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# Polyaniline/metal oxide nanocomposites: synthesis, characterization and study of their applications

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## Abstract

This study included the synthsised of new nanocomposites using hydrothermal and sol-gel methods by preparing a mixture of metal oxides from their salts using the hydrothermal method, and then using the sol-gel method, the mixture of prepared metal oxides was reacted with polyaniline (as a substrate) prepared immediately through the polymerization of aniline (monomer). Nanocomposites consisting of polyaniline and various metal oxides (PANI-MO) were synthesized using a combination of hydrothermal and sol-gel techniques. This study employed metal oxides (MO) including zinc oxide (ZnO), copper oxide (CuO), and cobalt oxide (CoO). Metal oxides were added to the PANI to create nanocomposites nickelcobalt oxides (NiO: CoO) and nickel-copper oxides (NiO: CuO) were synthesized [NiO: CuO/PANI] and [NiO: ZnO/PANI] by the hydrothermal method and the sol-gel method, where a mixture of metal oxides is created and impregnated onto polyaniline, which serves as a support material. The nanocomposites were investigated using FT-IR, AFM, SEM and EDX techniques. Because of its cylindrical shape, variety in nature, and affordability, selective adsorption has been investigated by utilizing surfactants, which have made it unique and useful in a variety of applications. In this work, aniline was treated with sodium dodecyl sulfate (SDS) and hydrochloric acid (HCl) as a cross-linked method to change the composite of one polymer. SDS serves as an effective dispersing agent for aniline in aqueous solutions. The observed decrease in peak intensity may be attributed to the semi-crystalline nature of polyaniline (PANI) and the amorphous structure of the metal oxides (MO). Given these nano-characteristics of the synthesized nanocomposites, the research focused on exploring their potential applications in industrial and biological fields. Then next evaluated their industrial activities using anti-corrosion and biological processes using antioxidants. The effectiveness of the [NiO: CoO/ PANI] and [NiO: CuO/ PANI],[ NiO: ZnO/ PANI] nanocomposites were tested on antioxidant activity and showed a good scavenging percentage, where the highest antioxidant activity was obtained for the compounds [NiO: CoO/ PANI] and [ NiO: ZnO/ PANI]. Corrosion measurements revealed that the nanocomposite [NiO: CuO/ PANI] had the highest inhibition activity. PANI/CuO:NiO stands out as the most effective corrosion inhibition compound due to its superconductivity, strong protective layer formation, and synergistic effects between CuO and NiOleads to enhanced stability and corrosion resistance due to improved mechanical properties and chemical interactions.

**Keywords:** Poly aniline, Metal oxide, Nanocomposites, Hydrothermal method, Sol-gel method.

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# متراكبات نانوية من متعدد الانيلين/ خليط اكاسيد فلزية: تخليق وتشخيص ودراسة تطبيقاتها

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## الخلاصة

تضمنت هذه الدراسة تحضير مركبات نانوية جديدة باستخدام الطرق الحرارية المائية والسول-جل وذلك بتحضير خليط من أكاسيد المعادن من أملاحها باستخدام الطريقة الحرارية المائية، ومن ثم باستخدام طريقة السول-جل تم تفاعل خليط أكاسيد المعادن المحضرة مع البولي أنيلين (كمادة ركيزة) المحضر بشكل فوري من خلال بلمرة الأنيلين (المونومر) ثم تم تحضير مركبات البولي أنيلين/أكسيد الفلز المختلط PANI-MO باستخدام العمليات الحرارية المائية والسول جل. في هذا العمل، تم استخدام أكاسيد المعادن (MO) مثل ZnO، CuO، CoO. تمت إضافة أكاسيد المعادن إلى PANI لإنشاء مركبات نانوية، وتم تحضير أكاسيد النيكل والكوبالت (NiO: CoO) وأكاسيد النيكل والنحاس (NiO: CuO) واكاسيدالنيكل والزنك ( NiO:ZnO) وتم تحضير [NiO:CoO/PANI],[NiO: CuO/ PANI]و [NiO: ZnO/PANI] بالطريقة الحرارية المائية و طريقة sol-gel، حيث يتم إنشاء خليط من أكاسيد المعادن وتشريبها على البولي أنيلين، الذي يعمل بمثابة مادة داعمة. تم فحص المركبات النانوية باستخدام تقنيات FT-IR، AFM، SEM وEDX. نظرًا لشكله الأسطواني، وتنوع طبيعته، وسعره المعقول، فقد تمت دراسة الامتزاز الانتقائي باستخدام المواد الخافضة للتوتر السطحي، مما جعله فريدًا ومفيدًا في مجموعة متنوعة من التطبيقات. في هذا العمل، تم معالجة الأنيلين بكبريتات دوديسيل الصوديوم (SDS) وحمض الهيدروكلوريك (HCl) كطريقة متصالبة لتغيير مركب بوليمر واحد. في المحلول المائي، يعمل SDS بشكل جيد كمشتت الأنيلين. تجدر الإشارة إلى أن PANI شبه بلوري و MO غير متبلور . فمن بين هذه الخصائص النانوية التي تم النظر فيها للمركبات النانوية المركبة، كان الاتجاه هو استثمار هذه الخصائص أو الصفات في التطبيقات الصناعية والبيولوجية. ثم تم بعد ذلك تقييم أنشطتهم الصناعية باستخدام العمليات المضادة للتآكل والعمليات البيولوجية باستخدام مضادات الأكسدة. تم اختبار فعالية المركبات النانوية [NiO: CoO/ PANI] و[NiO: CuO/ PANI] و[NiO: ZnO/ PANI] على نشاط مضادات الأكسدة وأظهرت نسبة كسح جيدة، حيث تم الحصول على أعلى نشاط مضاد للأكسدة للمركبات [NiO: CoO/ PANI] و [NiO: ZnO/ PANI]. أظهرت قياسات التآكل أن المركب النانوي [NiO: CuO/ PANI] كان له أعلى نشاط تثبيطي. PANI/CuO: يبرز NiO باعتباره مركب منع التآكل الأكثر فعالية بسبب الموصلية الفائقة وتكوين طبقة واقية قوية وتأثيرات تآزرية بين CuO وNiO تؤدي إلى تعزيز الاستقرار ومقاومة التآكل بسبب الخواص الميكانيكية المحسنة والتفاعلات الكيميائية.

## 1. Introduction

In recent years, there has been a significant surge in the development and application of polymers in natural environments, marking a substantial advancement in this field. Directing polymers and their subordinates for tissue design applications has sparked a ton of interest. Metals and inorganic semiconductors show electrical and optical properties that are like CPs [1]. The interesting electrical and optical characteristics of naturally conducting polymers (ICPs) have made them an emerging area in material research [2]. The potential practical application of conductive polymers in energy conversion systems, such as photovoltaic and solar cells, biosensors, and others, has recently drawn significant attention in the field of organic electronics [3]. The polarons and bipolarons that are joined with counterions to form the polymer during preparation are thought to be the main carriers of charge for the PANI [4]. PANI has unique physical and chemical qualities like good electrical conductivity and great environmental stability, making it one of the most attractive conductive polymers. This is because its chain contains NH group. Furthermore, PANI has the ability to be manufactured chemically and electrochemically, is very affordable, and is very light. As a result, PANI is used in a variety of applications, such as the manufacturing of sensors, batteries, electronic devices, and supercapacitors [5]. Nanocomposites are created by hydrothermal and sol-gel processes. at the nanoscale to get the desired properties. Because nanoparticles (NPs) have a larger surface area that enables them to interact more than regular or micro-sized materials, which reduces the empty space between polymer lattices and improves their execution and Resistant qualities. Because of their inherent properties $Al_2O_3$, $SiO_2$, $TiO_2$, $ZrO_2$, CoO, and ZnO are the NPs that are most frequently used in coatings [6]. The study of nanostructures incorporating conductive polymers, such as PANI and polypyrrole, along with their nano-layered composites, has emerged as a fascinating area of research. This field shows promise in the development of innovative materials for cutting-edge technologies [7]. The nano-layered state of metal oxide/polymer composites uncovers further developed electrical and attractive properties compared to primitive metal oxide and conduction polymers. The transporters of charge for the PANI are generally respected to be polarons and bipolarons, which are steadied by counterparticles joined into the polymer during the readiness[8]. As of late, the directing polymer metallic oxide nanocomposites have been considered another class of materials because of their better properties when compared to those of polymers and metal oxide [9]. PANI and MO worked to improve execution and work on mechanical elements. PANI/MO nanocomposites are essential in several scientific disciplines, such as materials science and physical science[10]. MO nanoparticles have specific chemical and physical characteristics. Effective preparation of nanocomposites using material and electrochemical arrangement techniques these nanocomposites have a wide range of properties. The several metal and MO particles that have been incorporated into PANI to create nanocomposites [11]. The science and design of nanocomposites have found applications in many industries, including biomedical, plastics, ceramics, electronics, and metals [11]. The release of plastic additives, enhanced bioavailability, and a sizable percentage of exposed surface molecules are all present [12]. Aside from their low cost of manufacturing, plastics have a broad spectrum of chemical and physical properties. Numerous industrial applications employ them, and they are mass-produced [13]. This study aimed to synthesize PANI/MO NPs using a sol-gel method as a chemical method that affected the properties and performance of nanocomposites, where AFM, FTIR, XRD, and SEM/EDX were used to analyze the films. Surface roughness, crystal structure and morphological characteristics were also examined, providing a valuable information about the nanocomposite materials[14].

