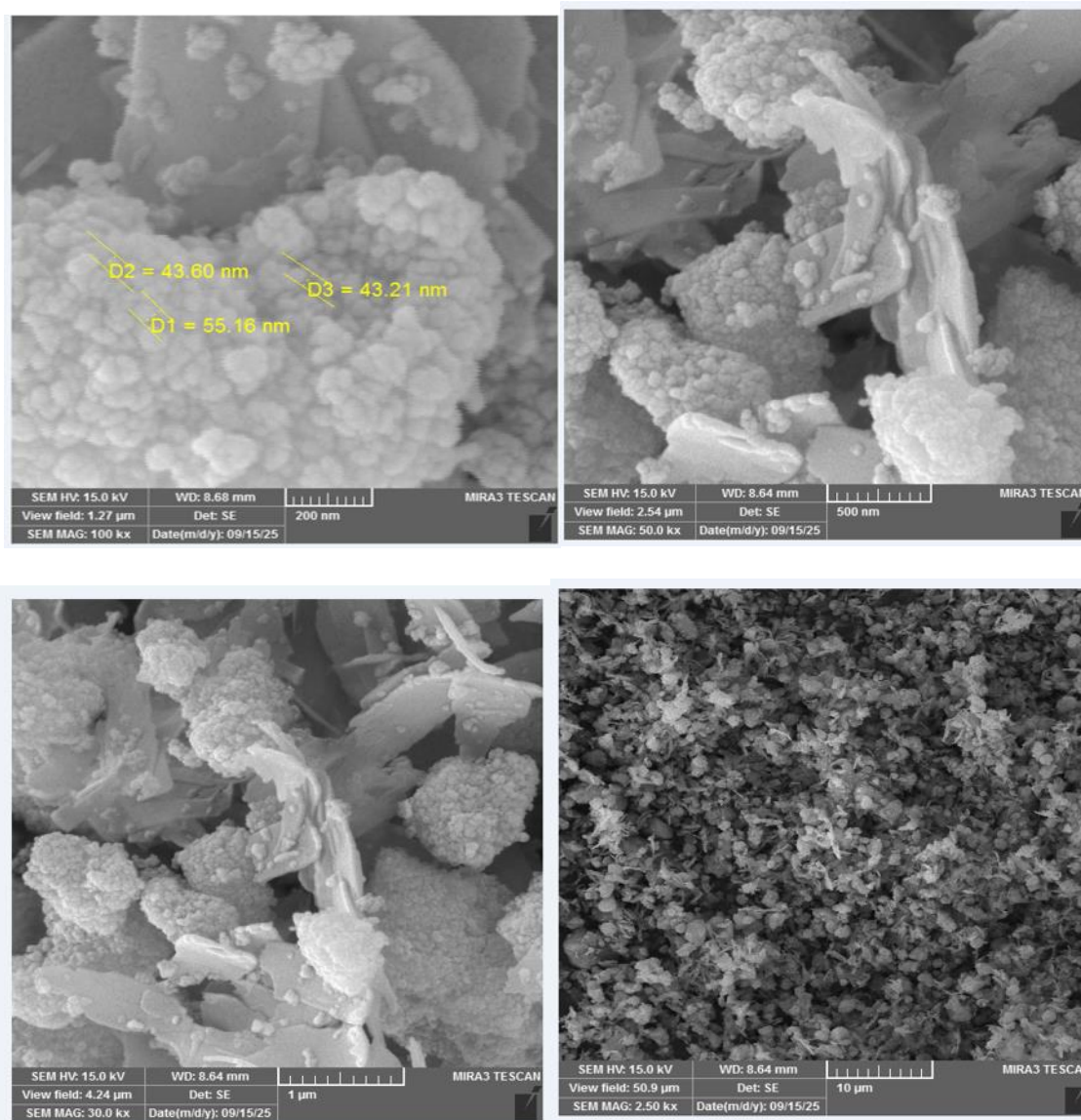


Figure 5: SEM Sample of the TiO_2 -CuO oxide composite

3.4.1 Sample of the PPy/ TiO_2 -CuO hybrid nanocomposite

The scanning electron microscope (SEM) images shown in Figure (6) reveal a nanocomposite with a spherical morphology comprising agglomerates of varying sizes ranging from 156 –328 nanometres. The surface is characterised by a porous, wrinkled texture with a high surface area, where the sheets are hidden and the particles are encapsulated within the polymer matrix. This enhances physical synergy and interparticle contact, demonstrating the successful encapsulation of the oxide nanoparticles within the polyparol matrix And this is entirely consistent with the study[14].

