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تحضير وتشخيص مركب نانوي جديد عن طريق بلمرة الأنيلين

عمار عادل حسين
قسم الكيمياء/ كلية العلوم/ الجامعة المستنصرية / بغداد / العراق
علي محمد عبد الامير المقرم
قسم الكيمياء/كلية الصيدلة/جامعة تكريت/ تكريت/ العراق
قبس ناجي رشيد
قسم الكيمياء/ ثانوية كلية بغداد/ وزارة التربية/ العراق
amaradel61@gmail.com*, ali75@uomustansiriyah.edu.iq*, qabas.naji@tu.edu.iq

الملخص

يقدم هذا العمل طريقة جديدة لتحضير البولي أنيلين النانوي (PANI) مع مركبين نانويين بطريقة بلمرة الأكسدة الكيميائية. تم إنشاء مركب نانوي $PANI/NiCo_2O_4/CeO_2$ باللون الأسود المخضر باستخدام البلمرة بطريقة معدلة. تم وتشخيص المركب النانوي باستخدام تقنيات مثل حيود الأشعة السينية (XRD) ، مطيافية رامان و مطيافية الأشعة فوق البنفسجية المرئية. وضح حجم الجسيمات النانوية بصور المجهر الإلكتروني الماسح بالانبعاث الميداني(FE-SEM) و عن وجود طبقة PANI متناسقة على المركب النانوي$NiCo_2O_4/CeO_2$ ، مما يؤكد بنية المركب النانوية. تم الكشف عن جميع النطاقات الاهتزازية المميزة للبولي أنيلين بالإضافة إلى النطاقات الإضافية من أكاسيد المعادن بواسطة مطيافية رمان. تم تأكيد وجود $NiCo_2O_4/CeO_2$ من خلال تحليلXRD ، حيث تم الإشارة إلى أكاسيد المعادن من خلال قمم أكثر حدة والبولي أنيلين من خلال قمم أكبر.

الكلمات المفتاحية: بولي أنيلين، أوكسيد النيكل والكوبالت، أوكسيد السيريوم، مركب نانوي، حيود الأشعة السينية(XRD)

Preparation and Characterization a new Nanocomposite by polymerization of aniline

Ammar Adil Hussein
Department of Chemistry, College of Science, Mustansiriyah University, Baghdad, 10052, Iraq
Ali M.A. Abdul Amir AL-Mokaram
Department of Chemistry, College of Pharmacy,, Tikrit University, Tikrit-Iraq
Qabas Naji Rashid
Department of Chemistry, Baghdad College High School, Ministry of Education. Iraq
amaradel61@gmail.com, *ali75@uomustansiriyah.edu.iq*, *qabas.naji@tu.edu.iq*

Abstract

This work presents novel in situ chemical oxidation polymerization research on the synthesis of pure polyaniline (PANI) and a binary nanocomposite. A greenish-black $PANI/NiCo_2O_4/CeO_2$ nanocomposite was created using a modified interfacial polymerization process. The nanocomposite was characterized using techniques such powder X-ray diffraction (XRD), Raman spectroscopy, UV-visible spectroscopy. Particle size examination of Field Emission Scanning Electron Microscopy (FE-SEM) pictures revealed a consistent PANI coating on the

$NiCo_2O_4/CeO_2$ nanocomposite, confirming the composite's nanoscale structure. All of the distinctive vibrational bands of polyaniline as well as extra bands from the spinel metal oxides were detected by Raman spectroscopy. The existence of $NiCo_2O_4/CeO_2$ was confirmed by XRD analysis, where the metal oxides were indicated by sharper peaks and polyaniline by larger peaks.

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

1. Introduction

New material classes with improved functioning and applications have been developed in large part because to nanotechnology. According to scientific definitions, a nanocomposite is a multiphase material with at least one dimension less than 100 nm in one or more phases. Since the advent of nanotechnology, attention has been focused on creating effective immobilizing nanostructures in order to accomplish desired results [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 serves 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 though 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 a suitable dopants and oxidant or supporting electrolyte[6, 7]. Scientific researchers have been recording and exploring the deposition of nanoscale PANI into numerous pores of $NiCo_2O_4$ 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/$NiCo_2O_4$ nanocomposite /CeO_2.

