

# State of the Art of Synthesized PANI-(Sn<sup>+2</sup>/TiO<sub>2</sub>) Nanocomposites for Conductive Application

Qusay N. Al-Haidary<sup>1,2</sup>, Ali M. Al-Mokaram<sup>1,\*</sup>, Ahmad H. Ismail<sup>1</sup>, Fadhela M. Hussein<sup>1</sup>

<sup>1</sup>Department of Chemistry, College of Science, Mustansiriyah University, Baghdad, IRAQ.

<sup>2</sup>Dijlah Intermediate School for Boys, General Directorate of Education for the third Karkh, Ministry of Education, Baghdad, IRAQ.

\*Correspondent contact: [ali75@uomustansiriyah.edu.iq](mailto:ali75@uomustansiriyah.edu.iq)

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## ABSTRACT

Conducting polymer nanocomposites are the forthcoming materials for developing technologies, as they possess a combination of unique properties of their components. This study used the sol-gel technique to prepare and fabricate nanocomposite of polyaniline (PANI), with nanomaterial (TiO<sub>2</sub>), doped by tin (Sn<sup>+2</sup>) (PANI/Sn<sup>+2</sup>/TiO<sub>2</sub>). Novel nanocomposites were prepared in different ratios (5%, 10%, 15%, 20%, and 25%) of weight for the nanomaterial (Sn<sup>+2</sup>/TiO<sub>2</sub>) to the polymer (PANI). The prepared nanocomposites were characterized with several techniques including Fourier transform infrared (FT-IR) Spectrometer, Ultraviolet-visible (UV-vis.) spectroscopy, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and Atomic Force Microscopy (AFM). This research has presented the role of nanomaterial and described the easy process of their homogenous distribution in the (PANI/Sn<sup>+2</sup>/TiO<sub>2</sub>) nanocomposites. The electrical conductivity of the prepared materials was examined and confirmed by electrical conductivity. The combination of Sn<sup>+2</sup>/TiO<sub>2</sub> in the PANI matrix will be very valuable for the improvement of the physical and chemical properties of the polymer's conductivity.

**KEYWORDS:** Polyaniline, Conductivity, Nanocomposites, Nanomaterial.

## الخلاصة

إن مركبات البوليمر النانوية هي المواد الواعدة لتطوير التقنيات، لأنها تمتلك مجموعة من الخصائص الفريدة لمكوناتها. تضمنت هذه الدراسة استخدام تقنية sol-gel لتحضير وتصنيع المركب النانوي من البولي أنيلين (PANI)، مع مادة متناهية الصغر (TiO<sub>2</sub>)، مشوب بالقصدير (Sn<sup>+2</sup>). ومكونة (PANI/Sn<sup>+2</sup>/TiO<sub>2</sub>) تم تحضير المركبات النانوية الجديدة بنسب مختلفة (5%، 10%، 15%، 20%، 25%) من وزن المادة النانوية (Sn<sup>+2</sup>/TiO<sub>2</sub>) إلى البوليمر (PANI) وتم تمييز المركبات المحضرة بعدة تقنيات مثل مطياف فورييه للأشعة تحت الحمراء (FT-IR)، مطياف الأشعة فوق البنفسجية المرئية (UV-vis)، حيود الأشعة السينية (XRD)، أيضًا فحص المسح المجهر الإلكتروني (SEM)، ومجهر القوة الذرية (AFM). قدم هذا البحث دور المواد النانوية ووصف العملية السهلة لتوزيعها المتجانس في المركبات النانوية (PANI/Sn<sup>+2</sup>/TiO<sub>2</sub>). تم فحص التوصيل الكهربائي للمواد المحضرة والتأكد من الموصلية الكهربائية. وبين أن الجمع بين Sn<sup>+2</sup>/TiO<sub>2</sub> في مصفوفة PANI مفيداً جداً لتحسين الخواص الفيزيائية والكيميائية لموصلية البوليمر.