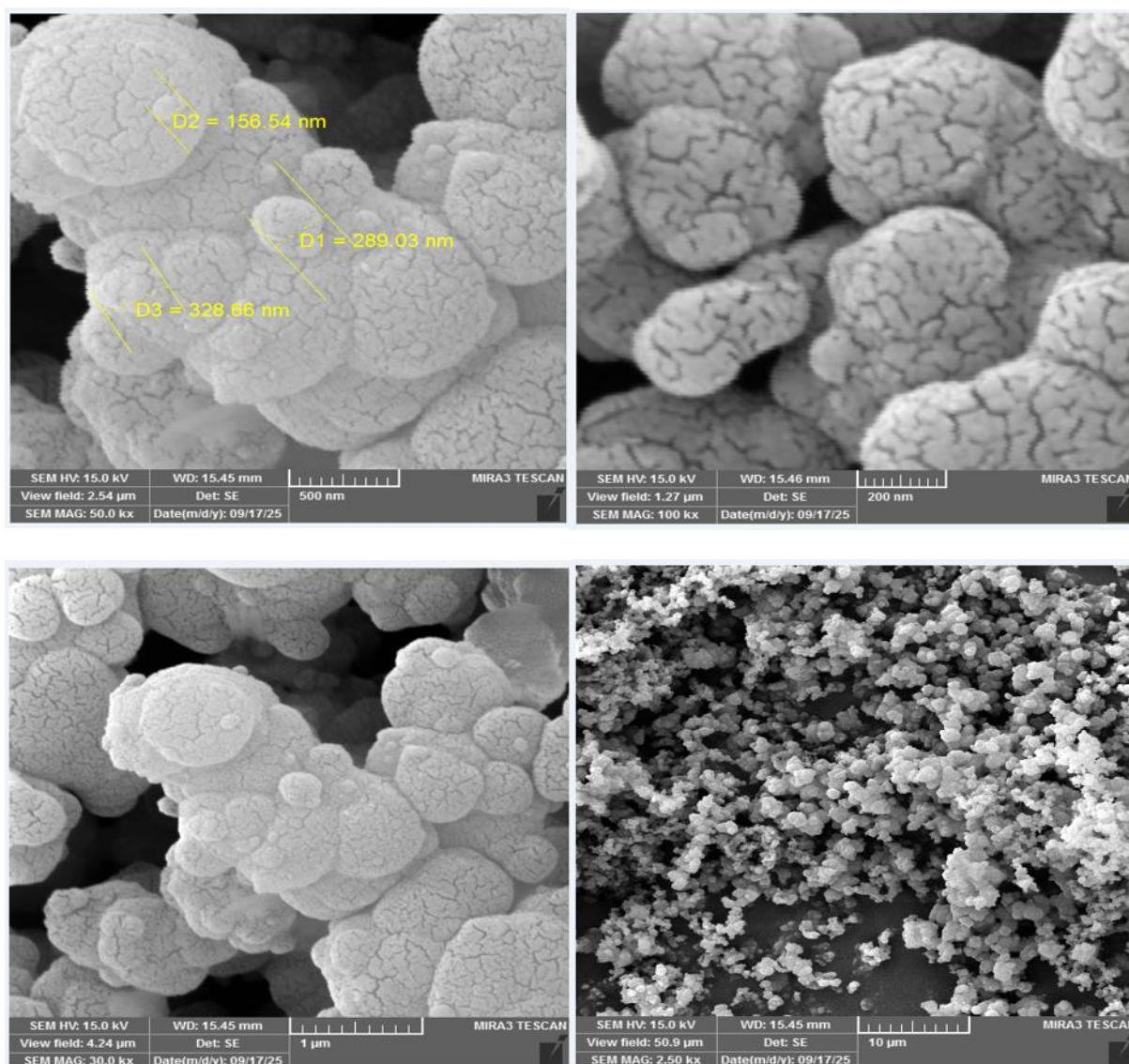


Figure 6: SEM of the PPy/TiO₂-CuO hybrid nanocomposite

3.5 FTIR Analysis

3.5.1 Pure polypyrrole (PPy) sample

Figure (7) shows the infrared spectrum of the sample prepared by polymerisation of pyrrole oxide monomer using FeCl_3 in an acidic medium, where the (N-H) band was recorded at 3302.13cm^{-1} . The carbon-carbon double bond peaks in the pyrrole ring at 1587cm^{-1} and 15233cm^{-1} , as well as the carbon-nitrogen peak at 1338cm^{-1} , can be interpreted as confirming that the polymer has reached a cross-linked and reticulated state resulting from chain oxidation by the oxidising agent during the polymerisation process. These confirmed results in the infrared spectrum, showing the characteristic bonds of titanium dioxide and copper oxide as well as the pyrrole ring vibration in this study, are fully consistent with the source[10].

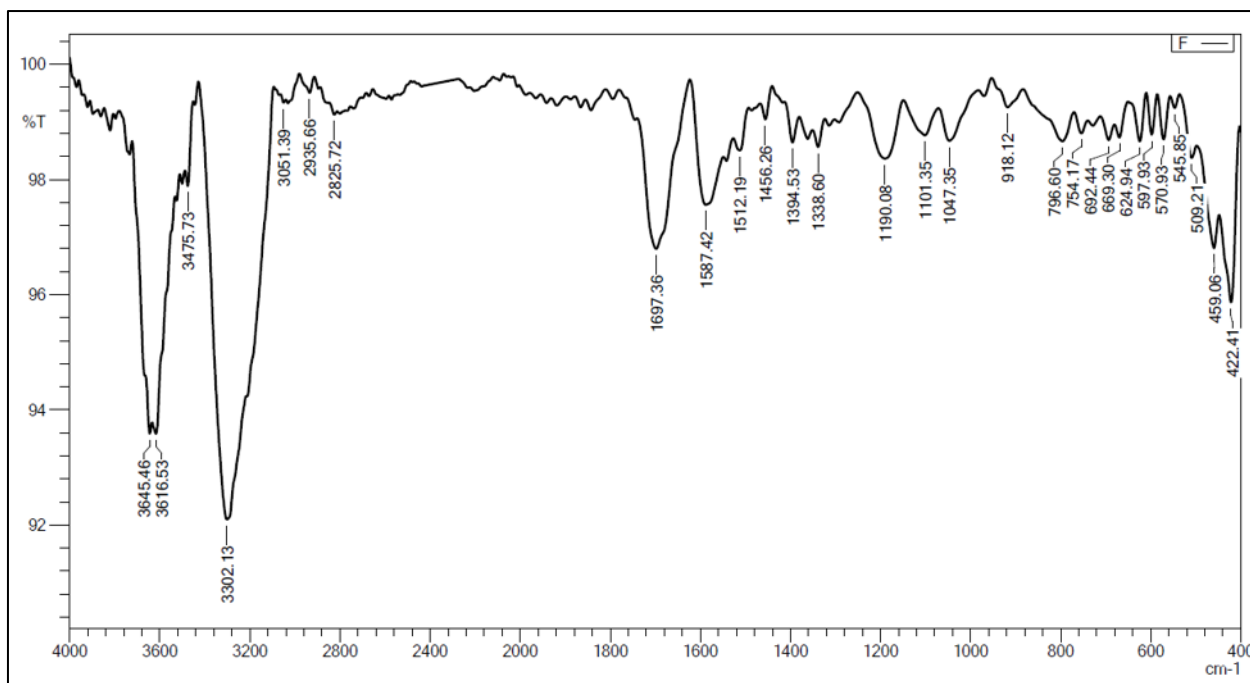


Figure 7: FTIR of Pure polypyrrole (PPy)

3.5.2 Sample of the $\text{TiO}_2\text{-CuO}$ oxide compound

Figure (8) shows the absence of spectra from organic compounds or their influence; the figure highlights the absorption bands of the metal-oxygen bond, which show vibrational bands for the titanium oxide bonds at frequencies of 491 and 459, as well as for the copper oxide bond at frequencies of 426 and 426.67. In addition, the hydroxyl bands of surface-adsorbed water appear at 3304. Ultimately, these highly pure and clean bands confirm the high purity of the prepared mixture and the success of the mixing process without the appearance of any anomalous peaks. Similarly, researchers carried out a study of this kind, and the results were very similar, as they used the same method or one similar to it, which is consistent with the study[13].