## 2. Materials and method

Aniline $C_6H_7N$ (ANI) (Pub chem) (99.5%), zinc acetate tetrahydrate ($Zn(CH_3CO_2)_2.4H_2O$) (BDH chemical Poole, England), copper sulfate pentahydrate ($CuSO_4.5H_2O$), cobalt acetate tetrahydrate ($Co(CH_3CO_2)_2.4H_2O$), nickel (II) acetate ($Ni(CH_3CO_2)_2$) and nickel (II) chloride hexahydrate ($NiCl_2.6H_2O$) and Hydrochloric acid were purchased from Sigma-Aldrich company, with particle size < 10 µm and purity of 98% since it was used without any further purifications, cobalt acetate tetrahydrate ($Co(CH_3CO_2)_2.4H_2O$), nickel (II) acetate ($Ni(CH_3CO_2)_2$) and nickel (II) chloride hexahydrate ($NiCl_2.6H_2O$). Distilled water was used as a medium for the polymerization of aniline.

### *2.1 Synthesis of [NiO: CoO] metal oxide*

Two salts are utilized in this process. Firstly, 3.12 grams of nickel acetate (Ni(CH3CO2)2), equivalent to 0.5 M, is dissolved in deionized water to prepare the first oxide precursor. The second salt, $Co(CH_3CO_2)_2.4H_2O$ (3.1135 gm, 0.5 M), is taken with the water that has been deionized To prepare the second oxide. At that point, the two salts are blended then added to to NaOH (0.08 gm, 0.5 M) after being disintegrated with 10 ml of deionized water. Next, the mixture is taken and put into the cell for Teflon direct autoclaving, where it is warmed for 24 hours the warming rate is increasing by (2°C/min). After cooling the cell, the mixture was separated using a 4000 rpm centrifuge for 15 minutes. The resulting compound was then dried at 75 °C in an oven.

### *2.2 Synthesis of nanocomposite of PANI\ [NiO: CoO] composite by in situ polymerization method using Sol-gel method*

The *PANI\ [NiO: CoO]* nanocomposite was prepared by using ammonium persulfate (APS) as an oxidizing specialist in an acidic solution to facilitate aniline oxidative polymerization in situ with CoO: NiO nanoparticles. To create the *PANI\ [NiO: CoO]* nanocomposite, 70 ml of watery solutions containing 2 M HCl and 4.5 ml of refined aniline were vigorously mixed for 30 minutes at room temperature by a magnetic stirrer. Subsequently, 0.05g of SDS and 4.5 g of APS were dissolved in 20 mL of deionized water containing 10 wt% CoO: NiO and added drop by drop to the blended. Upon the addition of ammonium persulfate (APS) containing a mixture of cobalt oxide (CoO) and nickel oxide (NiO), the color of the resultant compound transitioned from blue to green, indicating an acceptable degree of polymerization. In addition, the blending continued for two hours. The material was then dried out and sieved. The composite PANI\ [NiO: CoO] (10 wt%) was obtained.

### *2.3 Synthesis of [NiO: ZnO] metal oxide*

The first salt, Ni $(CH_3CO_2)_2$ (3.73 gm, 0.5 M), it was dissolved in 25 ml of deionized water to prepare the first oxide. The other salt, $Zn(CH_3CO_2)_2.4H_2O$ (2.29 gm, 0.5 M), was taken from 25 ml of deionized water and disintegrated in 25 ml of deionized water to form the subsequent oxide. At that point, the mixture of arrangements (1) and (2) was added to NaOH (0.08 gm, 0.5 M) after being broken up with 10 ml of deionized water. After that, the mixture was put in the Teflon direct autoclave cell and heated for 24 hours at a rate of 2 °C / min to 150 °C. Centrifugation for 15 minutes at 4000 rpm was used to isolate the mixture after the cell had cooled. Following that, The resulting compound was dried in an oven at 75 °C .

### *2.4 Synthesis of nanocomposite of PANI/[NiO:ZnO] composite by in situ polymerization method using Sol-gel method*

The *PANI/[NiO: ZnO]* nanocomposite experiment was synthesised by using ammonium persulfate (APS) as an oxidizing agent in an acidic solution to facilitate aniline oxidative polymerization in situ with ZnO/NiO nanoparticles added. To synthesis of *PANI/[NiO: ZnO]* nanocomposite, 70 mL of a 2 M HCl solution and 4.5 ml of refined aniline were thoroughly mixed for 30 minutes at room temperature in a magnetic stirrer. Subsequently, 0.05g of SDS nanoparticles and 10% weight percent ZnO: NiO was added wisely to the aforesaid blended solution after 4.5 g of APS had been Dissolved in 20 milliliters of deionized water. The composite's color changed from blue to green when the APS with ZnO:NiO was added, indicating an acceptable degree of polymerization. Additionally, mixing lasted for two hours. The material was then dried out and sieved out at that moment. The composite *PANI/[NiO:ZnO]* (10 wt%) was obtained.

### *2.5 Synthesis of [NiO: CuO] metal oxide.*

In order to create binary metal oxide composites with CuO and NiO in the same molar ratio, 0.5 M $CuSO_4.5H_2O$ solution and 0.5 M $NiCl_2.6H_2O$ solution were separately combined with the same volume of 1 M NaOH solution. After one hour of stirring the latter solution at room temperature, it was added to the former solution. Following, the entire mixture was placed in an autoclave for a 24-hour hydrothermal reaction at 180°C. Centrifugation was used to separate the mixture when the cell cooled for 15 minutes at 4000 rpm. Following that, The resulting compound the material was dried in a furnace at 75°C. The dehydrated powder was placed inside a muffle and dried for an extra three hours at 300 °C.

### *2.6 Synthesis of nanocomposite of PANI/[NiO: CuO ] composite by in situ polymerization method using Sol-gel method.*

To prepare *PANI/[NiO: CuO ]*, Ammonium Persulfate (APS) was used as an oxidizing agent in an acidic solution to prepare NiO nanocomposite which aided in the in situ oxidative polymerization of aniline in a solution consisting of CuO and nickel oxide (NiO) nanoparticles. 4.5 mL of refined aniline and Using a magnetic stirrer 70 mL of a 2 M liquid HCl solution was rapidly mixed for 30 minutes at room temperature to create the *PANI/[NiO: CuO ]* nanocomposite. Subsequently, 0.05g of SDS and 4.5 g of APS were melt in 20 mL of deionized water containing 10 weight percent CuO/NiO and added drop by drop to the mixture mentioned above. The color of the arrangement shifted from blue to green upon the addition of APS with CuO:NiO, indicating a satisfactory level of polymerization. In addition, the blending continued for two hours. The material was then separated and allowed to dry at that stage. The result was the composite PANI/[NiO: CuO ] (10 wt%).

## 3. Results and Discussion

To characterize the properties of synthesised nanocomposites, a series of techniques were employed such as AFM device of model AA3000/ Angstrom Advanced USA, which was used to determine the roughness PANI-nano- composites, while, Fourier Transform Infrared Spectroscopy (SHIMADZU FT-IR 8400S/Japan) was used to determine the chemical components and absorption band of synthesised material. Finally, a SEM/EDS device was used to monitor the distribution of nano-NPs within the PANI, to assess a variety of material properties, morphology and dispersion of nanoparticles and nanocomposites.

### *3.1 Atomic force microscopy (AFM)*

Atomic force microscopy (AFM) analysis was conducted using a microscope model AA3000, manufactured by Angstrom Advanced Inc., USA. The appearance of the surface and harshness of the *PANI\ [NiO: CoO]* nanocomposite formed using an aqueous process are shown in Table 1, Figure 1. The outcomes confirm the roughness average (Ra) was 8.49 nm, and 10.76 nm was the root mean square roughness (Rq). The examination of the molecular-size nanocomposite yielded a mean value of 53.99 nm for the molecule sizes. As shown in Figure 2, the results of the *PANI/[NiO: CuO]* nanocomposite test show that the surface (Ra) has decreased to 7.43 nm, with a root mean square deviation (Rq) of 9.70 nm. The graph reveals a mean translational diffusion width of 61.90 nm, which corresponds to the measured size of the nanoparticles [12]. The AFM architecture of PANI/ [NiO: ZnO] polyaniline NiO and ZnO is shown in Figure 3 individually. We saw the rising stacking of ZnO in the polyaniline increment. The dimensions of the polymer were similar to packs. The distinctions in the surface shape are because of the solid communication between the polymer and the nanoparticles because of the various stackings of ZnO. A common high goal, the consequences of the *PANI/ [NiO: ZnO]* nanocomposite test demonstrate a decline in the surface harshness (Ra) to 7.93 nm, with a root mean square deviation (Rq) of 9.70 nm, as displayed in Figure 3. The mean width circulation of nanoparticles in the chart shows that the molecule size range was 65.01 nm, as seen in Figure 3, with a root of the square variance (Rq) of 9.70 nm. According to the chart's mean width circulation of nanoparticles, the range of molecule sizes was 65.01 nm [13].