2. Materials and Methods

Aniline, $NiCl_2.6H_2O$, $CoCl_2.6H_2O$, H_2SO_4, and ammonium persulfate (APS) were acquired from Merck and Sigma-Aldrich in India. We bought CeO_2 nanoparticles from Hongwu, a new Chinese supplier. PerkinElmer UV/Vis spectroscopy and JASCO FT-IR-4200 analysis have been used to examine the samples at Al-Mustansiriyah University's polymer research unit library of scientific collage. In Hungary, a Raman shift was captured using a TEKSAN N1–541 device. For X-ray diffraction examination, nanocomposites are inspected and put on a glass substrate. The XRD-6000 Shimadzu was the instrument utilized, and TESCAN Mira3 in Iran has been investigating the FE-SEM pictures.

2.1 Preparation PANI/$NiCo_2O_4$

PANI/$NiCo_2O_4$ nanocomposite is the product of the oxidation of aniline with ammonium persulfate (APS) in aqueous solution. The process of preparing PANI/$NiCo_2O_4$ goes through the following stages. Add 1 ml of aniline is diluted with 55 ml of H_2SO_4 (0.5 M) and stirred well in a cooling bath[11, 12]. A measured amount of $NiCo_2O_4$ (15 wt%) nano powder is dispersed in the prepared solution with the help of ultrasonic machine. After one hour the solution color starts to change gradually. Afterwards, the solution is moved to a magnetic stirrer for about 1 hour. 50 ml of the cold solution, the oxidizing agent of APS, 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 filtrate losses its color. When it gets colorless, the filtrate is left at 80°C for 24 hours to dry[13, 14].

2.2 Preparation PANI/CeO_2

Like the previous procedures, PANI/CeO_2 nanocomposite is also prepared by of the oxidation of aniline with ammonium persulfate (APS) in aqueous solution but with the addition of CeO_2 nanoparticle. Similarly, 1 ml of aniline is diluted with 55 ml of H_2SO_4 (0.5 M) and stirred well in a cooling bath. A specific amount of CeO_2 (15

wt%) nano powder is distributed in the solution and well sonicated for 1 hour till the color of the mixture changes from white to gray to cyan color. Then it is transferred to magnetic stirrer for about 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.

Afterwards, the product of the reaction obtained from filtration was gathered and washed thoroughly with acetone and distilled water till the filtrate becomes colorless. After that, the filtrate was left at 80°C for 24 hours to dry[15, 16] [17].

2.3 Preparation PANI/$NiCo_2O_4$/CeO_2 nanocomposites

PANI/$NiCo_2O_4$/CeO_2 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 H_2SO_4 (0.5 M) and stirred well in a cooling bath. Nano powder of $NiCo_2O_4$ and CeO_2 (15wt %) with equal amount of each is mixed with the previously prepared solution in an ultrasonic device. After being sonicated for 1 hour, the color of the mixture starts to change based on the amount of $NiCo_2O_4$ and CeO_2. 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 filtrate losses its color. When it gets colorless, the filtrate is left at 80°C for 24 hours to dry.

3. Result and discussion

The study of UV-Visible absorption spectral can contribute to the understanding of electronic structure of the optical band gap of the material[18]. The absorption that occurs close to the ultraviolet region is resulted by electronic transitions associated within 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 Figure (1) [19, 20]. The nanocomposite is found to be in 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[21]. 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 resulted as the integrate effect of cerium oxide nanoparticle and transition from highest occupied energy level to lowest unoccupied energy level of quinonoid ring.

The observation of the absorbance peaks of PANI and CeO_2 can be done at different values. For PANI, they can be observed at 356 nm and 635 nm, whereas for CeO_2 they can be observed at 232 nm. 271 nm peaks can be observed for PANI/CeO_2 with different CeO_2 contents for nanocomposites, where the absorbance increases as the ratio of addition to CeO_2 to the polymer increases[22, 23]. As the inorganic contents increase, the intensity on the absorbance increases. Generally speaking, the absorption edge for the nanocomposite is transformed to short wavelengths (high energy) in relation to PANI, which indicated that a strong quantum confinement has taken place as a result of hybridization between PANI and CeO_2 material[24].

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 -$NiCo_2O_4$ with a small shift towards the longer wavelength (red shift). This could be resulted due to the interfacial interaction between the PANI and $NiCo_2O_4$. This could hit to a strong interaction between PANI and nickel cobaltite, which may have boosted the ion transportation path, to improve the capitative performance[25]. As a result, the absorbance spectra of the prepared samples within wavelength (210-1000 nm) for PANI/$NiCo_2O_4$/CeO_2 contained peaks referring to the presence of polyaniline, $NiCo_2O_4$ and CeO_2. This indicates the preparation of prepared PANI/$NiCo_2O_4$ /CeO_2.