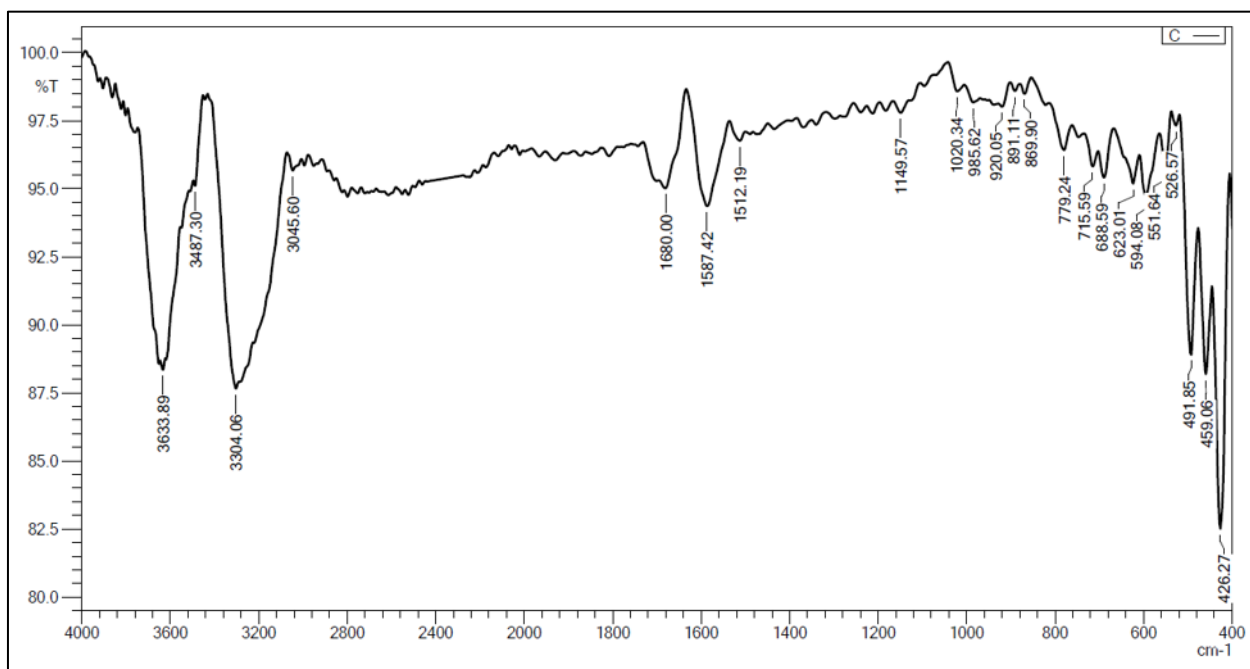


Figure 8: FTIR of the $\text{TiO}_2\text{-CuO}$ oxide compound

3.5.3 Sample of the PPy/TiO₂-CuO hybrid nanocomposite

In Figure (9) the nanocomposite was prepared with the oxides via a polymerisation process to form the hybrid nanocomposite; the figure clearly shows the appearance of polypyrrole peaks as the main band (N-H) at a frequency of 3294, as well as (C=C) peaks at a frequency of 1593, whilst the spectrum retains the vibrational bands of the oxides for both titanium oxide and copper oxide in the frequency range 422 and 569, The occurrence of a wavelength shift in the main peaks compared with the pure sample, and the overlap of the oxide and polymer bands, clearly indicates the occurrence of physical and chemical interactions and the successful encapsulation of oxide particles within the polymer matrices. These results are similar to those obtained using the same preparation method and tests carried out in the study[7].

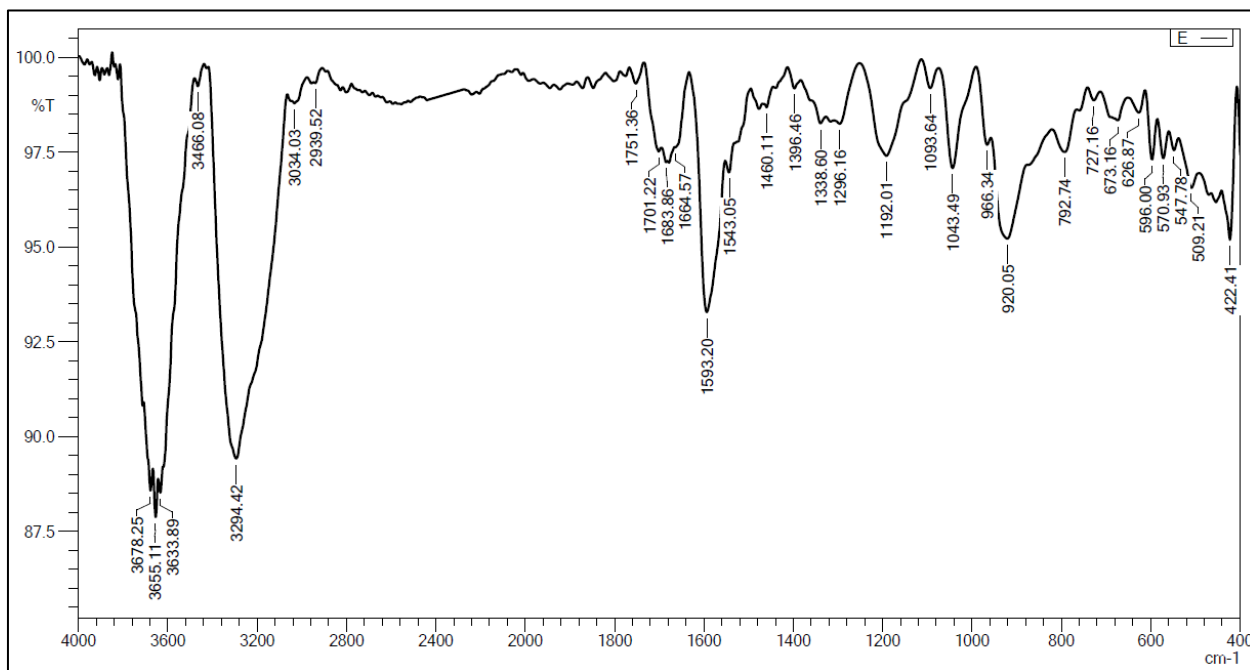


Figure 9: FTIR of the PPy/TiO₂-CuO hybrid nanocomposite

3.6 EDS Analysis

3.6.1 Pure polypyrrole (PPy) sample

Figure (10) clearly shows the presence of chlorine at a concentration of approximately (5.03W%), which generally results from successful doping of the polypyrrole chains using chloride ions derived from the oxidising agent used in the polymerisation process. We also investigate the cause of the presence of oxygen at a concentration of approximately (18.96 W%) is attributed to surface-adsorbed moisture and partial oxidation resulting from the polymerisation process in air. Furthermore, the presence of titanium at a concentration of (24.21 W%) indicates volatiles in the measurement chamber or contamination resulting from the preparation of the sample series on the same instrument , the result in this research agree with [20].

