Iraqi Journal of Physics, 2022 *Vol.20, No. 3, PP.29-39*

DOI: 10.30723/ijp.v20i3.1018 *P-ISSN: 2070-4003*
E-ISSN: 2664-5548

Structural Properties of Prepared PANI/TiO_2 Nanocomposite by Chemical Polymerization

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Abstract

A progression of Polyaniline (PANI) and Titanium dioxide (TiO_2) nanoparticles (NPs) were prepared by an in-situ polymerization strategy within the sight of TiO_2 NPs. The subsequent nanocomposites were analyzed using Fourier-transform infrared spectra (FTIR), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy Dispersive X-Ray Analysis (EDX) taken for the prepared samples. PANI/TiO_2 nanocomposites were prepared by various compound materials (with H_2SO_4 0.3 M and without it, to compare the outcome of it) by the compound oxidation technique using ammonium persulfate (APS) as oxidant within the sight of ultrafine grade powder of TiO_2 cooled in an ice bath. Nanocomposites were prepared by the addition of TiO_2 with two weight ratios (0.3 and 0.5 wt. %) during the polymerization of PANI. The outcomes showed good collaboration between PANI and TiO_2. FTIR spectral shows a shift to higher wave numbers in the peaks of PANI/TiO_2 nanocomposites, due to the Coulomb force that resulted from the interaction between the TiO_2 nanoparticles with PANI. SEM results show that the TiO_2 nanoparticles enwrap the polyaniline and agglomeration of uneven distribution of TiO_2 particles can be seen in the PANI matrix. The intensity of the peak in the EDX analyses was found to appear by adding the nanoparticles. XRD pattern of PANI polymerization and PANITNCs shows that the TiO_2 NPs and PANI affected the crystallization performance of nanocomposites, it was identified that the TiO_2 NPs form a relatively irregular distribution in the PANI chain.

Article Info.

Keywords:
TiO_2 nanocomposite, XRD, Polyaniline (PANI), In situ Polymerization, FTIR.

Article history:
Received: Jun. 27, 2022
Accepted: Aug. 17, 2022
Published: Sep. 01,2022

1. Introduction

1.1. Polyaniline (PANI)

Polyaniline (PANI) is a renowned conductive polymer, and it got gigantic consideration from specialists in the areas of nanotechnology to improve sensors and optoelectronic devices. PANI is effectively doped with various acids because of its simple blend and amazing ecological stability [1]. Researchers have focused on creating conductive polymers that are accomplished in the fields of optics, devices, energy, etc. [2, 3]. PANI was known first as dark aniline and then came in various structures relying upon its oxidation levels. Moreover, PANI is known for its simplicity [4], natural steadiness, and capacity to be doped by protonic acids [5]. PANI is associating 1, 4-coupling of aniline monomer parts. PANI could exist in various oxidation states, and it may be described with the FTIR benzenoid to the quinonoid proportions [6]. Formed conductive polymers mainly include polyaniline (PANI), polythiophene (PTH), polypyrrole (PPY), and their items [7].

PANI has various potential applications including electromagnetic impedance protecting, photothermal treatment, battery-powered, photovoltaic cell, gas partition layer, substance sensor, anticorrosion covering, microwave ingestion, etc. [8, 9].

Moreover, conductive polymers are used, for example, leading fillers in protecting polymer substrates to gain directing polymer compounds [10-12]. These mixtures have possible applications in electromagnetic interference shields, electronic equipment, and display device electrodes [13]. PANI is a kind of material showing reflecting detection at room temperature and useful activity, this way is an appealing possibility for the improvement of various gas sensors. PANI properties, including its detecting qualities, were viewed as changed by dopants or by connection point collaboration in the composite [14].