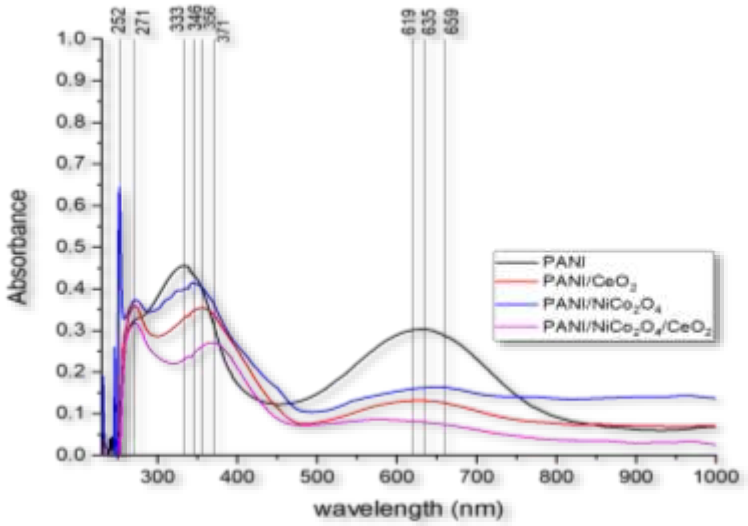

Fig (1) UV–Visible of PANI, PANI/ CeO_2, PANI /$NiCo_2O_4$ and PANI/$NiCo_2O_4$ /CeO_2 nanocomposite

The spectrum depicts Raman spectroscopy analytical technique conducted on the synthesized PANI for the purpose of characterizing the structure and composition of the samples as illustrated in Figure (2-A). Two significant distinct peaks at 1350 and 1600 cm^{-1} are observed in the Raman spectra of PANI, which relate to the band associated to sp^2 carbon atoms chains spread in the complete polymer respectively.

Though still localized polaronic structure, a shoulder noticed at 1350 cm^{-1} corelates to the $C{\sim}N^{+\bullet}$ stretching vibrations of the developing. The Raman spectra band at 1600 cm^{-1} is designated to the C=C stretching vibration in the ring. Therefore, these top values of 1350 and 1600 cm^{-1} corresponds to C–N vibration of delocalized polaronic structure and the C–C stretching of the ring in the order given occupying the very position yet with grater intensities. This could be concluded as a deprotonating of ammine nitrogen atoms in PANI[26].

The spectrum illustrates Raman spectroscopy analytical technique conducted on the synthesized PANI/CeO_2 is demonstrated in Figure(2-B). Two significant distinct peaks at 1350 and 1600 cm^{-1} are observed in the Raman spectra of PANI. The symmetrical stretching mode of the CeO_2 vibrational unit is in correlation with the peak value of 436 cm^{-1} in the spectra of PANI/CeO_2. The CeO_2 nanoparticles are conclusively fixed on PANI based on the peak shown in the PANI/CeO_2 nanocomposite with a blue shift to 460 cm^{-1}. The blue shift is correlated to the charge transfer between CeO_2 and PANI[27].