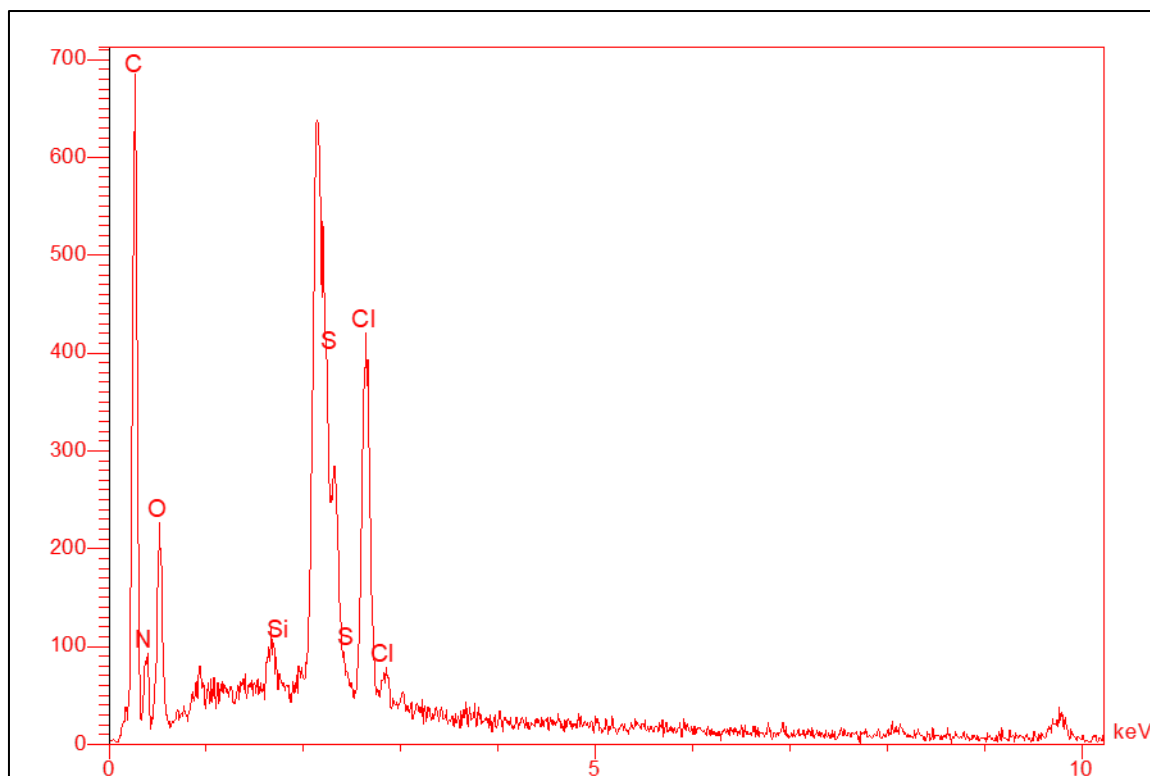


Figure 10: EDS of Pure polypyrrole (PPy)

3.6.2 Sample of the TiO_2 -CuO oxide compound

Figure (11) shows sharp, high-intensity peaks for titanium ($K\alpha$ peak at 4.5 keV) and copper ($K\alpha$ peaks at 40.9 keV and 0.8 keV), with a high oxygen content of approximately (52.12 A%) This provides clear and direct evidence of the purity of the oxide mixture and also indicates the high weight percentages of titanium and copper in the mixture, prepared with equal proportions of the two oxides. Furthermore, the weak appearance of the carbon peak (6.33 W%) is attributed to the carbon strip on the microscope slide These results have demonstrated the pure chemical decomposition of the element, and the dual and combined oxidation process involving chloride ions and sulphur dioxide is consistent with the research[19].

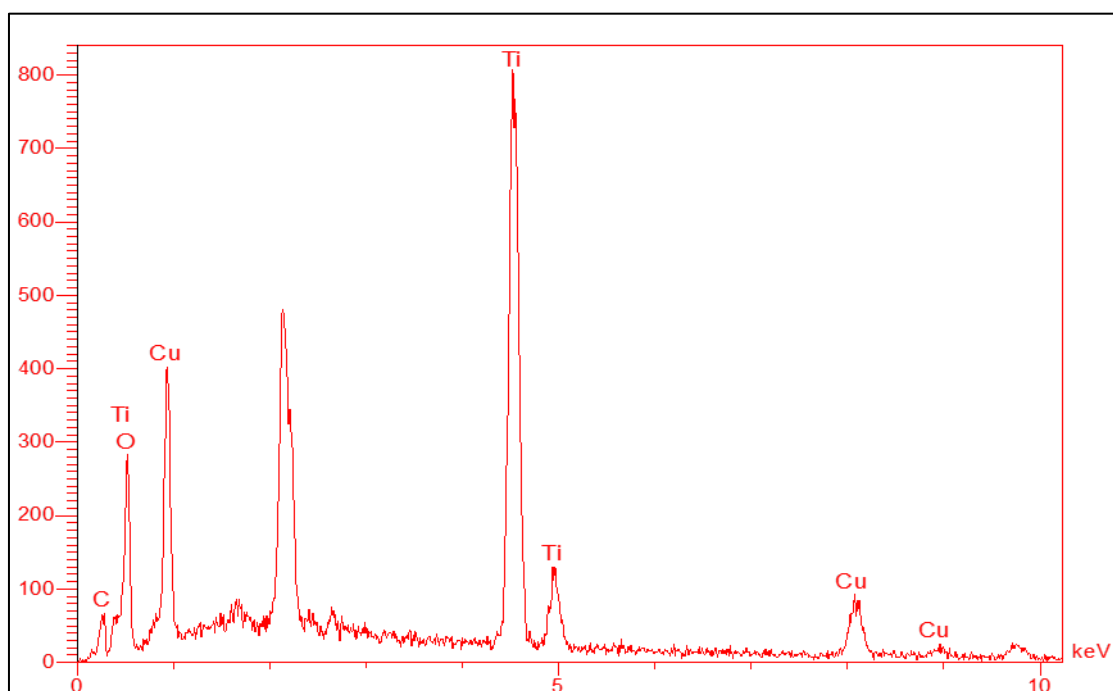


Figure 11: EDS of the TiO_2 -CuO oxide compound

3.7 Sample of the PPy/TiO₂-CuO hybrid nanocomposite

Figure (12) shows results that provide clear and definitive information on the organic polymer component, represented by C and N with atomic percentages exceeding approximately 79A%. This confirms the success of the polymerisation process in forming polypyrrole and the successful external encapsulation of the components added to it during the reaction. It is also possible to investigate the reason for the presence of chlorine at a concentration of 5.99W%, which may be attributed to the oxidising agent ions FeCl₃. The results also indicate the presence of sulphur at a proportion of approximately 9.50W%, attributable to the use of sulphuric acid in the preparation process as well as to the activation of the polymerisation process; this leads to the introduction of sulphate ions as an additional cross-linking agent that accelerates the stabilisation of the conductive material. It is also possible to examine the presence of oxygen, estimated at approximately 15.23W%, which indicates the oxidic charge of the compound on the side of the hydroxyl groups as well as the sulphate clouding agents Findings of this nature were reported in the research, which confirmed a similarity or correspondence with the study in this research[21].