1.2. Titanium Dioxide (TiO_2) Nanoparticles

The titanium dioxide (TiO_2) nanoparticles are a typical oxide metal, n-type of semiconductor that displays fascinating photocatalytic and some electronic properties that were able to open many promising applications in photograph voltaic, photocatalysis, photograph electrochromic, and sensors. TiO_2, particularly in the rutile structure, can oxidize natural materials straightforwardly because of its solid oxidative action. The presence of TiO_2 nanoparticles in the PANI lattice may cause a few changes and achieve some new fascinating properties [15]. Because of such presumptions, some research was done on the impact of the TiO_2 nanoparticles on gas detecting qualities of the PANI/TiO_2 blended by the in-situ method of synthetic polymerization [16].

2. Experimental

2.1. Materials and reagents

Aniline (ANI) $C_6H5.NH_2$ (AR, Beijing Chemical Reagent Co., China) was distilled and stored to use. Hydrophilic Rutile TiO_2 NPs Powder (Spec: 30-50 nm, 99.9%), ammonium persulfate $(NH_4)_2S_2O_8$ (APS, AR), and ethanol (AR) were used as oxidants as shown in Table 1.

Table 1: The name of the samples and percentage of the Nanoparticles.

Sample name	**Nano present (TiO_2) %**	**Chemical materials**
H	-	H_2SO_4/Aniline/Ammonium persulfate
H1	0.3	H_2SO_4/Aniline/Ammonium persulfate
H2	0.5	H_2SO_4/Aniline/Ammonium persulfate
W	-	Distilled water/Aniline/Ammonium persulfate
W1	0.3	Distilled water/Aniline/Ammonium persulfate
W2	0.5	Distilled water/Aniline/Ammonium persulfate

2.2. Preparation

Aniline 99.0% (ANI), TiO_2 NPs rutile, and ammonium persulphate (APS, Kanto Chemical Co. Inc.) were used as starting materials for synthesizing the PANI/ TiO_2 nanocomposites. Every one of the materials was used as received. The system was completed with various compound materials (with H_2SO_4 0.3 M and without it) as follows: a solution of 10 ml of ethanol and different proportion of TiO_2 (as displayed

in Table 1) was on the stirrer for 30 min to make a solution of TiO_2. The TiO_2 solution was then treated with a solution of 0.5 ml aniline (0.1M) and 0.3 M H_2SO_4 (total solution 50ml). Then, a solution of 5 g ammonium persulphate with 50 ml distilled water was added dropwise to the ANI and TiO_2 solution blend. The two solutions were kept for 1 hr at 0 ^oC to polymerize. The solution was left to settle down until the following day. The precipitated PANI was gathered in filter papers. From that point onward, PANI powder was kept in a desiccator and left to dry in the air for about 3 days at room temperature to keep away from any impact of moisture absorption. The nanocomposite synthetic construction was investigated by FTIR, X-Ray diffraction analysis, Scanning Electron Microscopy (SEM), and Energy Dispersive X-Ray Analysis (EDX) taken for the prepared samples.

3. Result and discussion

3.1. FTIR studies

The most important peaks observed in the FTIR spectra of raw materials shown in Fig. 1 and the FTIR spectra of the PANI/TiO_2 nanocomposites (PANITNCs) were it contains (PANI 0.1 M water-based only) and (PANI 0.1 M with H_2SO_4 0.3M) are shown in Fig. 2. Pure aniline characteristic peaks (in Fig.1) were observed at 965 cm^{-1} C=C bending, 1141 cm^{-1} C=N imines bending, the stretching mode 1295 cm^{-1} C-N for benzenoid ring N=Q=N 1562 cm^{-1} C=C for the quinoid ring, 1491 cm^{-1} C=C benzenoid ring stretching N=B=N [17].