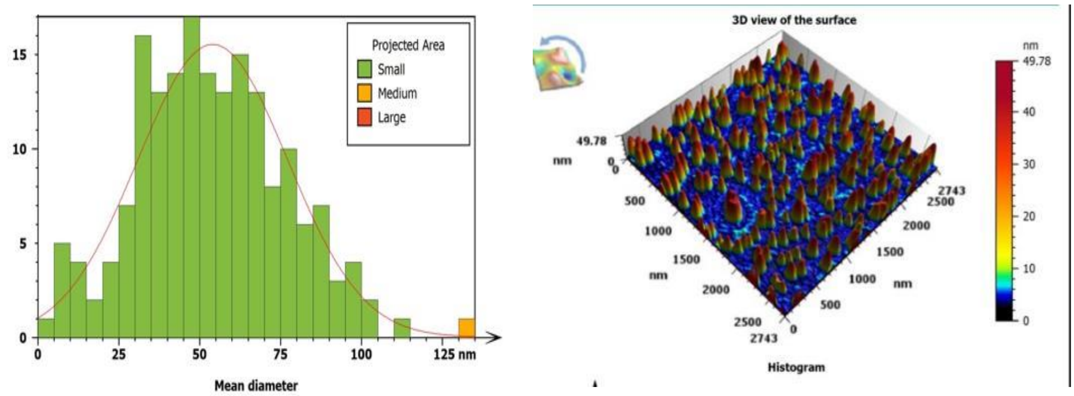


**Figure 1:** shows the synthesized CoO:NiO and PANI nanocomposite.

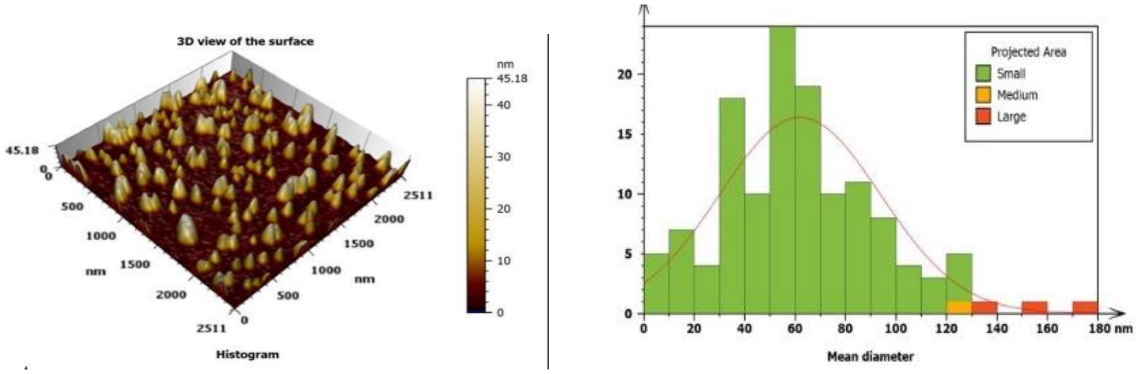


**Figure 2:** AFM analysis image and particle size distribution of the [CuO: NiO / PANI] nanocomposite.

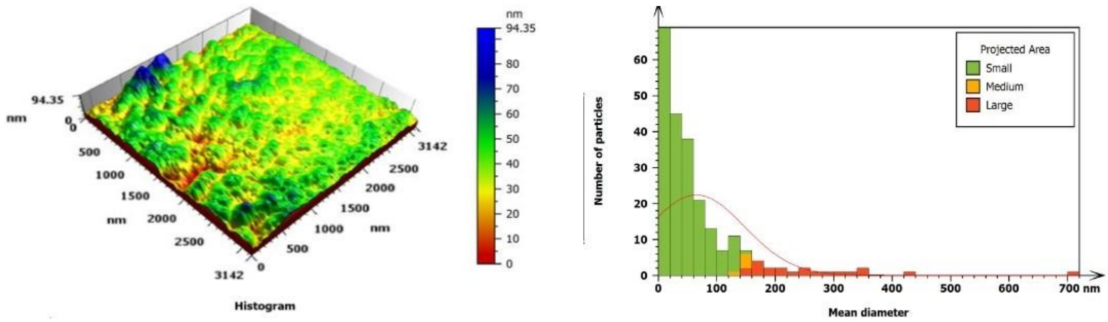


**Figure 3:** AFM image and particle size distribution of ZnO: NiO and PANI nanocomposite.

**Table 1:** Calculated information from nanocomposites' AFM measurements

| Composites | Ra (nm) | Rq (nm) | Mean diameter (nm) |
|---|---|---|---|
| PANI/ [NiO : CoO] | 8.49 | 10.76 | 53.99 |
| PANI/ [NiO : CuO] | 7.43 | 9.70 | 68.04 |
| PANI/ [NiO : ZnO] | 7.93 | 9.70 | 65.01 |

### *3.2 Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy (SEM/EDX)*

The surface morphology of the various PANI/MO composite films was examined using Field Emission Scanning Electron Microscopy (FE-SEM) on a Quattro S model from Thermofisher, Czech industry, to gain insights into their topographical features. the resulting samples are displayed in Fig. 4. PANI/CoO: NiO sporadic particles have aggregated and a few spikes are protruding from them. Minute circular particles are attached, maintaining a uniform conveyance. Fig. 5 gives all the data about the weight percent and nuclear percent of the singular molecules present in the particular mixtures. From the ranges, it very well may be seen that the normal components exist in the composites, and the extent of PANI is CoO: NiO is coordinating with its compositional proportion [14].

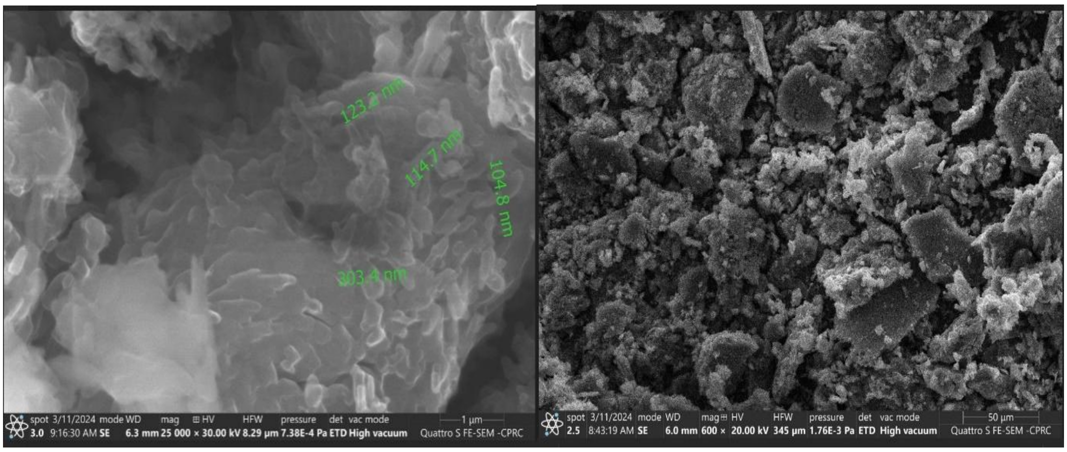
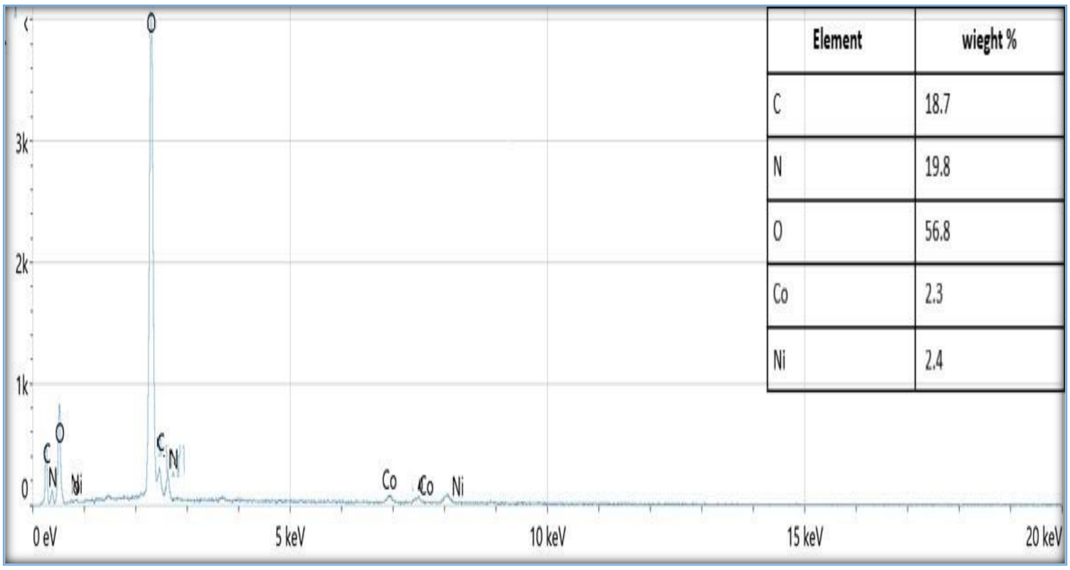