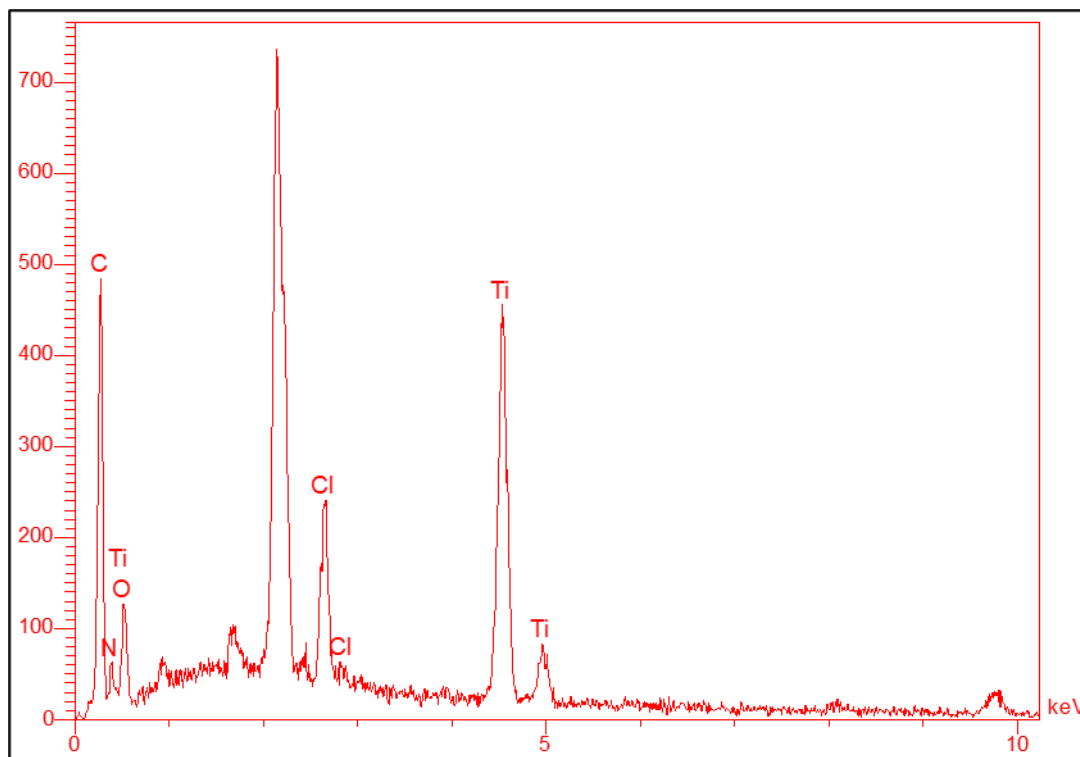


Figure 12: EDS of the PPy/TiO₂-CuO hybrid nanocomposite

3.8 CV Analysis

3.8.1 Pure polypyrrole (PPy) sample

Figure (13) shows the voltage curve, where the oxidation and reduction peaks are clearly visible above the current curve. These results confirm the polymer's high electrochemical response, which relies primarily on rapid, reversible oxidation and reduction reactions accompanied by the entry and exit of dopant ions within the polymer matrix. Regarding the peaks shown, for the oxidation peak, two distinct peaks appear during the positive potential sweep: the first peak occurs at a relatively late potential (-0.52 V) and represents the onset of the polymer chains' transition to the doped state; as for the second peak, it is a sharp peak—the strongest recorded—at a potential of approximately (-0.23 V), recorded at a higher current than the previous peak; this represents the peak of oxidation, which forms what are known as bipolarons in polypyrrole. There are other peaks representing reduction peaks, which appear in the reverse (negative) region and were recorded at a potential of approximately (-0.84 V). This peak corresponds to the ion exit process at (20.62 μA), indicating good and rapid conductivity in charge transfer. Furthermore, the presence of oxidation and reduction peaks is a strong indicator of the polymer's stability and smoothness during the ionic charging and discharging process. In conclusion, it can be said that the results demonstrate excellent electrochemical activity of the pure polymer, as evidenced by the high and sharp peak. Table (2) shows the voltage and current values for the peaks shown in the figure, as determined by the analyser.

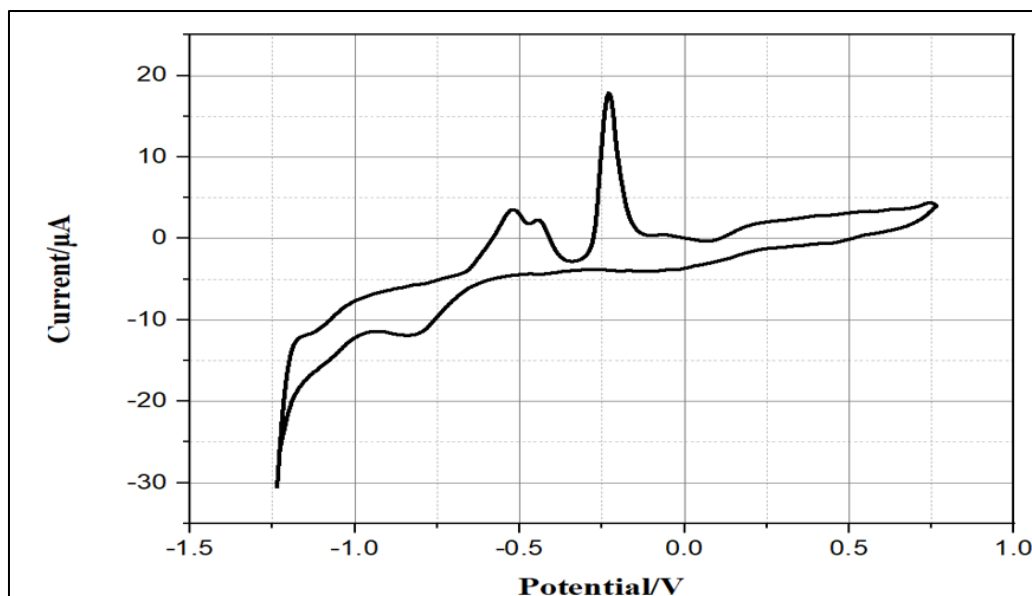


Figure 13: CV of Pure polypyrrole (PPy) sample

Table 2: The voltage and current values for the peaks

Peak	Sum/ $\mu\text{A/V}$	Min slope/ $\mu\text{A/V}$	Max slope/ $\mu\text{A/V}$	Y Offset/ μA	Height/ μA	Potential/V
1	55.6574	17.8008	37.8567	9.34644-	2.11185	0.97339-
2	837.072	340.298-	496.774	4.15974-	7.69792	0.52259-
3	837.072	340.298-	496.774	2.74082-	20.6104	0.23031-
4	837.072	340.298-	496.774	4.29490-	7.61371-	0.84459-