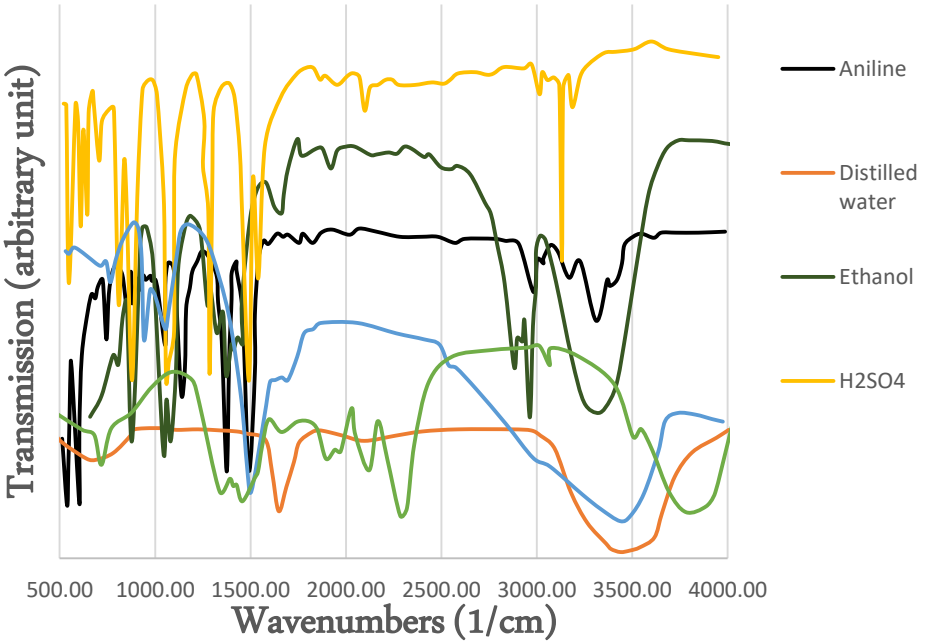

Figure 1: FTIR spectra of pure components.

The bands at 760 cm^{-1}, 680 cm^{-1}, 560 cm^{-1}, 500 cm^{-1} and 468 cm^{-1} are related to (Ti-O-Ti) vibrations [18]. From the FTIR spectrum of TiO_2 nanoparticles in Fig. 1, it can be seen that the bands for Ti-O and Ti-O-Ti bonds are available in the 800 - 400 cm^{-1}, the former being determined in a better wave number than the latter [19]. The 800 cm^{-1} peak is assigned to a (Ti-O) vibration, in which the oxygen atom is in a non-binding situation.

The origins of the vibration bands are: the bands in the area 1114–1126 cm^{-1} are because of in-plane bending vibrations of C–H mode of N=Q=N, Q=N H=B, and

B–N H–B, which is shaped for the duration of protonation. The stretching frequency at 3431–3456 cm^{-1} is of the NH of the aromatic amine. Areas at 2852 to 2923 cm^{-1} are of CH vibration. The peaks in 1298–1301, and 1242–1245 cm^{-1} belong to the C–N stretching of the benzenoid ring. Peaks at 1242 cm^{-1} indicate the protonated form of polyaniline. The PANI bands at 1558, 1577, and 1481cm^{-1} belong to the C=N and the C=C modes of vibrations for quinonoid and benzenoid [20].