**Figure 4:** SEM images of PANI/CoO:NiO nanocomposite .



| Element | wieght % |
|---|---|
| C | 18.7 |
| N | 19.8 |
| O | 56.8 |
| Co | 2.3 |
| Ni | 2.4 |

**Figure 5.** EDS of PANI/CoO: NiO nanocomposite.

The surface morphology of CuO, NiO nanoparticles, and PANI nanocomposite is shown in Fig. 6. The SEM image of the PANI/CuO: NiO nanoparticles shows small, granular, and circularly created particles. The total number of particles during washing also results in a conglomerate. The SEM image presented in Figure 6 revealed areas of robust integration between the CuO:NiO particles and the structure of the PANI chain in the nanocomposite. This fusion appears to result in a reduction of crystallinity in the material. This is a result of PANI's amorphous concept. Because of the oxide particles present in the SEM image of PANI-CuO: NiO nanocomposites, certain smooth and robust blocks can be observed, contributing to the glass-like appearance of the carefully crafted materials. The CuO nanoparticle arrangement is confirmed to be free of contaminants by the EDX range shown in Fig. 7. The range reveals the pure and stoichiometric nature of the combined nanoparticles [15].

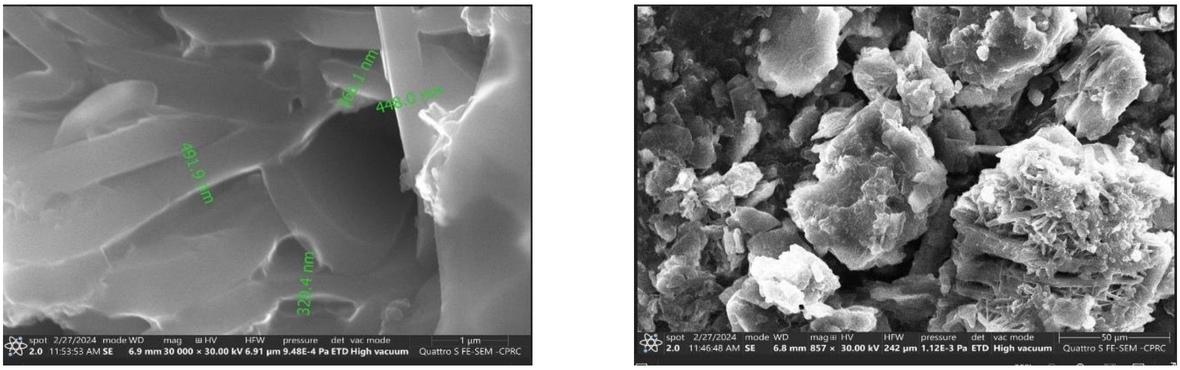
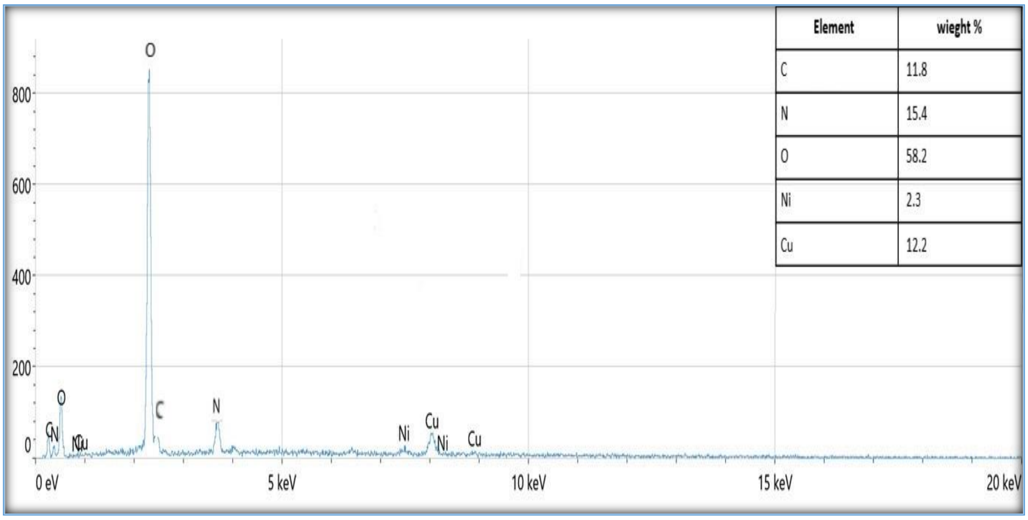


**Figure 6**. SEM images of PANI/CuO:NiO  nanocomposite .



| Element | wieght % |
|---|---|
| C | 11.8 |
| N | 15.4 |
| O | 58.2 |
| Ni | 2.3 |
| Cu | 12.2 |

**Figure 7:** EDS of PANI/CuO: NiO nanocomposite.

In Figure 8, SEM images appear of the outer layer of the movies obtained by saving the colloidal scatterings and can notice the development of a homogeneous film of PANI particles. It was also present an SEM image of newly pre-arranged ZnO: NiO tests, where pole-like designs can be recognized. Nonetheless, as it tends to be found in Figure 8, a more sinewy construction is by all accounts related to the ZnO_NPs/PANI composites. An EDS examination of the last Fig. 9 can provide significant data about their compositions: while the NiO and ZnO tops are and to the PANI substrate utilized, individually, construction of the ZnO_NPs/PANI [16].