3.8.2 Sample of the $\text{TiO}_2\text{-CuO}$ oxide compound

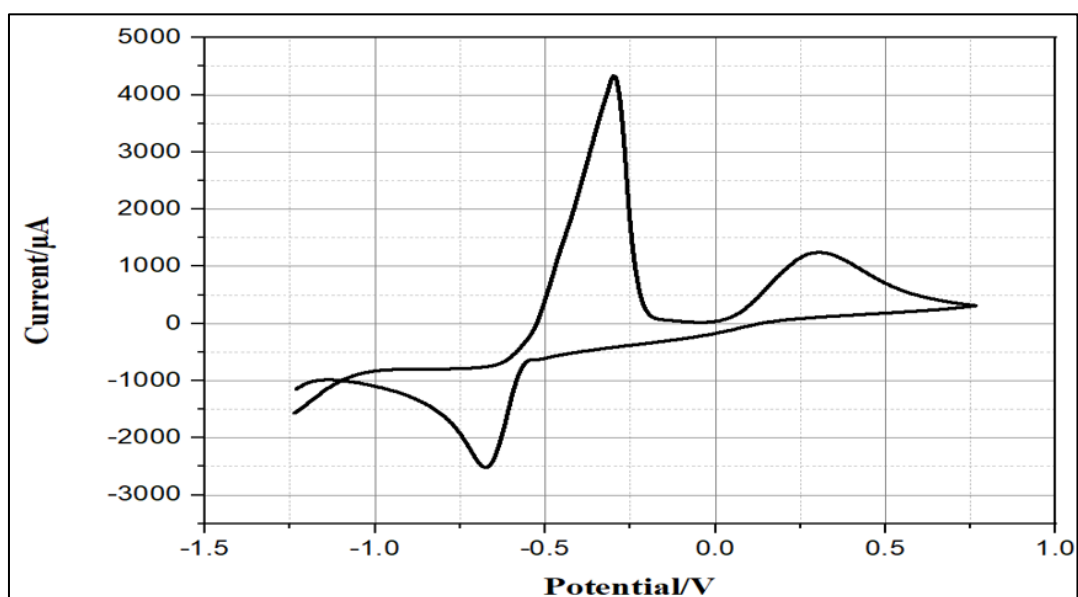
Figure 14: CV of the $\text{TiO}_2\text{-CuO}$ oxide compound

Figure (14) shows distinct peaks from oxidation and reduction processes, which are strong, sharp and independent at the same time. This behaviour is typical of transition metal oxides that undergo oxidation, reduction and electron transfer reactions at the surface. Furthermore, the current curve records a high response of approximately (1000–5000

μA), Furthermore, two oxidation peaks appear in the positive region, representing the oxidation transitions of copper and titanium, whilst a single strong and sharp reverse reduction peak was recorded in the negative region (-0.67 V). It can be said that these results indicate strong Faradaic activity, which demonstrates the high capacity of the oxide mixture to rapidly transfer charges via oxidation and reduction reactions. Table (3) shows the voltage and current values for the peaks recorded by the instrument.

Table 3: The voltage and current values for the peaks

Peak	Potential/V	Height/ μA	Y Offset/ μA	Max slope/ $\mu\text{A/V}$	Min slope/ $\mu\text{A/V}$	Sum/ $\mu\text{A/V}$
1	-0.29967	5042.24	-708.634	-675.307	-1092.84	1768.15
2	-0.67120	-1631.39	-882.245	-694.430	-920.310	1614.74
3	0.28984	952.220	285.872	6427.85	-3599.75	10027.6

3.8.3 Sample of the PPy/TiO₂-CuO hybrid nanocomposite

Figure (15) illustrates a dual electrochemical behavior curve, characterized by a hybrid response that integrates the capacity derived from the oxidation and reduction processes of polypyrrole, alongside the electrochemical properties of oxidation, which are further amplified by the surface activity of the oxides. Two distinct oxidation peaks are identified: the first occurs at (-0.52 V) and (-0.4 V), indicative of initial oxidation, while a more pronounced oxidation peak is noted at (-0.21 V) with a current measurement of ($11.73\text{ }\mu\text{A}$). In contrast, the reduction peak is observed in the negative voltage region, specifically at (-0.84 V). The potential values recorded by the instrument during the testing phase are summarized in Table (4).

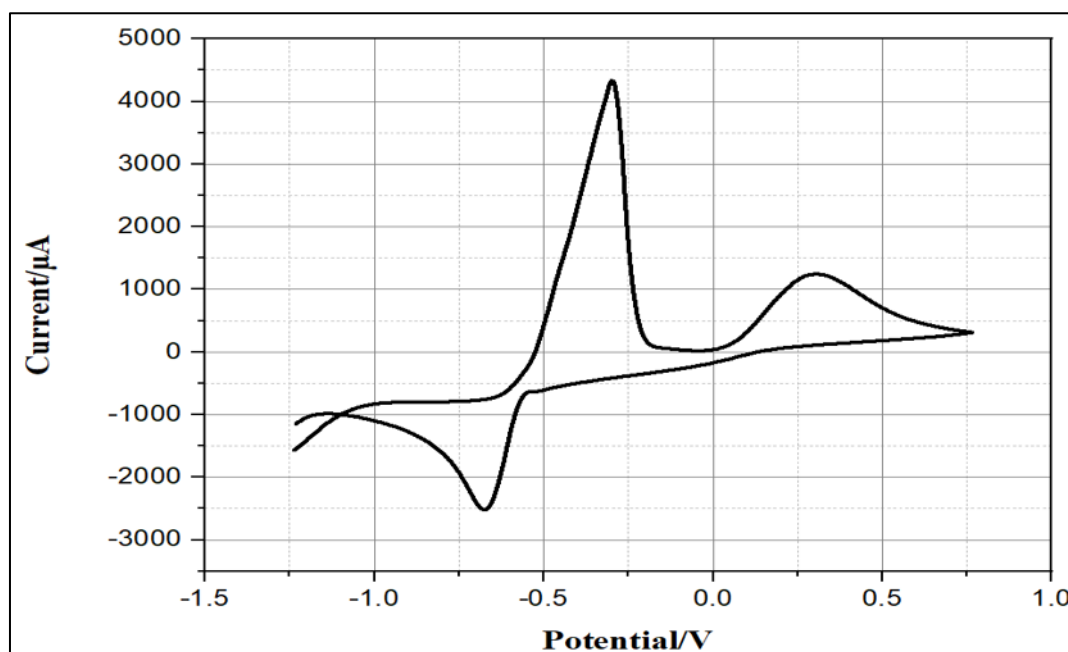


Figure 15: CV of the PPy/TiO₂-CuO hybrid nanocomposite

Table 4: The voltage and current values for the peaks

Peak	Potential/V	Height/ μA	Y Offset/ μA	Max slope/ $\mu\text{A/V}$	Min slope/ $\mu\text{A/V}$	Sum/ $\mu\text{A/V}$
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3	0.28984	952.220	285.872	6427.85	-3599.75	10027.6