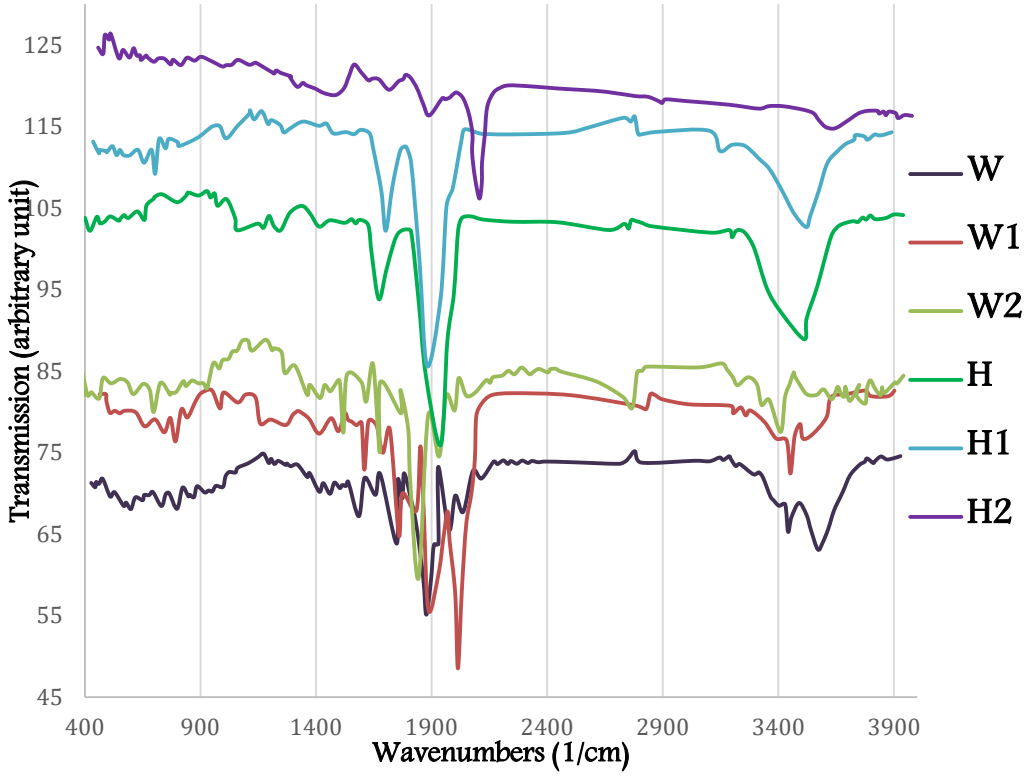

Figure 2: FTIR spectra of the pure PANI with H_2SO_4 (H), without it (W), PANITNCs W1, H1(0.3%), and W2, H2(0.5%).

The results of FTIR demonstrated a shift of the peaks of PANI/TiO_2 nanocomposites to higher wave numbers due to the Coulomb force that resulted from the interaction between the TiO_2 nanoparticles and PANI, confirming that the structural and morphological changes in the nanocomposites have occurred. These assignments of vibrational modes of PANI and nanocomposites bands are listed in Table 2.

3.3. X-Ray diffraction analysis

Fig. 3 shows the X-ray diffraction pattern for pure TiO_2. The TiO_2 NPs peaks appeared at 2θ values of 25.2854°, 37.8654°, 47.8217°, 53.3905°, 62.4606°, and 68.8698° correlated to Millar indices of (101), (004), (200), (105), (213), and (116). The high intensity peaks are 25.2854°, 37.8654°, and 47.8217° with d spacing 3.5181 Å, 2.3732 Å, and 1.8998 Å.

Table 2: Peaks position in the PANITNCs (H, H1, H2, W, W1, and W2) and Assignments of vibrational modes.

Description of vibrations & stretching modes		Wavenumbers [cm^{-1}]					
		H	**H1**	**H2**	**W**	**W1**	**W2**
Ti-O & Ti-O-Ti		464	420	460	532	468	**437**
	CH2 & C–H	648	649	646	696	698	**696**
		846	923	858	860	864	**862**
C-O-C		1043	1045	1012	1080	1043	**1045**
	C–N	1269	1294	1301	1298	1298	**1298**
CH3 & C-H	C=C	1409	1423	1423	1413	1415	**1413**
		1579	1573	1575	1510	1510	**1512**
	C=O	1724	1778	1647	1635	1735	**1639**
O-H & N-H		3431	3425	3436	3434	3267	**3267**