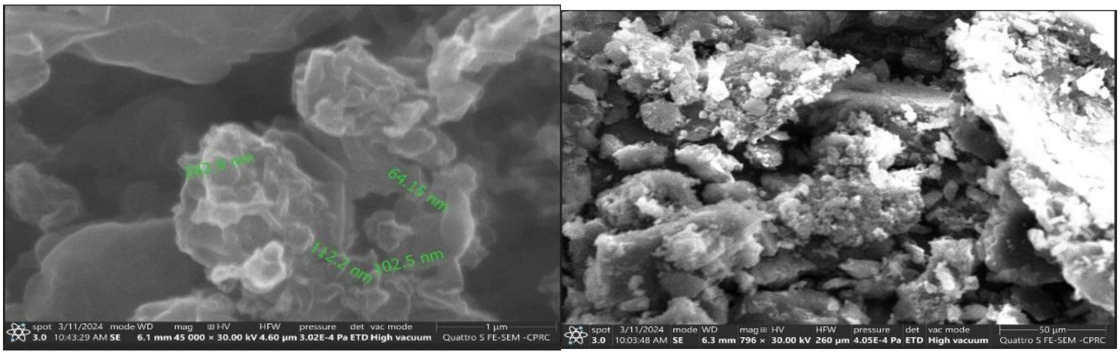


**Figure 8.** SEM images of PANI/ZnO:NiO nanocomposite.

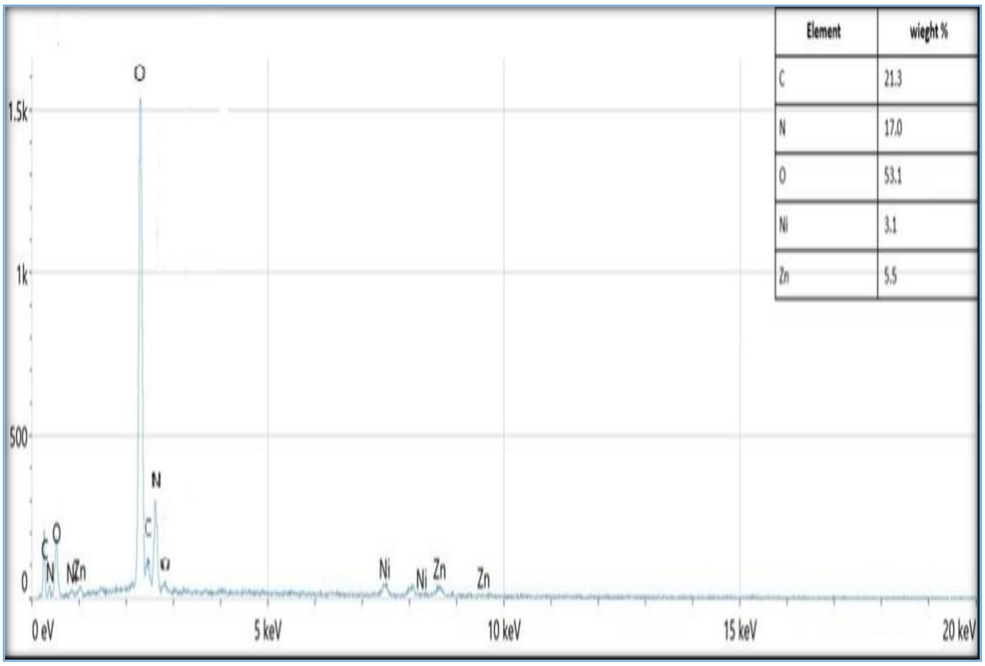


**Figure. 9:** EDS of PANI/ZnO: NiO nanocomposite.

*3.3 Fourier transform infrared spectroscopy analysis (FT-IR).*

Fourier Transform Infrared Spectroscopy (FT-IR) was used to determine the chemical components and absorption band of synthesised compounds. The frequency range used for the FTIR analysis was 4000–400 cm-1. The blended pure polyaniline (PANI)/CoO: NiO FT-IR spectrum Figure 10. deals with the FT-IR spectra of pure PANI, CoO, and NiO mixed. The characteristics of PANI's pinnacles include expansive pinnacles between 3016.67 and 3417.86 cm$^{-1}$, which relate to (N-H extending vibrations of amine), and sharp tops at 1560.41 cm$^{-1}$ (C=C extending the vibration of Quinonoid ring (N=Q=N)), 1535and 1487 cm$^{-1}$ ((C=C extending the vibration of the benzenoid ring (N-B-N)), 1244.09 and 1298.09 C–N stretching of the benzenoid ring, 1130.29 cm$^{-1}$ (C-H), 611.43, 719.45, and 804.32 cm$^{-1}$are attributed to C-H vibrations [17]. in the pure CoO: NiO FT-IR spectra. As can be seen, the main groups of interest for the PANI/CoO: NiO test appeared at approximately 3400–3500 cm$^{-1}$1050 cm$^{-1}$, and 500–650 cm$^{-1}$. These values correspond to the presence of individual practical groups for hydroxyl (O-H extending), metal hydroxyl (Co, Ni extending), and metal oxide (Co, Ni-O extending). The ingestion groups of all mixture tests were compared with unadulterated CoO-NiO. Additionally the peak at 1528 cm$^{-1}$relates to the C=C stretching of the benzenoid rings [18]. The band at 1037 cm$^{-1}$in the spectra of the NiO and PANI samples is explained by the merging of bands at 1049 and 1021 cm$^{-1}$, which came from the C^O stretching bond between NiO and PANI [19]. FT-IR spectra of both the CuO:NiO mixture and all nanocomposite samples were obtained using potassium bromide (KBr) as the medium for sample preparation.The IR spectra of the PANI/CuO: NiO nanocomposites are displayed in Figure 11. For the quinonoid and benzenoid units of PANI, the bands at 1560 cm$^{-1}$and 1498 cm$^{-1}$ are ascribed to the C=N and C=C stretching modes of vibration. The benzenoid ring's C-N stretching mode is attributed to the peaks located approximately at 1300 cm$^{-1}$. The presence of CuO: NiO in the nanocomposite is the cause of the bands in the 480–510 cm$^{-1}$region. These findings suggest that PANI and CuO: NiO nanoparticles may interact in certain ways [20]. Ni-O bonding is the cause of the absorbance band at 474 cm$^{-1}$. It is indicated by the presence of this bond that NiO nanoparticles are forming and being incorporated into NiO/PANI nanocomposites [21]. PANI /ZnO: NiO nanocomposites FT-IR spectra in Figure 12 were employed to examine the connection between ZnO nanoparticles and PANI. Quinone and benzene rings, respectively, are responsible for the peaks at 1562 cm$^{-1}$and 1495 cm$^{-1}$. The peaks located at 1562 $cm^{-1}$ and 1496 $cm^{-1}$are attributed to the asymmetric and symmetric extension vibrations of phenyl ring. The C-H appeared at the peak at 2875 $cm^{-1}$. Figure 12 displays the PANI/ZnO:NiO nanocomposite's FT-IR spectra of metal oxides inside the nanocomposite, specifically at 3223, 852, 745, and 477 $cm^{-1}$. N-H bonding is responsible for the peak at 3223 cm1, while Zn-O-Zn bond-free oxides are linked to the peaks at 852, 745, and 477 $cm^{-1}$[22]. FT-IR spectroscopy was employed to validate the successful synthesis of PANI-NiO nanocomposites and to identify the specific absorption bands corresponding to stretching and bending vibrations, thereby characterizing the molecular structure of the produced materials. Pure PANI, pure NiO, and PANI -NiO nanocomposites with varying NiO NP loadings are all shown in their FT-IR spectra. Ni-O stretching vibration is seen in NiO NPs at 411 $cm^{-1}$[23].