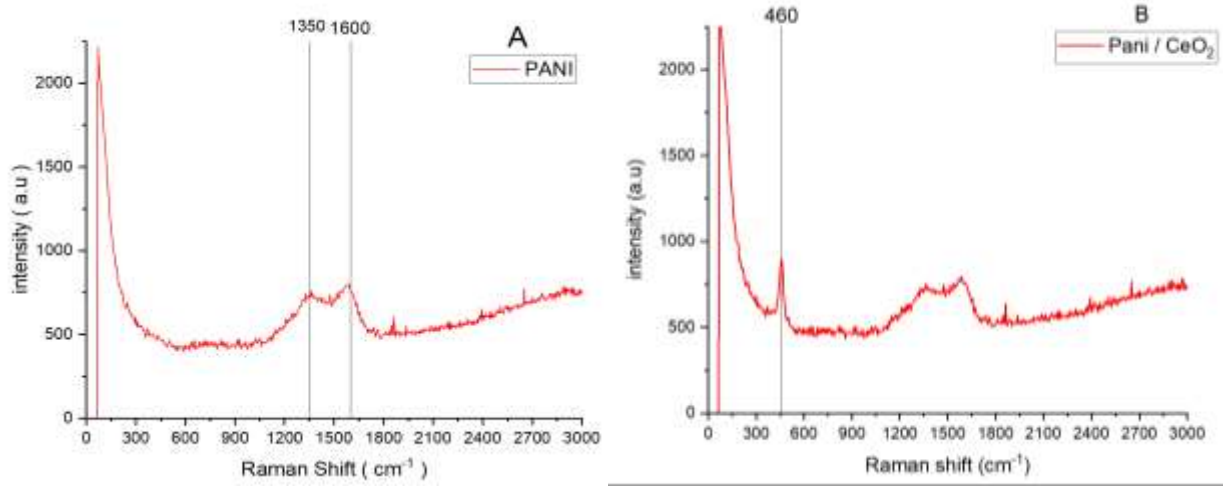

Fig (2) A- The Raman spectra of PANI and B- PANI/ CeO_2

Figure (3-A) demonstrates the composition and structural features of the PANI/$NiCo_2O_4$ nanostructures characterized by Raman spectroscopy. Three peaks found at 417, 450,530 and 640 cm^{-1} connected to F_2g, Eg, F_2g and A_1g Raman active modes of $NiCo_2O_4$ nanoparticles, in the given order, which are primarily associated to Ni–O and Co–O vibrations of the $NiCo_2O_4$ spinel oxide. This phonon is a result

of the lattice vibrations of the spinel structure. Ni atoms located at octahedral sites and Co atoms are distributed over both octahedral and tetrahedral sites in the spinel structure of $NiCo_2O$[28].

The spectrum for the Raman spectroscopy analysis method applied on the synthesized PANI/$NiCo_2O_4$/CeO_2 is presented in Figure (3-B). At 1350 and 1600 cm^{-1} for PANI, two prominent peaks can be distinguished. The spectra of CeO_2 spectra peak at 434 cm^{-1} is linked to the symmetric stretching mode of the CeO_2 vibrational unit, which overlaps with $NiCo_2O_4$ peak 415 cm^{-1}. Peaks (415, 517, 575, and 640 cm^{-1}) reveals clearly the presence of $NiCo_2O_4$. These peaks involving the nanoparticles of CeO_2 and $NiCo_2O_4$ confirms the successful attachment of these nanoparticles to PANI.

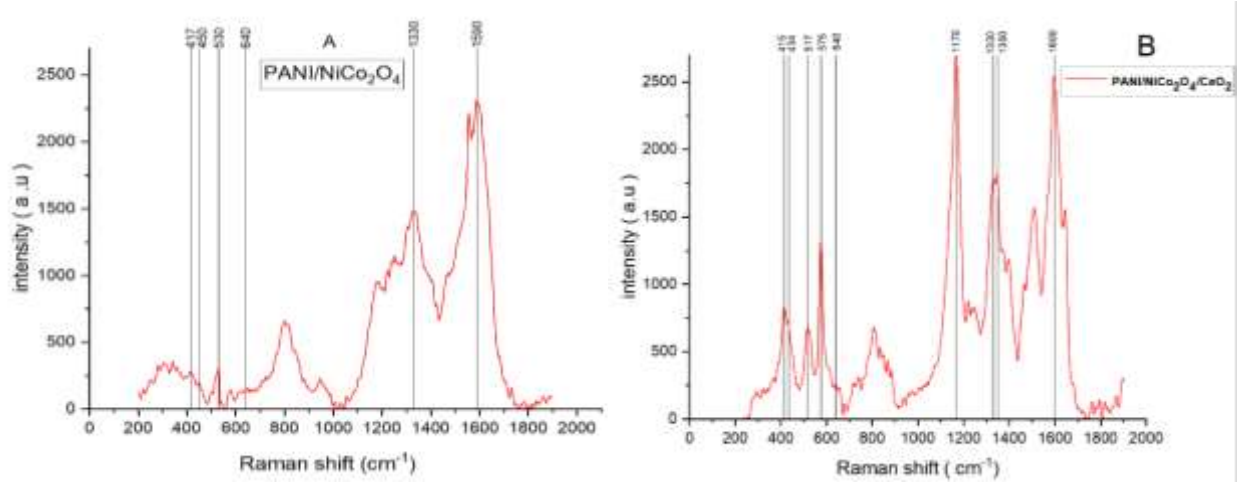

Fig (3) A- The Raman spectra of PANI /$NiCo_2O_4$ and B -PANI/$NiCo_2O_4$ /CeO_2 nanocomposite