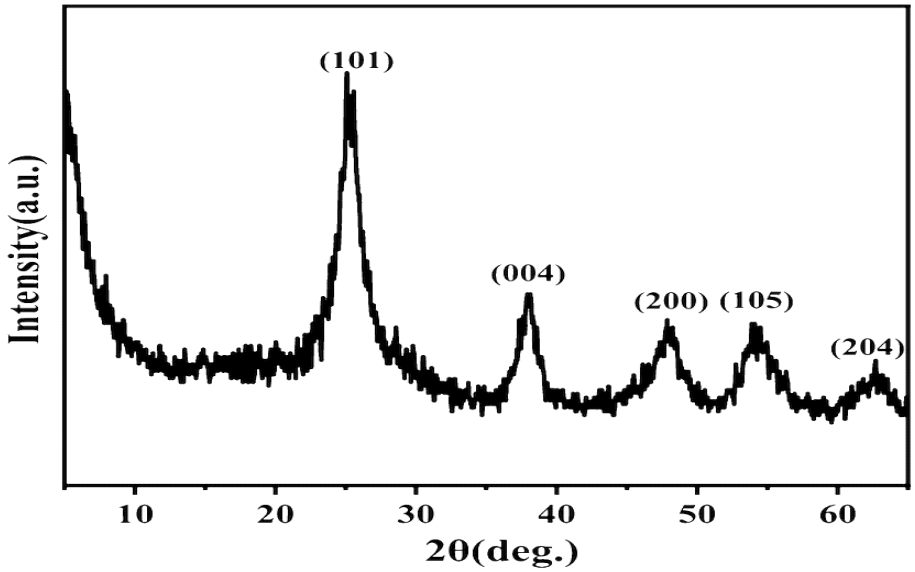

Figure 3: XRD pattern of TiO_2 nanoparticles.

These peaks, in Fig.3, confirm the TiO_2 degree of crystalline, which is confirmed with JCPDS card numbers 21-1272 [21]. The XRD patterns are correlated with the same articles explaining and confirming the preparation of TiO_2 NPs. The peak value of 25.654°, which is one of the peaks for TiO_2 NPs, its correlated crystalline plane was (101). The crystalline size for the samples was observed from the Scherrer equation (Eq. (1) below) [22]:

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \qquad (1)$$

where: λ is the wavelength and D is the crystallite size, β-is the FWHM (full width out half maximum) of the XRD peak. The average crystalline size calculated for the prepared TiO_2 NPs was around 12 nm. The pure PANI peaks showed that a small

peak is around 2θ = 18.495, 20.148, and 25.195, with a d spacing of 4.7933Å, 4.4037Å, and 3.5318Å indicating the conducting PANI; this also confirms the low crystalline structure. The crystalline size of the PANI is around 11 nm, which was calculated from the Scherrer equation [23].

For PANI/TiO_2 nanocomposites, the peak of the pure TiO_2 NPs phase indicates the amorphous nature of nanocomposites. PANI is polycrystalline in structure as shown in Fig.4, which may be allocated to the scattering of PANI chain at d spacing. Ammonium persulfate is mixed to the preparation technique; polymerization happens initially on the surface of TiO_2 NPs due to the restrictive impact of the surface. Thereafter, crystalline PANI combines the crystalline behaviors of TiO_2 NPs to obtain a polycrystalline structure. Therefore, the degree of crystalline of PANI decreases, and the X-ray diffraction peaks revealed combined and decreased with TiO_2 NPs peaks, and then, cannot be distinguished [24].

Comparing pure TiO_2 and pure PANI with nanocomposites, it is clear that the XRD of the PANI/TiO_2 nanocomposites is not identical to the crystalline structure of those of TiO_2 NPs. This explains that PANI polymerization of TiO_2 NPs has affected the crystallization performance of nanocomposites, it was identified that the TiO_2 NPs form a relatively irregular distribution in the PANI chain. Tables 3 & 4 listed the values of (2θ) of the strong peaks for the pure PANI and the nanocomposites.

