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Article Fabrication of PANI/ NiCo₂O₄ /CeO₂ Nanocomposite: Preparation and Characterization

Ammar Adil Hussein^{1, 2, 3*}, Ali M. A. Abdul Amir AL-Mokaram^{1*}, Qabas Naji Rashid²

¹ Department of Chemistry, College of Science, Mustansiriyah University, Baghdad, 10052, Iraq

² Department of Chemistry, College of Pharmacy, Tikrit University, Tikrit, Iraq

³ Department of Chemistry, Baghdad College High School, Ministry of Education, Iraq

Correspondence: amaradel61@gmail.com, ali75@uomustansiriyah.edu.iq*, qabas.naji@tu.edu.iq

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Copyright: © 2024 by the authors. Submitted for open access publication under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses /by/4.0/) Abstract: This study introduces innovative research on synthesizing pure polyaniline (PANI) and a binary nanocomposite via in situ chemical oxidation polymerization. A modified interfacial polymerization technique was used to PANI/NiCo₂O₄/CeO₂ produce а greenish-black nanocomposite. Characterization methods such as Fourier transform infrared spectroscopy (FT-IR), UV-visible spectroscopy, and powder X-ray diffraction (XRD) were applied to the nanocomposite. The nanoscale structure of the composite was confirmed through particle size analysis in Field Emission Scanning Electron Microscopy (FE-SEM) images, which showed a uniform PANI coating on the NiCo2O4/CeO2 nanocomposite. Infrared spectroscopy identified all the characteristic vibrational bands of polyaniline and additional bands from the spinel metal oxides. XRD analysis verified the presence of NiCo2O4/CeO2, with broader peaks representing polyaniline and sharper peaks indicating the metal oxides.

Keywords: polyaniline, nickel cobalt oxide, cerium oxide, nanocomposite, X-ray diffraction (XRD)

Introduction

Nanotechnology has contributed significantly to developing new classes of materials with enhanced functionality and applications. The scientific definition of nanocomposite is multiphase materials where one or several phases have at least one dimension less than 100 nm. Since the introduction of nanotechnology, the focus has been directed towards designing and developing efficient immobilizing nanostructures to achieve desired outcomes [1].

As a result of its environmental stability and electrical and electrochemical characteristics, Polyaniline, known as PANI, has received enormous attention and has been the focus of many studies conducted on polymers to improve some of its properties. It is also up to be hybridized with some nanocomposites [2]. Metal oxide nanoparticles serve as a significant doping agent for modifying the characteristics of polymer chains [3]. The doping process that occurs through the oxidation of nitrogen atoms of polyaniline chain rings by cation and radical cations through the conducting polymer with inorganic nanoparticle composites in different combinations for two components has been under considerable consideration and interest [4]. Due to their interesting physical and electro-conductive properties, electronically conducting polymers, like polypyrrole, polyaniline, polythiophene etc. have been the focus of many researchers in the scientific community [5]. Conducting polymer composite can be fabricated from an aqueous solution of monomer and insulating polymer in different methods such as chemical or electrochemical polymerization by using suitable dopants and oxidants or supporting electrolytes [6, 7]. Scientific researchers have been recording and exploring the deposition of nanoscale PANI into numerous pores of NiCo₂O₄ nanorod arrays. The referred-to method modifies the integrity of the electrode by enabling the porous support to better accommodate the strain and stabilize PANI [8, 9, 10]. This research work contributes to the synthesis of pure polyaniline and binary nanocomposite by situ chemical oxidation polymerization and fabricated a novel PANI/NiCo₂O₄ nanocomposite /CeO₂.

Materials and Methods

Ammonium persulfate (APS), H₂SO₄, NiCl₂.6H₂O, CoCl₂.6H₂O and aniline were obtained from Merck and Sigma-Aldrich, India. CeO₂ nanoparticles were purchased from Hongwu new material from China. Samples have been investigated by JASCO FT-IR- 4200 analysis and PerkinElmer UV/Vis spectroscopy in the polymer research unit library of the Science College of Al-Mustansiriyah University. A Raman shift was recorded with a TEKSAN N1–541 instruments in Hungary. Nanocomposites are examined and placed on a glass substrate for X-ray diffraction analysis, a device that was used is XRD- 6000 Shimadzu and FE-SEM images have been investigated by TESCAN Mira3 in Iran.

2.1. Preparation PANI/NiCo₂O₄/CeO₂ nanocomposites

PANI/NiCo₂O₄/CeO₂ nanocomposite is the resultant of the oxidation process of aniline with ammonium persulfate (APS) in aqueous solution. Likewise, 1 ml of aniline is diluted with 55 ml of H₂SO₄ (0.5 M) and stirred well in a cooling bath [11, 12]. Nano powder of NiCo₂O₄ and CeO₂ (15wt %) with an equal amount of each is mixed with the previously prepared solution in an ultrasonic device. After being sonicated for 1 hour, the colour of the mixture starts to change based on the amount of NiCo₂O₄ and CeO₂ [13, 14]. Then the resultant mixture is taken out of the ultrasonic device and brought to a magmatic stirrer for 1 hour. 50 ml of the cold solution, the oxidizing agent of APS (0.1 M), is carefully added to the mixture with continuous stirring for more than 2 hours. After the stirring, the reaction is left for about 4 hours at 0 to 5°C. The product of the reaction obtained from filtration was gathered and washed thoroughly with acetone and distilled water till the filtrate lost its colour. When it gets colourless, the filtrate is left at 80°C for 24 hours to dry [15, 16].