3.9 UV-Vis Analysis

3.9.1 Pure polypyrrole (PPy) sample

Figure (16) shows the absorption spectrum of pure poly-pyrrole on its own, revealing high absorbance covering the visible region and extending into the infrared region, with a clear, continuous and gradual increase in absorbance towards longer wavelengths. Absorption peaks were recorded at wavelengths of 521 nm, 608 nm, 745 nm and 757 nm, reaching a final peak at a wavelength of 904 nm, where the spectral response demonstrated a high increase in electrical conductivity as well as the successful doping of originating from the oxidising agent. These results are consistent with those of the researcher [20] in his study, which demonstrated that the absorption spectrum of the doped poly-pyrrole exhibits broad peaks extending up to 700 nm; this correlates with electrical conductivity and results in a narrowing of the polymer's optical bandgap.

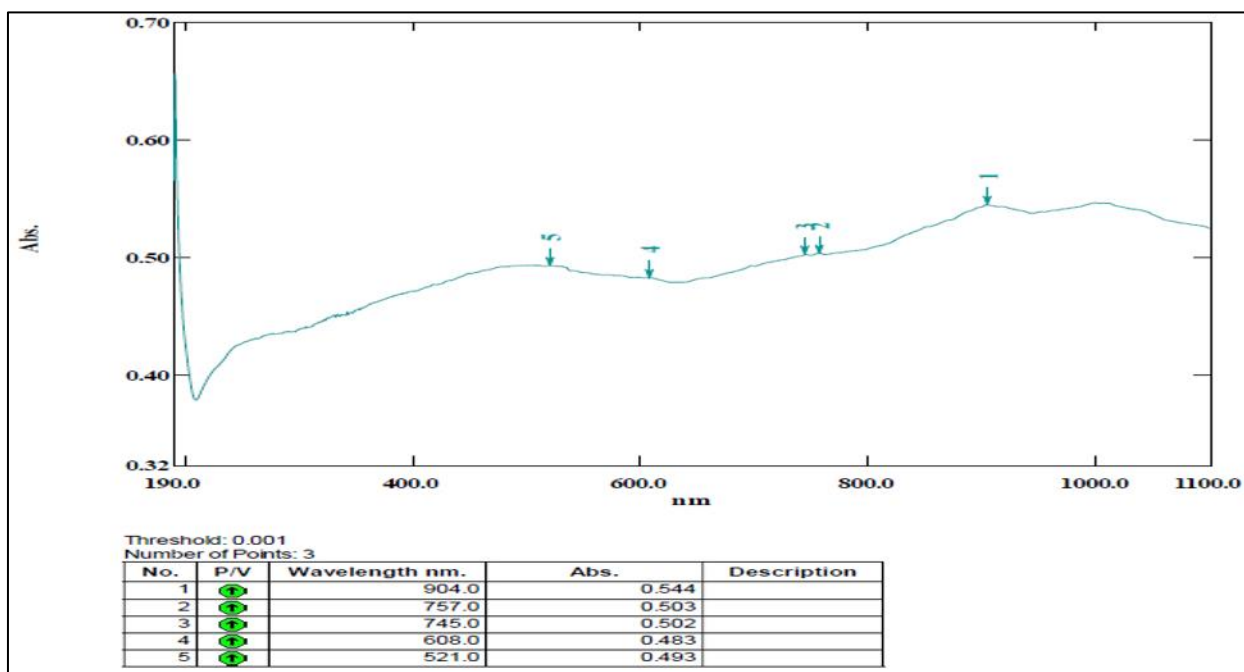
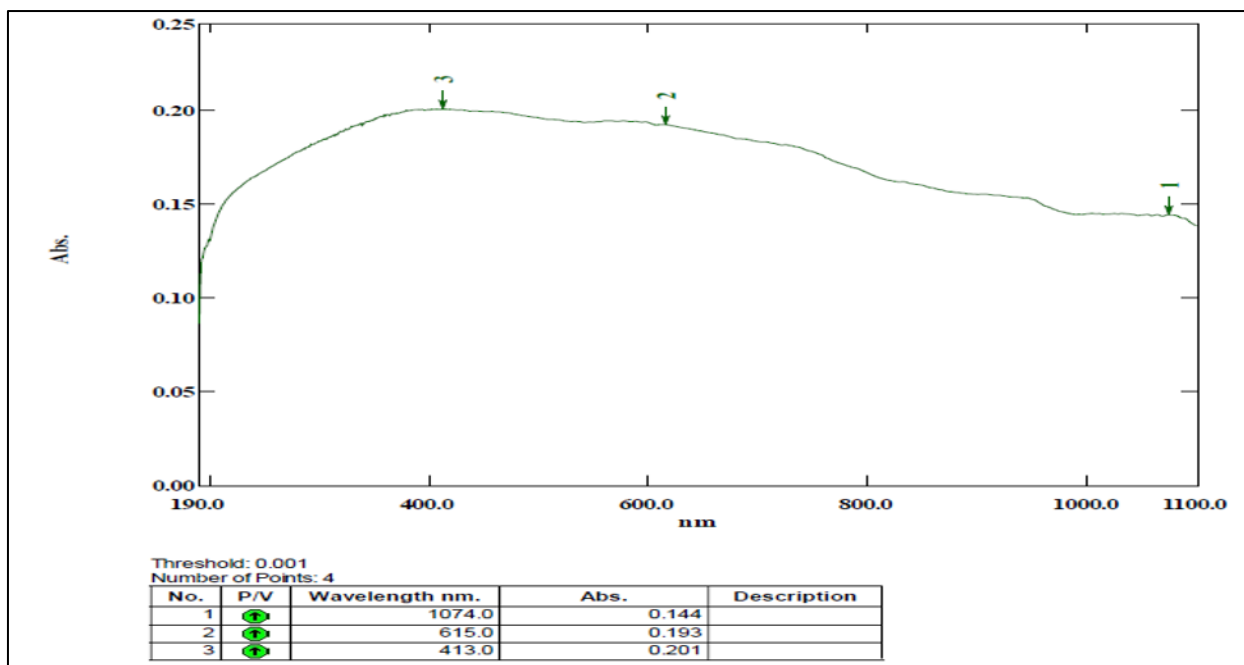