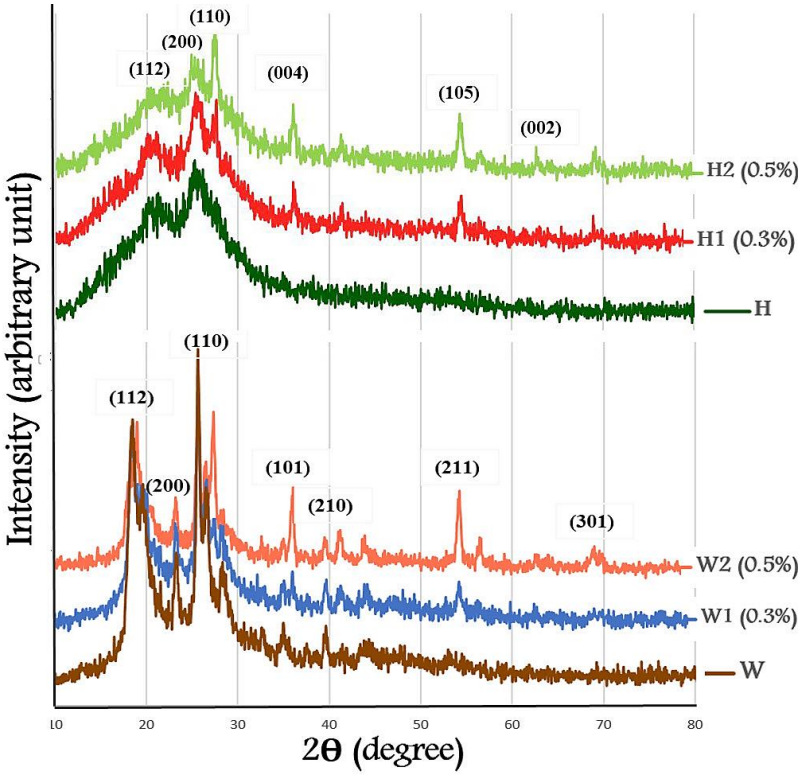

Figure 4: XRD patterns of the pure PANI with H_2SO_4 (H), without it (W), PANITNCs W1, H1 (0.3%), and W2, H2(0.5%).

Table 3: The values of (2θ) of the strong peaks of pure PANI with H_2SO_4 (H , PANITNCs H1(0.3%) and H2(0.5%).

Sample name	2Theta [Deg.]	d Exp.[Å]	FWHM [Deg.]	crystalline size [nm]
H	20.148	4.4037	0.72	11
	21.219	4.1837	0.246	33
	25.195	3.5318	0.83	9
	25.75	3.4569	1.113	7
H1	21.139	4.1994	0.117	72
	25.218	3.5286	0.819	9
	27.57	3.2327	0.231	35
	36.006	2.4923	0.26	32
	41.413	2.1785	0.122	71
	54.43	1.6843	0.362	24
	89.511	1.094	0.13	87
H2	24.856	3.5791	0.651	12
	25.537	3.4852	0.507	16
	27.444	3.2472	0.368	22
	36.005	2.4923	0.274	30
	41.257	2.1864	0.174	49
	44.16	2.0492	0.264	32
	54.248	1.6895	0.374	23
	56.568	1.6256	0.344	26
	62.665	1.4813	0.139	68

Table 4: The values of (2θ) of the strong peaks of the pure PANI with water based only (W), PANITNCs W1(0.3%) and W2(0.5%).

Sample name	2Theta [Deg.]	d Exp.[Å]	FWHM [Deg.]	crystalline size [nm]
W	18.495	4.7933	0.731	11
	23.251	3.8224	0.358	22
	25.654	3.4696	0.386	21
	26.548	3.3547	0.46	17
	28.348	3.1457	0.241	34
W1	19.843	4.4706	0.67	12
	25.654	3.4696	0.362	22
	32.938	2.7171	0.456	18
	35.985	2.4937	0.399	21
	39.692	2.2689	0.417	20
	48.058	1.8916	0.107	83

	49.317	1.8463	0.522	16
	54.193	1.6911	0.544	16
	68.956	1.3607	0.578	16
W2	18.993	4.6687	0.574	14
	20.032	4.4288	0.381	21
	27.347	3.2586	0.37	22
	36.038	2.4902	0.279	30
	41.19	2.1898	0.42	20
	43.89	2.0612	0.202	42
	54.247	1.6895	0.358	25
	56.585	1.6252	0.214	42
	63.944	1.4547	0.21	44
	68.801	1.3634	0.326	29

3.4. Scanning Electron Microscopy (SEM & EDX)

The morphologies of the pure PANI and 0.5 wt% TiO_2 (PANI/ TiO_2) nanocomposites were studied by SEM and EDX as shown in Fig.5. The SEM images show 2D nanosheet morphology. The nanocomposite sheets are rippled and crumpled, with dimensions ranging from 5 to 500 micrometers.