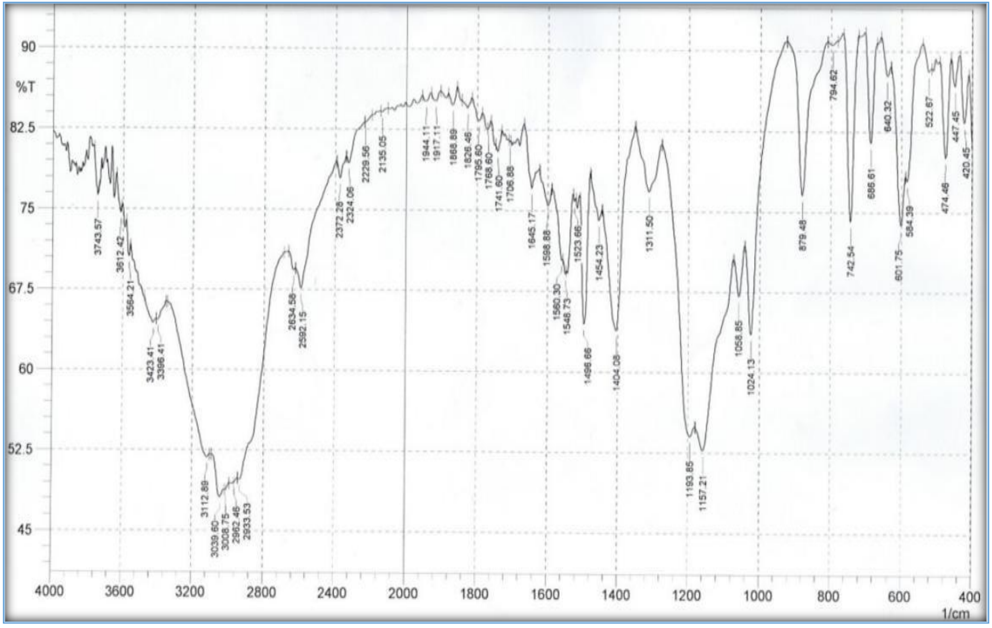


**Figure 10:** FT-IR spectrum of PANI /CoO : NiO nanocomposite.

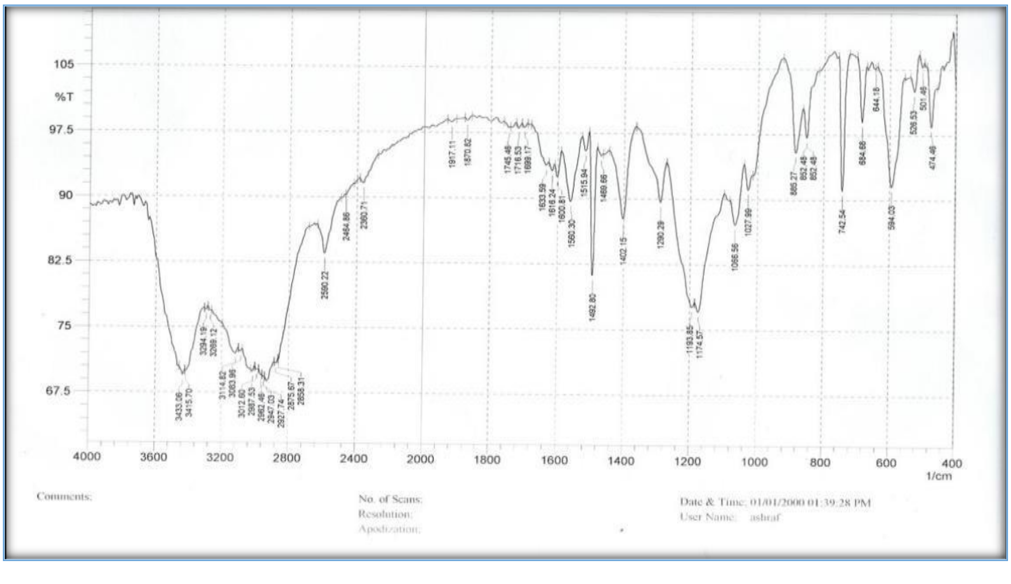


**Figure 11:** FT-IR spectrum of PANI /CuO: NiO nanocomposite .

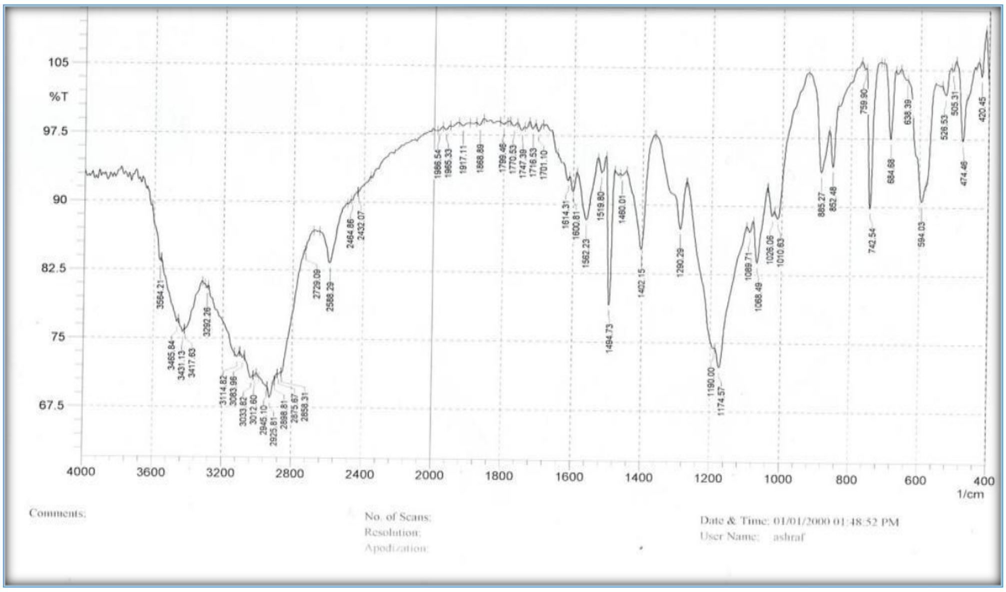


**Figure 12:** FT-IR spectrum of PANI /ZnO: NiO nanocomposite .

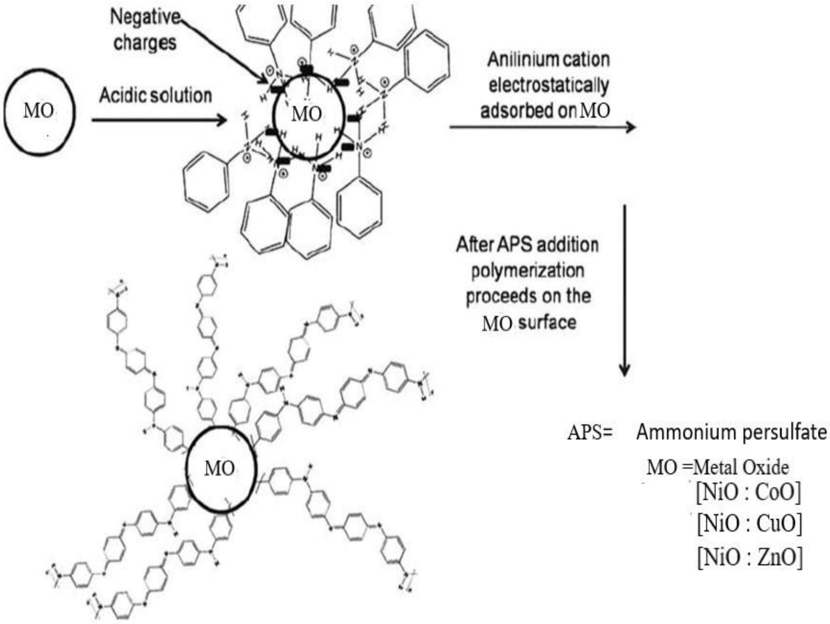


**Figure 13:** The suggested structure of PANI/MO nanocomposite.

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## 4. Corrosion protectiveness studies

Corrosion parameters were assessed based on the information presented in Table 2 and Figure 13. Corrosion current rate (icorr) in addition to the possibility of corrosion (Ecorr) solution were acquired by extrapolating the arrow's cathodic and anodic Tafel separated in 0.1M HCl (blank) and without inhibitor molecules. The anode (ba) and cathode (bc) Tafel slopes are also calculated from Figure 13. Table 2 shows the results of corrosion potential Ecorr (mV), corrosion current density icorr (A/ $cm^2$), cathodic and anodic Tafel slope (mV/Dec), and protection efficiency PE(%)[24].

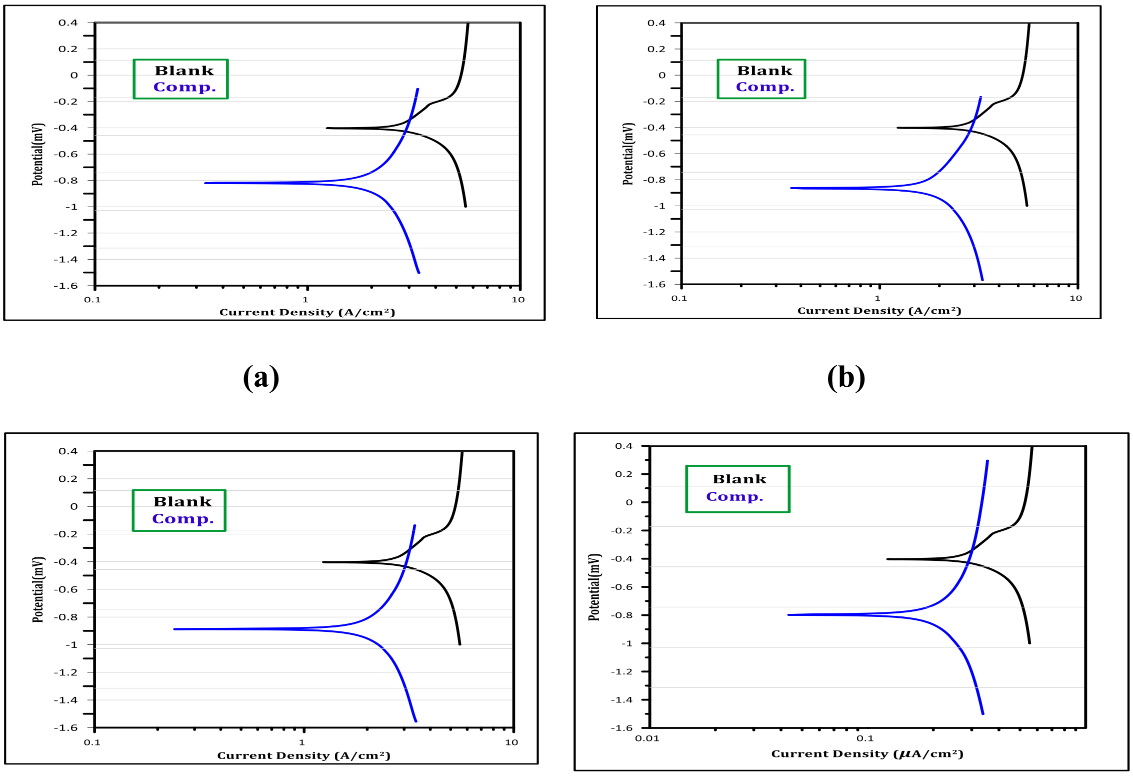
$$\%\mathrm{IE} = \frac{(\mathrm{icorr})\mathrm{o} - (\mathrm{icorr})}{(\mathrm{icorr})\mathrm{o}} * 100 \qquad (1)$$

where $(icorr)_i$ is the corrosion current density in the absence of inhibitors, and (icorr) is the corrosion current density in the presence of inhibitors[25].

**TABLE 2.** Corrosion parameters for Blank (HCl) solution and different nanocomposites.

| Comp. | E corr. | I corr. | I corr./ r | Resis. | Anodic β | Cathodic β | Corr. rate, | IE% |
|---|---|---|---|---|---|---|---|---|
| **Blank** | -0.405 | 376.3 | 7.526E-4 | 62.73 | 0.139 | 0.089 | 3.694 | - |
| **PANI/CoO:NiO** | -0.805 | 62.17 | 1.243E-4 | 1635 | 0.462 | 0.474 | 0.610 | 83 |
| **PANI/CuO:NiO** | -0.826 | 34.41 | 6.883E-5 | 1844 | 0.308 | 0.278 | 0.338 | 91 |
| **PANI/ZnO:NiO** | -0.789 | 49.36 | 9.873E-5 | 1880 | 0.473 | 0.390 | 0.485 | 87 |
| **PANI** | -0.783 | 79.37 | 1.587E-4 | 1366 | 0.495 | 0.504 | 0.779 | 78 |

The sample under test was placed inside a corrosion cell the surface area exposed to the solution was 16.55 cm$^2$. The full polarization measurement system configuration is displayed in Figure 13 [26] .