Results and Discussion

The FTIR spectra of PANI and PANI/ CeO₂ are respectively demonstrated in the (1-A) and (1-B) figures. Near 3224 cm⁻¹, the N–H stretching vibration reaches its highest value of PANI. The peck of 1556 cm⁻¹ for C=N of quinone ring as well the peck of 1481 cm-1 for C=C of benzene ring in PANI. The distinguish peak 1285 cm⁻¹ of the stretching vibration of secondary amine C–N associated with ring and C=C of quinone ring observed at 943 - 1230 cm⁻¹ for the absorption peak. Almost near the peak at 1008 cm⁻¹ shows the stretching vibration peak of C–H for PAINI absorption [17-20]. The FTIR spectra of PANI/ CeO₂ between (490 to 3224) are presented in Figure (1-B). Around peaks 3224 cm⁻¹,1561 and 1481 cm⁻¹, were observed for N–H, C=N and C=C in PANI respectively. The peaks at 1295 cm⁻¹, and 927 - 1230 cm⁻¹ indicate C–N and for quinone of C=C individually. In PANI, the absorption value nearby 1024 cm⁻¹ for stretching vibration peak of C–H. In the main stretching vibration peak of CeO₂, the absorption reaches its highest value around 490, 540,693 cm⁻¹ [21-25].



Figure 1. A- The FTIR spectra of PANI and B- the FTIR spectra of PANI/ CeO2nanocomposite

Figure (2-A) and (2-B) respectively demonstrate the FT-IR spectrum of the PANI /NiCo₂O₄ and PANI/NiCo₂O₄ /CeO₂. In Figure (2-A), the acute peaks of the electro-active material at 540 and 640 cm⁻¹ could be a result of the Ni-O and Co-O stretching vibrations and formation of the spinel NiCo₂O₄ nanostructure of the FTIR spectra with PANI. The N–H stretching vibration value reaches its highest in PANI around 3224 cm⁻¹. The values of C=N for quinone, C=C for benzene ring and C–N confirmed around 1556 cm⁻¹, 1450 cm⁻¹ and 1285 respectively. Additionally, the C=C of the quinone ring is at 870 - 1230 cm⁻¹. The peak is at 1020 cm⁻¹, for PANI approval. The Ni-O and Co-O stretching vibrations and formation of the spinel NiCo₂O₄ nanostructure cause the acute peaks at 540 and 640 cm⁻¹ [26, 27]. On the other hand, figure (2-B) presents the N–H, C=N for quinone, and C=C for benzene ring at 3224 cm⁻¹, 1556 cm⁻¹ and 1481 cm⁻¹ respectively. The characteristic peak of 1285 cm⁻¹ is related to C–N associated with the ring, whereas the absorption peak of 945-1230 cm⁻¹ relates to the C=C of the quinone ring. The high-value peak of stretching vibration of C–H is the nearby 1050 cm⁻¹ in PANI. The acute peaks at 540 and 640 cm⁻¹ are linked to the Ni-O and Co-O stretching vibrations and formation of the spinel NiCo₂O₄



Figure 2. A- The FTIR spectra of PANI /NiCo2O4 and B -PANI/NiCo2O4/CeO2 nanocomposite

The study of UV-visible absorption spectra can contribute to the understanding of the electronic structure of the optical band gap of the material. The absorption that occurs close to the

ultraviolet region results from electronic transitions associated with the prepared sample. The PANI spectrum containing two distinguished peaks at 333 nm and 635 nm can be observed by UV–visible absorption spectra of pure PANI nanoparticles in Figure (3). The nanocomposite is found to be in an emeraldine state with absorbance peak values at 312-338 nm and 605-648 nm resulting from electron transition between benzenoid rings (π - π *) and charge move from benzenoid ring to quinoid ring respectively [28]. The absorption band from 600-700 nm is presumed to be caused by the agglomeration of cerium oxide nanoparticles. The wide band at 480-800 nm is probably the result of the integrated effect of cerium oxide nanoparticles and the transition from the highest occupied energy level to the lowest unoccupied energy level of the quinonoid ring.

The observation of the absorbance peaks of PANI and CeO₂ can be done at different values. For PANI, they can be observed at 356 nm and 635 nm, whereas for CeO₂ they can be observed at 232 nm. 271 nm peaks can be observed for PANI/CeO₂ with different CeO₂ contents for nanocomposites, where the absorbance increases as the ratio of addition to CeO₂ to the polymer increases [29, 30]. As the inorganic contents increase, the intensity of the absorbance increases. Generally speaking, the absorption edge for the nanocomposite is transformed to short wavelengths (high energy) about PANI, which indicates that strong quantum confinement has taken place as a result of hybridization between PANI and CeO₂ material [31].