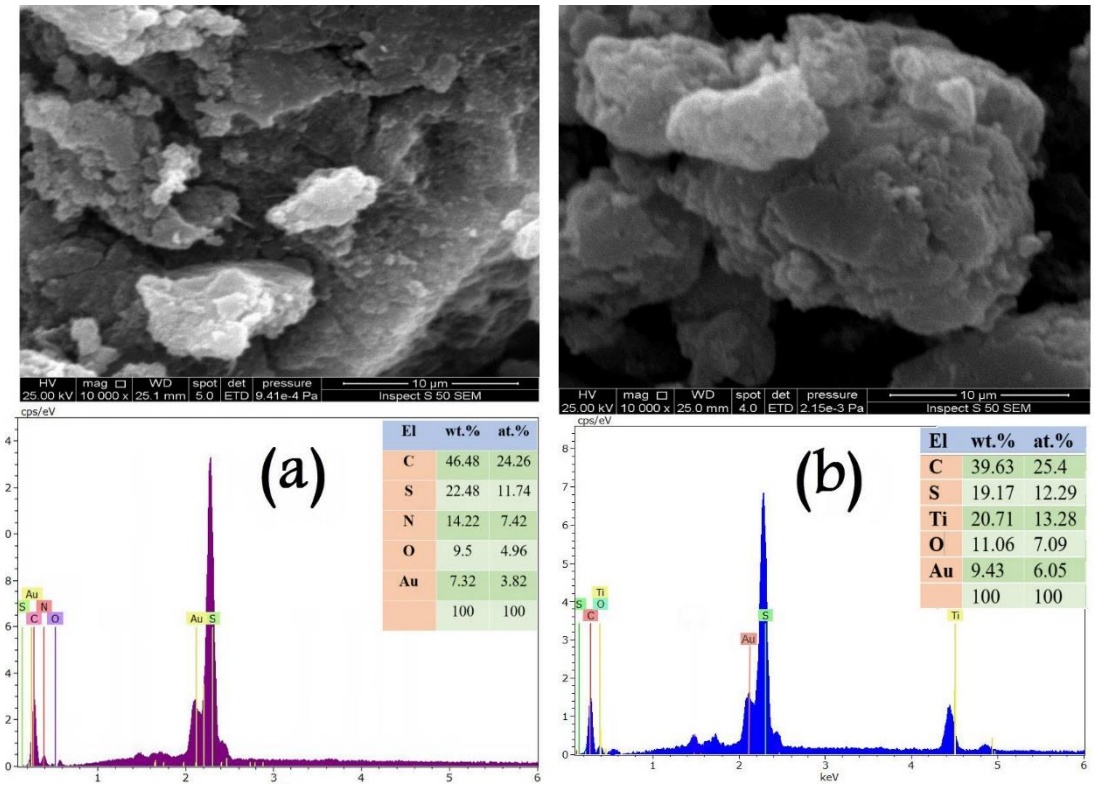

Figure 5: SEM images and DEX of (a)pure PANI (H) (b) PANI/TiO_2 (0.5%) (H2), prepared by chemical polymerization.

Fig. 5-a shows the SEM morphology of pure PANI film acquired in a solution containing 0.1 M aniline and 0.3 M H_2SO_4. From this figure, the spread PANI fibrils

are noticed, and the strands are ordinary and uniform. Additionally, these strands will generally agglomerate into interconnected networks, which show numerous multiple different pores [25].

Fig.5-b shows the SEM images of the (PANI/TiO_2) nanocomposites. The change in surface morphology is observed with the addition of TiO_2 (0.5%) in PANI. An agglomeration of uneven distribution of TiO_2 particles can be seen in the PANI matrix. The diffraction pattern from particles of nanocomposite suggests that the TiO_2 nanoparticles are deposited on the surface of PANI and show a typical rutile phase. The EDX analyses of the polymer and nanocomposite are also performed to confirm the incorporation of the nanoparticles in the PANI matrix. The intensity of the NPs in the nanocomposites peaks is found to appear by adding the nanoparticles.