## INTRODUCTION

Conducting polymers are commonly used as a matrix to metal nanoparticles [1]. The conducting polymer/nanoparticle-composites, having useful and unique properties [2]. An efficient method to enhance the mechanical stability of conducting polymer (CP) is to make composites with nanoparticles or other polymers, which have better mechanical properties for their purposed applications than their original analogs [3]. Conducting polymer nanocomposites have interesting physical and chemical properties and, important application

possibilities, and a rise in the efficiency of materials, due to their high surface area. [4]. Polyaniline (PANI), as a conducting polymer, has attracted a lot of attention nowadays. [5]. As well, the conducting polymer with inorganic nanoparticle composites in different combinations for two components has attracted much attention. [6]. In 2014, W.-H. Jeong and his co-worker were prepared a novel polyaniline (PANI) nanotube/TiO<sub>2</sub> composite as an effective chemical and biological sterilizer. [7]. A. D. Chowdhury groups in the same year were made a biosensing platform by the covalent link of

biomolecules on PANI-nanowire, which contained the gold nanoparticles. [8]. In the same year too, R. Bushra and co-worker introduced the preparation and environmental applications of conducting polyaniline/(Sn<sup>+4</sup>) tungsten molybdate as nanocomposite. [9].

At the same time, according to the study presented by, H. Sun, with research groups, the titanium dioxide/nanoparticles were introduced into poly (ethylene oxide) with silver tetrafluoroborate polymer electrolyte membranes to separation propylene/propane [10]. Furthermore, N. A. K. Umiati and the research group were tested the electrical properties of polyaniline with molarity dopant variation. It was observed that the electrical property on the polyaniline was carried out by using a High Precision LCR meter. The results of this study confirmed that the increase in dopant molarity gives an effect on the increment of conductivity [11].

G. M. Hou, and co-worker, in 2016, showed a new polymer nanocomposite of nano-titanium dioxide/polyethylene oxide within situ synthesis hyper-branch poly (amine-ester) and its applications as a polymer electrolyte [12]. Al-Mokaram and his groups prepared Ppy/CH/TiO<sub>2</sub> Nanocomposite Films [13]. Additionally, León studied the modifying of TiO<sub>2</sub> nanoparticles with polyethylene glycol full with (2-ethoxyestradiol) drug and revealed that the nanocarrier TiO<sub>2</sub> has a functional adsorption ability [14]. In 2017

X. X. He, and researcher group, they were the fabrication of porous polyaniline (PANI) composite, prepared by a situ polymerization, and the ability to use it as a good performance pressure sensor. [15].

R. Megha and co-worker investigate the enhancement of the alternating conductivity of (PANI/TiO<sub>2</sub>) fabrication by a one-step synthesis thermal way, which was used for preparing polyaniline-titanium dioxide composite through in-situ polymerization. [16]. Polyaniline is considered to be one of the special polymers amongst conducting polymers in highly conducting doped formed, it can be done by two completely different processes-protonic acid doping and oxidative doping. [17]

In this research prepare a novel composite polymer "polyaniline (PANI)", is prepared with a nanomaterial tin-doped and titanium dioxide,

in different ratios of (Sn+2/TiO<sub>2</sub>), to obtain the properties of electrically conducting materials.

## MATERIALS AND METHODS

All solvents and reactants utilized in this research were reagent grade and were purchased from Sigma Aldrich and Fluka. The Infrared spectra (FT-IR) were acquired with the use of a SHIMADZU FT-IR8400S spectrophotometer. The electrical conductivity value of some prepared compounds, by an Electrical conductivity device, model EC.71 Sens ION made in Germany. Ultraviolet-visible schematics a visible Spectrophotometer using the device, Roya LAB, model 721 has all been tested before. At the Dept. of Chemistry-Collage of Science/Univ. of Mustansiriyah. Atomic force microscopy (AFM) was taken by using AFM (spm-AA300 contact mode spectrometer, Angstrom) and has been done in the chemistry department at The University of Baghdad Scanning electron microscopes (SEM) was taken by using the instrument, ZELZZ, 400-kz-012, The X-ray, Diffraction (XRD) diagrams of the samples prepared by a device SHIMADZU 7000, and has been done in Iran.