Figure 16: UV-Vis of Pure polypyrrole (PPy) sample

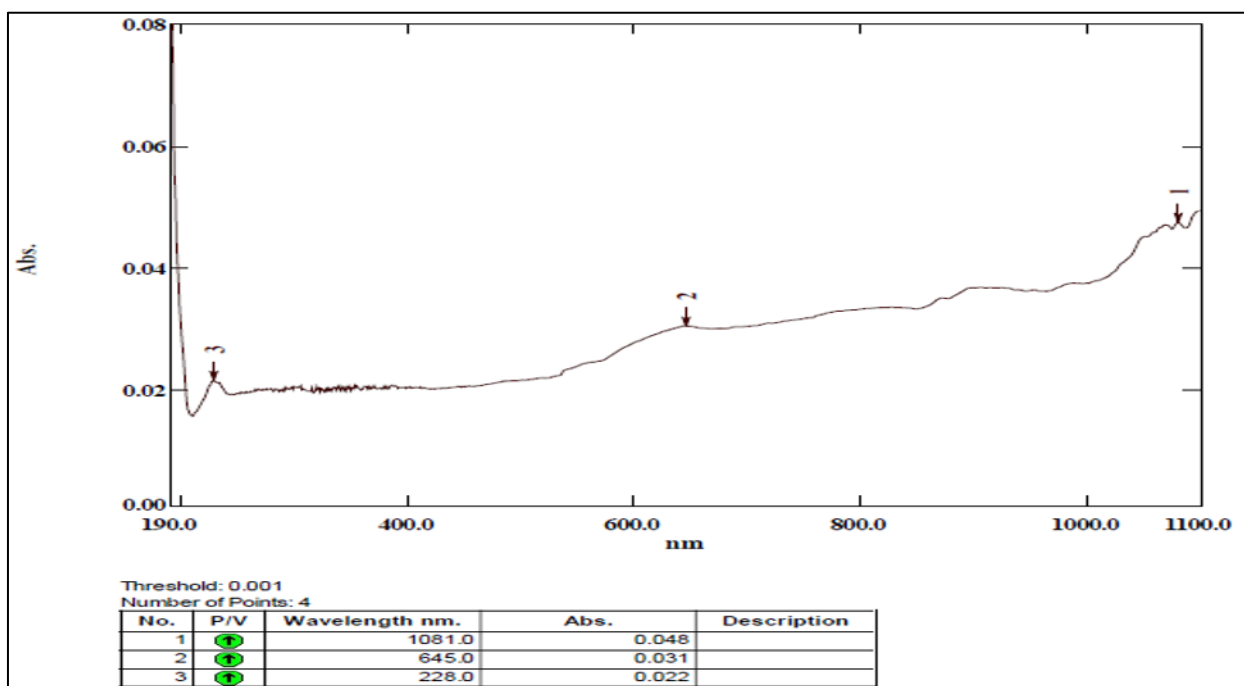
3.9.2 Sample of the TiO₂-CuO oxide compound

Figure (17) shows the absorption spectrum of a titanium dioxide and 50 per cent copper oxide blend. A distinct, broad-band response was observed, extending from a wavelength of 190 nm in the visible light region to the near-infrared region at a wavelength of 1100 nm. This result can be summarised by three peaks that were recorded, the first of which is at a wavelength of 413 nm at the absorption edge, which has been shifted towards lower energies; as for the second region, it was recorded at a wavelength of 615 nm, resulting from electronic transitions of copper ions present in monolayer copper oxide; as for the third peak, it was recorded at a wavelength of 1074 nm and is related to lattice defects and surface resonance states. This result is spectrally consistent with the findings of [17], in which the researchers noted the combination of monoclinic copper oxide with anatase titanium dioxide, leading to a noticeable red shift at the end of the absorption edge at wavelengths below 400 nm.

Figure 17: UV-Vis of the TiO₂-CuO oxide compound

3.9.3 Sample of the PPy/TiO₂-CuO hybrid nanocomposite

Figure (18) shows the absorption spectrum of the nano-composite with poly-pyrrole, which exhibits excellent hybrid behaviour, combining the properties of metal oxides with the absorbance of a conductive polymer, where a clear increase in absorbance is observed towards longer wavelengths, with three distinct peaks recorded. The first of these was recorded at a wavelength of 288 nm, resulting from a $\pi \rightarrow \pi^*$ electronic transition. This transition involves a contribution from both the polymer and titanium dioxide. As for the second distinct peak, it represents polymer absorption within the orbital transition of copper ions, recorded at a wavelength of 645 nm. As for the third peak, it was recorded at a wavelength of 1081 nm and represents the polarons band; this band confirms the occurrence of the doping process, which validated the success of the process by which a nanocoating was formed between the polymer and the oxides. This result is consistent with the work of researchers [15] in their study, which confirmed the successful encapsulation of oxides by polymers, resulting in absorption shifted towards the infrared region. This reduces the optical bandgap and supports charge transport in the hybrid compound.

Figure 18: UV-Vis of the PPy/TiO₂-CuO hybrid nanocomposite

4 CONCLUSION

Tests on the prepared samples demonstrated the success of the preparation process for the three materials, as well as the interplay between them and their electrochemical performance. The binary oxide composite ($\text{TiO}_2\text{-CuO}$), which accounted for 50 per cent of the mixture, was characterised by a sharp crystalline structure (monoclinic and anatase) and a morphology characterised by a granular, porous structure with very high purity of the constituent elements—titanium, copper and oxygen. This mixture exhibited the best performance in Faraday's law parameters, achieving an oxidation current of 5042. 24 μA at a voltage of (-0.299 V). This is due to the rapid transport of ionic charges through the transition oxides. As for polypyrrole, the results showed that it has a somewhat amorphous structure and a spherical morphology similar to cauliflower, with a successful chloride-ion-doped oxide doping process. It is noteworthy that it exhibited a transient capacitive response with an oxidation current of 20.61 μA at a potential of (-0.23 V). These results reflected strong, distinctive conductivity and rapid movement of ionic charges within the polymer. As for the material synthesised from pyrrole polymer with copper and titanium oxides, the results indicated properties common to both polymeric and crystalline materials, with a nanoscale encapsulated structure doped with sulphur and chlorine; this gives these materials the behaviour of a hybrid material with high ionic capacitance and long-term electrochemical stability

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

Disclosure of conflict of interest

The authors declare no conflicts of interest regarding this manuscript.

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