4. Conclusions

PANI/TiO_2 nanocomposite was synthesized with the aid of using in-situ chemical polymerization by various substance materials (with H_2SO_4 0.3 M and without it), and the resulting PANI and PANI/TiO_2 nanocomposite base on the water is less efficient when contrasted with doping by H_2SO_4. An acidic medium promotes the solubilization of the monomer, the aniline, in water and limits the secondary reactions. The nature of the acid influences the polymerization time, morphology, physicochemical properties, and molar mass. It was displayed in the attributes of PANI/TiO_2 by XRD and FTIR that the nanoparticles will generally fix along the PANI chain with expanding TiO_2 concentration. The nanocomposite results were obtained with FTIR studies, which confirmed the formation of PANI/TiO_2 nanocomposites. It showed a shift to higher wave numbers in the peaks of PANI/TiO_2 nanocomposites. XRD pattern showed that the TiO_2 NPs and PANI significantly affected the crystallization performance of nanocomposites. SEM images show that the TiO_2 NPs enwrap the polyaniline and agglomeration uneven distribution of TiO_2 NPs can be seen in the PANI matrix. It was identified that the TiO_2 NPs form a relatively irregular distribution in the PANI confirming that structural and morphological changes in the nanocomposites occurred.

Acknowledgments

The authors would like to thanks University of Baghdad/ College of Science/ Physics Department.

Conflict of interest

All authors declare that they have no conflicts of interest.

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الخصائص التركيبية لمتراكب نانوي محضر من $PANI/TiO_2$ بواسطة البلمرة الكيميائية

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الخلاصة

تم تحضير المتراكب النانوي المتكون من البولي انيلين (PANI) وثاني أكسيد التيتانيوم (TiO_2) من خلال استراتيجية البلمرة في الموقع باضافة الجسيمات النانوية TiO_2. تم تحليل المتراكبات النانوية ($PANI / TiO_2$) الناتجة باستخدام أطياف الأشعة تحت الحمراء (FTIR) وحيود الأشعة السينية (XRD) والفحص المجهري الإلكتروني (SEM) وتحليل الأشعة السينية المشتتة للطاقة (EDX) المأخوذة للعينات المعدة. تم تحضير نسب مختلفة من الـ $PANI: TiO_2$ بواسطة تقنية الأكسدة الكيميائية باستخدام بيرسلفات الأمونيوم (APS) كمؤكسد بوجود الجسيمات النانوية من TiO_2 المبرد في حمام جليدي. تم تحضير المتراكبات النانوية بإضافة نسبتين من TiO_2 هما (3.0 و 5.0 ٪) غرام أثناء عملية بلمرة البولي انيلين. أظهرت النتائج توافقاً جيدًا بين PANI و. TiO_2 حيث يظهر طيف FTIR انحرافاً نحو الاعداد الموجية الأعلى في قمم المتراكبات النانوية $PANI / TiO_2$، بسبب قوة كولوم الناتجة عن التفاعل بين جسيمات TiO_2 النانوية مع البولي انيلين. تُظهر نتائج فحص الـSEM أن جسيمات TiO_2 النانوية تغلف البولي انيلين والتوزيع غير المتكافئ لجزيئات TiO_2 التي يمكن رؤيتها ضمن جزيئات البولي انيلين. تم العثور على شدة الذروة للجسيمات النانوية المضافة من خلال تحليل الـEDX. يوضح تشخيص XRD لبلمرة PANI و PANITNCs أن الجسيمات النانوية TiO_2 و PANI لهما تأثير على أداء التبلور للمتراكبات النانوية، وقد وجد أن جسيمات TiO_2 النانوية تشكل توزيعًا غير منتظم نسبيًا على طول سلسلة البولي انيلين.