**(a)** **(b)**

**(c)** **(d)**

**Figure 13:** Polarization curves for corrosion of a ) PANI/CoO: NiO b) PANI/CuO: NiO c) PANI/ZnO:NiO (d) Poly aniline( PANI)

The inhibition efficiency increases to (83% - 91%) with an increase in corrosion rate. Depending on the concentration of the inhibitor, temperature, and phenyl group. This behavior might be explained by an increase in the number of molecules that have been adsorbed onto the metal surface. PANI/CuO: NiO stands out as the most effective corrosion inhibition compound due to its superconductivity, strong protective layer formation, and synergistic effects between CuO and NiOleads to enhance stability and corrosion resistance due to improved mechanical properties and chemical interactions. In contrast PANI/CoO: NiO suffers from instability and limited electron transfer capabilities, while PANI/ZnO: NiO (PAN/CoO: NiO) lacks the necessary conductivity to be effective in preventing corrosion.

## 5. Antioxidant activity

### *5,1-Diphenyl-2-picryl hydrazyl (DPPH)*

100 milliliters of methanol and four milligrams of 1,1-diphenyl-2-picryl hydrazyl (DPPH) were added to a test tube filled with aluminum foil. The solution was not exposed to light. Next, different concentrations (50, 25, 12.5, and 6.25 ppm) were generated by dissolving the tested chemical (1 mg) in 10 mL of methanol. This resulted in 100 ppm. After that, the concentrations were made by diluting this concentration.

### *5.2. Ascorbic acid.*

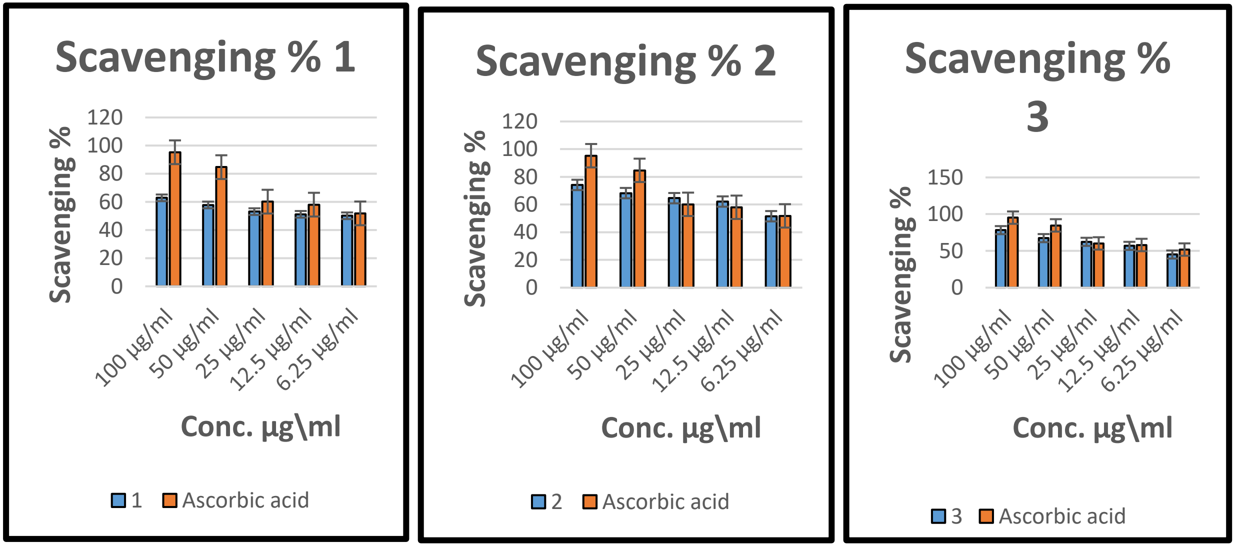
Concentrations that were comparable to those made in the earlier process were prepared. The antioxidant potency of the methanol extract was assessed, including its ability to scavenge free radicals, as well as its equivalence to vitamin C, using the stable free radical DPPH (2,2-diphenyl-1-picrylhydrazyl) assay. The DPPH solution (1 mL) was added to one milliliter of the standard or diluted solution (consisting of 100, 50, 25, 12.5, and 6.25 parts per million) in a test tube. All solvents were tested for absorbance at 517 nm using a spectrophotometer, and they were then incubated for just one hour at 37 °C. Using Equation 2, three measurements were taken. It can ascertain DPPH-scavenging potential.

$$I\% = \{(\text{Abs blank} - \text{Abs sample}) / \text{Abs blank}\} \times 100 \qquad (2)$$

When compared to the DPPH free, the recently created nanocomposite shown antioxidant activity [27].

**Table 3:** Antioxidant Activity according to Phongpaichit, 2007.

| compound NO. | 100 µg/ml | 50 µg/ml | 25 µg/ml | 12.5 µg/ml | 6.25 µg/ml | linear eq. | $R^2$ | IC$_{50}$ |
|---|---|---|---|---|---|---|---|---|
| **a)PANI/CoO:NiO** | 62.87 | 57.83 | 53.09 | 51.17 | 50.13 | y = -3.214x + 64.66 | $R^2$ = 0.9225 | 4.56 |
| **b)PANI/CuO:NiO** | 74.18 | 68.27 | 64.61 | 62.18 | 51.57 | y = -5.131x + 79.555 | $R^2$ = 0.9405 | 5.76 |
| **c)PANI/ZnO:NiO** | 78.26 | 67.39 | 62.35 | 56.83 | 45.09 | y = -7.69x + 85.054 | $R^2$ = 0.9755 | 4.56 |
| **Poly Aniline** | | | | | | | | |



**(a)** **(b)** **(c)**

**Figure 14.** Antioxidant activity of a) [PANI/CoO: NiO], b)[ PANI/CuO: NiO], c)[ PANI/ZnO: NiO] nanocomposites.

PANI/CoO: NiO and PANI/ZnO: NiO nanocomposites exhibit enhanced free radicals scavenging capabilities compared to PANI/CuO: NiO owing to several factors related to the properties of the respective metal oxides and their interactions. Polyaniline's resulting composite properties are influenced by the presence of Cobalt oxide (CoO) and zinc oxide (ZnO), which are known for their catalytic properties and ability to facilitate redox reactions. These metal oxides can enhance electron transfer processes in the compound, making them more effective at neutralizing free radicals compared to copper oxide (CuO), which may not exhibit the same level of catalytic efficiency. The superior performance of PANI/CoO: NiO and PANI/ZnO: NiO in free radical scavenging compared to PANI/CuO: NiO can be attributed to the unique properties of CoO and ZnO their interactions with polyaniline and the resulting improvements in catalytic activity.Surface area and stability. and electrochemical behavior.

## Conclusion

The nanocomposites [NiO: CoO/ PANI] and [NiO: CuO/ PANI and NiO: ZnO/ PANI] were synthesized. The analytical techniques employed, including FT-IR, SEM, EDX, and AFM, confirmed the successful synthesis of both composites at the nanoscale level. These measurements revealed that the average diameter of the nanoparticles falls within the nanometer range. The AFM measurements of a three-dimensional image indicate that the nanoparticles of oxides have formed a layer rough, and comparatively loose, as can be seen. The homogeneous coating exhibits a higher doping density Above the base material, which is polyaniline, where the morphology of the nanocomposites indicates, as mentioned, through this measurement homogeneity or symmetry between the particles of the nanocomposites. Also, the effectiveness of the [NiO: CoO/ PANI] and [NiO: CuO/ PANI],[ NiO: ZnO/ PANI] nanocomposites were tested on antioxidant activity and showed a good scavenging percentage. where the highest antioxidant activity was obtained for the compound, as it was found that the IC50 was (4.56)[NiO: CoO/ PANI], [ NiO: ZnO/ PANI]. The corrosion current density (icorr) decreased after adding the inhibitors into a blank solution. Tafel plots revealed that the corrosion potential (icorr) of the Chitosan (CS )coating in the presence of the inhibitors increased the shapes of the particles taken across a link with a sypherical shape.

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## Authors' Declaration

Conflicts of Interest:

- hereby attest that every Figure and Table in the manuscript is our own. Additionally, the essential permission for republication has been obtained for any Figures and images that are not our own, and this permission is linked to the manuscript.
- Ethical Clearance: The University of Baghdad's local ethical commission gave the project the go-ahead.

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## Authors' Contribution Statement

- This work was completed with the assistance of all authors in handling the test execution and sample preparation. Preparation of the text, conducted data analysis and assessed the material.

## References

**[1]** H. Sadik, W. R. Saleh, N. M. Hadi, and N. Kadhum, "Near IR Photoconductive Detector Based on f-MWCNTs / Polythiophene Nanocomposite ." *Iraqi Journal of Science*," vol. 58, no. 2, pp. 868–877, 2017, doi:10.24996.ijs.2017.58.2B.11

**[2]** G., Hryniewicz , B. M., Jasper, I., Silva, R. J, Klobukoski, V. Costa, F. S., and Vidotti. M "Recent trends of micro and nanostructured conducting polymers in health and environmental applications," *Journal of Electroanalytical Chemistry*, vol. 879, 2020, doi: 10.1016/j.jelechem.2020.114754.