UV-visible spectroscopy is conducted as a certain interfacial interaction of PANI in the composite has taken place that reveals the fact that the spectrum of PANI containing two prominent peaks at 330–350 nm and 610–680 nm which are associated with the π – π transition of the benzene ring and quinoid ring respectively. The same peak is observed for the composite PANI -NiCo₂O₄ with a small shift towards the longer wavelength (red shift). This could be a result of the interfacial interaction between the PANI and NiCo₂O₄. This could lead to a strong interaction between PANI and nickel cobaltite, which may have boosted the ion transportation path, to improve the capitative performance [32]. As a result, the absorbance spectra of the prepared samples within a wavelength (210-1000 nm) for PANI/NiCo₂O₄/CeO₂ contained peaks referring to the presence of polyaniline, NiCo₂O₄ and CeO₂. This indicates the preparation of prepared PANI/NiCo₂O₄ /CeO₂.



Figure 3. UV-Visible of PANI, PANI/ CeO2, PANI /NiCo2O4 and PANI/NiCo2O4 /CeO2 nanocomposite

To evaluate the structure and crystallinity of composite materials, XRD analysis is performed. Figure 4 illustrates the XRD patterns of the PANI, PANI/NiCo₂O₄, PANI/CeO₂, and PANI/NiCo₂O₄/CeO₂. Pristine PANI shows a highly amorphous broad hump from 20 to 35° without demonstrating any sharp crystalline peaks. This shows the highly amorphous nature of PANI. Similar XRD findings are made public elsewhere [33].

A small peak of 25.6° which is associated with PANI is shown by the XRD pattern of PANI/NiCo₂O₄ nanocomposite although the XRD peaks of NiCo₂O₄ do not show in the XRD pattern of PANI/NiCo₂O₄ nanocomposite which is resulted by the deep integration of NiCo₂O₄ nanoparticles in PANI matrix. NiCo₂O₄ nanoparticles behaved as a template for the growth of PANI during the synthesis. However, sharp and well-defined peaks of CeO₂ in the PANI/CeO₂ nanocomposite. The CeO₂ peaks showed up at 20 values of 28.6°, 32.9°, 47.4°, 56.3°, 59°, 69.6°, 76.8°, and 79.2°, which are linked to the (111), (200), (220), (311), (222), (400), (311), and (420) crystallographic planes of a cubic fluorite structure of CeO₂ [34, 35].

The peak location of CeO₂ remained of NiCo₂O₄ in the PANI/NiCo₂O₄/CeO₂, though the intensity of the CeO₂ peaks were significantly reduced. Nevertheless, a broader hump related to PANI is evident, which confirms the successful integration of CeO₂ and NiCo₂O₄ nanoparticle in the PANI matrix without showing any impurity phase.



Figure 4. X-ray diffraction patterns of the as-synthesized PANI, PANI/ CeO₂, PANI /NiCo₂O₄ and PANI/NiCo₂O₄ /CeO₂ nanocomposites

FE-SEM is used to investigate the surface morphology of the prepared composite. FE-SEM images of PANI, PANI/ CeO₂, PANI /NiCo₂O₄ and PANI/NiCo₂O₄/CeO₂ nanocomposite are illustrated in Figure 5. Figure (5-A) contains the FE-SEM image of pure PANI demonstrating the densely packed tubular shape of PANI. It is well known that the morphology of PANI is tubular while prepared [36].

The PANI morphology did not change upon incorporation of CeO₂ nanoparticles Figure (5-B). On the surface of PANI, a few particles were observed which confirms the fact that NiCo₂O₄ nanoparticles were embedded in the PANI tubes Figure (5-C). In the FE-SEM image of PANI/NiCo₂O₄ /CeO₂ nanocomposite (Figure 5D), a tubular-shaped PANI with a uniform decoration of CeO₂ and NiCo₂O₄ nanoparticles is noticed [37-40]. A more loosely packed PANI tube with a uniform distribution of CeO₂ and NiCo₂O₄ nanoparticles can be observed in PANI/NiCo₂O₄ /CeO₂ nanocomposite. Figure (5-D) illustrates the production of nanostructures with a minimum size of 18.40 nm.



Figure 5. The FE-SEM morphology of the as-synthesized of (A) PANI, (B) PANI/ CeO₂, (C) PANI /NiCo₂O₄ and (D) PANI/NiCo₂O₄ /CeO₂

Conclusion

To summarize, the innovative PANI/NiCo₂O₄/CeO₂ nanocomposite is highly effective, boasting a well-organized, metal-free structure. It has been thoroughly characterized by various studies. The integration of CeO₂ and NiCo₂O₄ nanoparticles into the PANI matrix is demonstrated by XRD patterns. The observed blue shift in the UV absorption threshold is due to size quantization effects. FE-SEM has been instrumental in analyzing the morphology and structure of the particles. FT-IR spectra have determined the functional groups in the samples.

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