### 1. Preparation of polyaniline

Polyaniline was prepared by situ chemical oxidative polymerization, according to J Stejskal and R. G. Gilbert [18]. Briefly, 0.2 M of aniline hydrochloride (2.59 g, 20 mmol) dissolved in (distilled water 50 mL)-(solution No.1). 0.25 M ammonium persulfate ((NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) (as oxidant agent), (5.71 g, 25 mmol) dissolved also in (distilled water 50 mL)-(solution No.2). Both solutions were cooled in the refrigerator for (two to three hours) and then added by drop wise solution No.2 to solution No.1, keeping the temperature of mixing between (0-4 °C) by using an ice bath, the color was changed gradually dark-green, with stirred (600 rpm) for three hours and then left for 24 hours at rest to complete the polymerize in the refrigerator. Then, the precipitate of polyaniline was collected on filter paper, following washed with 1M HCl and acetone until the filtrate becomes colorless. Finally, the powder was dried in the oven at (50-60°C) for 24 hours.

## 2. Polyaniline/Tin/Titanium-dioxide (PANI/Sn<sup>+2</sup>/TiO<sub>2</sub>) nanocomposite

PANI/Sn/TiO<sub>2</sub> nanocomposite was prepared by the technique of in-situ polymerization [19]. The various compositions of (PANI/Sn/TiO<sub>2</sub>) were prepared w/w% weight percentage of titanium dioxide, by denoted as (5%, 10%, 15%, 20%, and 25%). Briefly, the amount of (Sn dopants with titanium dioxide nanoparticles), were dispersed in deionized water 15 mL containing (0.0025 mol aniline and 0.2 M HCl), stirring for twenty minutes under ultra-sonication. Ammonium persulfate (APS) (0.0025 moles dissolved in 5 mL of deionized water was added drop wise to the above solution, and then left to react with constant stirring (300 rpm) for 12 hours. After that filtered by using filter paper (0.45 μm). A dark green precipitate was obtained and then washed with ethanol and distilled water many times until the filtrate become colorless. Finally, the precipitate was dried at 40 °C for 24 hours [20] as seen in Figure 1; the mechanism preparation is shown in figure 2.



Figure 1. Preparation of PANI/Sn/TiO<sub>2</sub>.

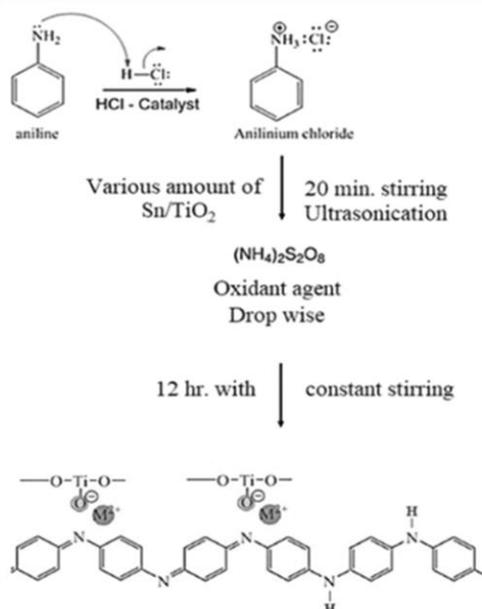


Figure 2. Illustrated the mechanism of fabrication PANI/Sn<sup>+2</sup>/TiO<sub>2</sub>.