**[3]** R. S. A. Al-Khafaji, "Synthesis of Blend Polymer (PVA / PANI)/Copper (1) Oxide Nanocomposite: Thermal Analysis and UV-Vis Spectra Specifications," *Iraqi Journal of Science*, vol. 62, no. 11, pp. 3888–3900, 2021, doi: 10.24996/ijs.2021.62.11.10.

**[4]** N. I. Mohamed, S. M. Hassan, and K. J. Khalil, "Preparation, characterization, and antimicrobial activity of polyaniline and $Fe_2O_3$/polyaniline composite nanoparticle," *Iraqi Journal of Physics*, vol. 20, no. 1, pp. 48–56, 2022.

**[5]** N. J. Abdullah, A. F. Essa, and S. M. Hasan, "Optical and structural properties of prepared polyaniline - Graphene (PANI/GN) nanocomposite," *Iraqi Journal of Science*, vol. 62, no. 1, pp. 138–146, 2021, doi: 10.24996/ijs.2021.62.1.13.

**[6]** R. Babaei-Sati, J. Basiri Parsa, and M. Vakili-Azghandi, "Electrodeposition of polypyrrole/ metal oxide nanocomposites for corrosion protection of mild steel—A comparative study," *Synthetic Metals*, vol. 247, no. November 2018, pp. 183–190, 2019, doi: 10.1016/j.synthmet.2018.12.009.

**[7]** P. Singh and S. K. Shukla, "Advances in polyaniline-based nanocomposites," *Journal of Materials Science*, vol. 55, no. 4, pp. 1331–1365, 2020, doi: 10.1007/s10853-019-04141-z.

**[8]** K. Sharma, M. S. Goyat, and P. Vishwakarma,"Synthesis of Polymer Nano-composite coatings as corrosion inhibitors: A quick review," *Institute of Physics (IOP) Conference Series: Materials Science and Engineering*., vol. 983, no. 1, 2020, doi: 10.1088/1757-899X/983/1/012016.

**[9]** A. Zadehnazari, "Metal oxide/polymer nanocomposites: A review on recent advances in fabrication and applications," *Polymer-Plastics Technology and Materials*, vol. 62, no. 5, pp. 655–700, 2023, doi: 10.1080/25740881.2022.2129387.

**[10]** G. Shao, R. Yu, N. Chen, M. Ye, and X. Y. Liu , "Stretchable Supercapacitors: From Materials and Structures to Devices," *Journal Small Methods*, vol. 5, no. 1, pp. 1–31, 2021, doi: 10.1002/smtd.202000853.

**[11]** P. Zarrintaj, R. Khalili, H. Vahabi, M. R. Saeb, M. R. Ganjali, and M. Mozafari "Mohammad Reza Saebf, Mohammad Reza Ganjalig,h, Masoud Mozafarii, *Polyaniline/metal oxides nanocomposites. In Fundamentals and emerging applications of polyanilineElsevier Inc*., 2019. doi: 10.1016/B978-0-12-817915-4.00008-7.

**[12]** Al-jubouri, Fatin A., and Basim I. Al-Abdaly,"Anti-oxidant and anti-microbial activities of [ZnO: CoO/eugenol] and [ZnO: $Fe_2O_3$/eugenol] nanocomposites." *Ibn AL-Haitham Journal For Pure and Applied Sciences''* vol. 37, no. 1, pp. 251–264, 2024. doi.org/10.30526/37.1.3233

**[13]** J. Chauhan, Varsha R mehto, "Preparation and Characterization of Polyaniline/ZnO Composite Sensor," *Journal of Nanomedicine Research*, vol. 5, no. 1, 2017, doi: 10.15406/jnmr.2017.05.00104.

**[14]** T. Das and B. Verma, "High performance ternary polyaniline-acetylene black-cobalt ferrite hybrid system for supercapacitor electrodes*," journal Synthetic Metals*, vol. 251, no. November 2018, pp. 65–74, 2019, doi: 10.1016/j.synthmet.2019.03.025.

**[15]** S. Vyas, A. Shukla, S. J. Shivhare, V. S. Bagal, and N. Upadhyay, "High-Performance Conducting Nanocomposites Polyaniline (PANI)-CuO with Enhanced Antimicrobial Activity for Biomedical Applications," *Engineered Science Materials and Manufacturing*, vol. 15, pp. 46–52, 2022, doi: 10.30919/esmm5f468.

**[16]** H. J. Imran, K. A. Aadim, and K. A. Hubeatir, "Two-step pulsed laser ablation for preparation NiO@ZnO core-shell nanostructure and evaluation of their antibacterial activity," *Advances in Natural Sciences: Nanoscience and Nanotechnology*, vol. 14, no. 4, p. 45003, 2023, doi: 10.1088/2043-6262/ad010c.

**[17]** S. Dey Sadhu, P. L. Meena, J. Kumar, J. Gupta, S. Choudhary, and A. Gupta, "Preparation and characterization of polyaniline- and polythiophene-based copolymer and its nanocomposite suitable for electro-optical devices," *Polymer Composites*, vol. 41, no. 11, pp. 4619–4630, 2020, doi: 10.1002/pc.25738.

**[18]** N. H. Khand, H. Shaikh, E. A. López-Maldonado, A. R. Solangi, and S. Tufail H. Sherazi, "Microwave-assisted synthesis of nickel-oxide/cobalt-oxide/reduced graphene oxide nanocomposite for highly sensitive electrochemical determination of epicatechin in food samples," *Results in Chemistry*, vol. 7, no. December 2023, 2024, doi: 10.1016/j.rechem.2024.101333.

**[19]** L. I. A. Ali, H. K. Ismail, H. F. Alesary, and H. Y. Aboul-Enein, "A nanocomposite based on polyaniline, nickel and manganese oxides for dye removal from aqueous solutions," *International*

*Journal of Environmental Science and Technology*, no. 0123456789, 2021, doi: 10.1007/s13762-020-02961-0.

**[20]** A. G. Ramu, M. L. A. Kumari, M. S. Elshikh, H. H. Alkhamis, A. F. Alrefaei, and D. Choi, "A facile and green synthesis of CuO/NiO nanoparticles and their removal activity of toxic nitro compounds in aqueous medium," *Chemosphere*, vol. 271, p. 129475, 2021, doi: 10.1016/j.'*chemosphere'*.2020.129475.

**[21]** D. Das, L. J. Borthakur, B. C. Nath, B. J. Saikia, K. J. Mohan, and S. K. Dolui, "Designing hierarchical NiO/PAni-MWCNT core-shell nanocomposites for high-performance supercapacitor electrodes," *Royal Society of Chemistry* Advances, vol. 6, no. 50, pp. 44878–44887, 2016, doi: 10.1039/c6ra01777k

**[22]** S. Vijayalakshmi, E. Kumar, M. Ganeshbabu, P. S. Venkatesh, and K. Rathnakumar, "Structural, electrical, and photocatalytic investigations of PANI/ZnO nanocomposites," *International Journal of Ionics – The Science and Technology of Ionic Motion*, vol. 27, no. 7, pp. 2967–2977, 2021, doi: 10.1007/s11581-021-04041-w.

**[23]** H. Saha Roy, M. Mominul Islam, M. A. Yousuf Mollah, and M. Abu Bin Hasan Susan, "Polyaniline-NiO Nanocomposites as Dielectric Materials," *Materials Today*: *Proceedings* , vol. 5, no. 7, pp. 15267–15276, 2018, doi: 10.1016/j.matpr.2018.05.005.

**[24]** N. A. Khudhair and A. M. A. Al-Sammarraie, "Study the Effect of SiO2 Nanoparticles as Additive on Corrosion Protection of Steel Rebar in Artificial Concrete Solution," *Journal of Engineering and Applied Sciences*, vol. 14, no. Special Issue 9, pp. 10616–10621, 2019, doi: 10.36478/JEASCI.2019.10616.10621.

**[25]** N. A. Khudhair and A. M. A. Al-Sammarraie, "Investigation on the effect of origin of carbon steel reinforcing bar on the corrosion behavior in artificial concrete solution," *Journal of Global Pharma Technology*, vol. 11, no. 5, pp. 76–81, 2019.

**[26]** S. Hamid, Aseel F. Abdullah, Mohammed M. Mohammed, and Fatma A. Khazaal, "Synthesis, characterization, Biological Activity, and corrosion inhibition study of two dyes from 4-aminophenol," *Wasit Journal for Pure Sciences*, vol. 2, no. 3, pp. 343–352, 2023, doi: 10.31185/wjps.215.

**[27]** M. A. Abdul-Zahra and N. M. Abbass, "Synthesis and Characterization of Nano-Composites of Polypropylene/Cr2O3 Nanoparticles Using Licorice Extract," *Iraqi Journal of Science*, vol. 65, no. 2, pp. 623–633, 2024, doi: 10.24996/ijs.2024.65.2.4.