In order to evaluate the structure and crystallinity of composite materials, XRD analysis is performed. Figure 4 illustrates the XRD patterns of the PANI, PANI/$NiCo_2O_4$, PANI/CeO_2, and PANI/$NiCo_2O_4$/CeO_2. 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[29]. A small peak of 25.6° which is associated with PANI is shown by the XRD pattern of PANI/$NiCo_2O_4$ nanocomposite although the XRD peaks of $NiCo_2O_4$ do not show in the XRD pattern of PANI/$NiCo_2O_4$ nanocomposite which is resulted by the deep integration of $NiCo_2O_4$ nanoparticles in PANI matrix. As a matter of fact, $NiCo_2O_4$ nanoparticles behaved as a template for the growth of PANI during the synthesis. However, sharp and well-defined peaks of CeO_2 in the PANI/CeO_2 nanocomposite. The CeO_2 peaks showed up at 2θ 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 CeO_2[30, 31].

The peak location of CeO_2 remained of $NiCo_2O_4$ in the PANI/$NiCo_2O_4$/CeO_2, though the intensity of the CeO_2 peaks were significantly reduced. Nevertheless, a broader hump related to PANI is evident, which confirms the successful integration of CeO_2 and $NiCo_2O_4$ nanoparticle in PANI matrix without showing any impurity phase.

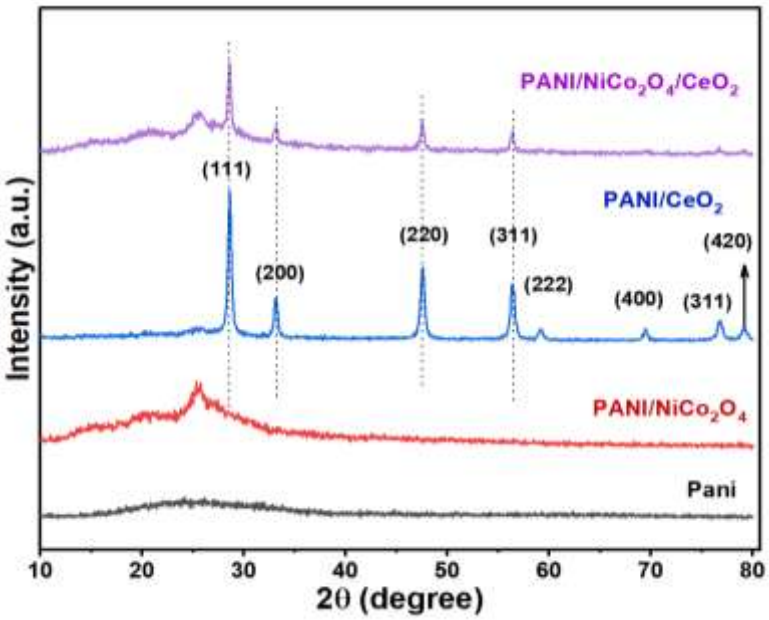

Fig (4) X-ray diffraction patterns of the as-synthesized of PANI, PANI/ CeO_2, PANI /$NiCo_2O_4$ and PANI/$NiCo_2O_4$ /CeO_2 nanocomposites

FE-SEM is used to investigate the surface morphology of the prepared composite. FE-SEM images of PANI, PANI/ CeO_2, PANI /$NiCo_2O_4$ and PANI/$NiCo_2O_4$/CeO_2 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. The PANI morphology did not change upon incorporation of CeO_2 nanoparticles Figure (5-B). On the surface of PANI a few particles were observed which confirms the fact that $NiCo_2O_4$ nanoparticles were embedded in the PANI tubes Figure (5-C). In the FE-SEM image of PANI/$NiCo_2O_4$ /CeO_2 nanocomposite (Figure 5D), a tubular-shaped PANI with a uniform decoration of CeO_2 and $NiCo_2O_4$ nanoparticles is noticed. A more loosely packed PANI tube with a uniform distribution of CeO_2 and $NiCo_2O_4$ nanoparticles can be observed in PANI/$NiCo_2O_4$ /CeO_2

nanocomposite. Figure (5-D) illustrates the production of nanostructures with a minimum size of 18.40 nm.

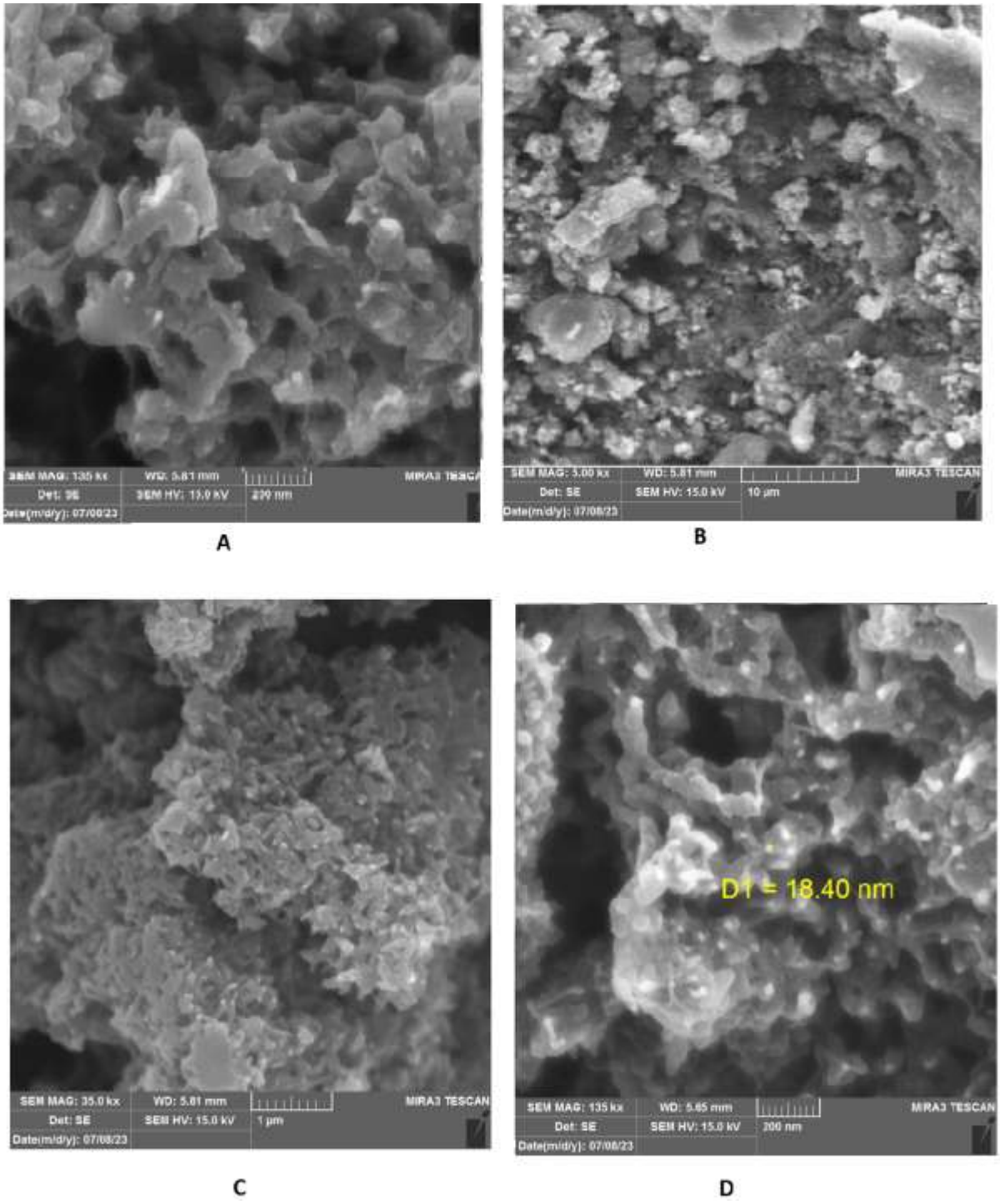

Fig (5) The FE-SEM morphology of the as-synthesized of (A) PANI, (B) PANI/ CeO_2, (C) PANI /$NiCo_2O_4$ and (D) PANI/$NiCo_2O_4$ /CeO_2

4. Conclusion

In summary, the novel PANI/$NiCo_2O_4$/CeO_2 nanocomposite is very efficient, has a well-organized, metal-free structure, and has been well described by a number of studies. XRD patterns show that CeO_2 and $NiCo_2O_4$ nanoparticles have been integrated into the PANI matrix, and size quantization effects are responsible for the blue shift in the UV absorption threshold that has been observed. FE-SEM has been helpful in examining the particles' morphology and structure, and Raman spectroscopy has mainly confirmed the structural features of the PANI/$NiCo_2O_4$/CeO_2 nanocomposite.

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