## RESULTS AND DISCUSSION

PANI/Sn/TiO<sub>2</sub> nanocomposite was studied by, Fourier transforms infrared spectroscopy (FT-IR) instrument, in rang (600-4000 cm<sup>-1</sup>). Then the comparison between them was carried out. This comparison is to ascertain the (Metal-Oxygen) bond and, the shift in frequency in the PANI with metal oxide inserted PANI composite sample. The spectrum of the prepared nanomaterial (Sn<sup>+2</sup>/TiO<sub>2</sub>) gives a strong peak at 661 cm<sup>-1</sup>, corresponding to (Ti-O-Ti) bonding, while at 630 cm<sup>-1</sup> related with (Sn-O) [21]. As shown in figure 3. The novel Sn<sup>+2</sup>/TiO<sub>2</sub> nanocomposites-particles, which intercalated on the PANI, were successfully prepared in this work by the chemical oxidative method.

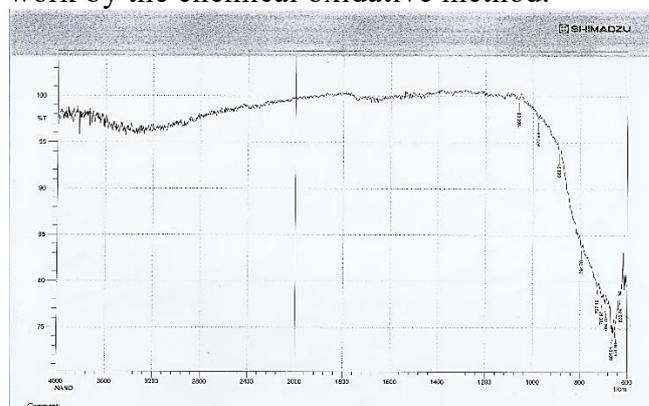
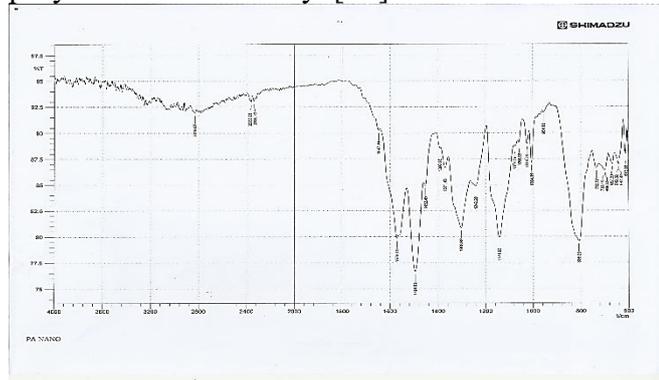


Figure 3. FT-IR Sn/TiO<sub>2</sub>.

In Figure 4 all ratios of samples, the peaks are presented at 808, 1141, 1303, 1494, and, 1570 cm<sup>-1</sup> respectively. The spectrum gives some peaks below 1000 cm<sup>-1</sup>, of metal oxide [22]. Some additional shifting in frequency was also observed in comparison with the pure PANI and Sn/TiO<sub>2</sub> spectrum. The composite of nano TiO<sub>2</sub> leads to the shift of several FT-IR bands [6]. The main peaks at 1570 cm<sup>-1</sup> and 1494 cm<sup>-1</sup> are related to (C=N) and (C=C) stretching-vibrations of the quinoid and the rings of benzene, respectively. The peaks at 1303 cm<sup>-1</sup> are attributed to the (C-N) stretching style, the secondary aromatic amine. The peak at 1141 cm<sup>-1</sup> is due to the quinoid unit of composite with PANI (the =N<sup>+</sup>H- stretching). As well as a peak at 808 cm<sup>-1</sup>, and 696 cm<sup>-1</sup>, are related to the (Ti-O) and (Sn-O) respectively. These peaks were confirmed the succeeded form of PANI/Sn/TiO<sub>2</sub> [23, 24, 25]. The absorption changes in the peaks of PANI/Sn/TiO<sub>2</sub> composites are perhaps due to the effective

integration of the Sn/TiO<sub>2</sub> within the chain of the polymer. In addition, the shifts in the absorption, peak position is related to the presence of the Sn/TiO<sub>2</sub> in the PANI matrix as mentioned in a previous study [6]. This provides a hint that the incorporation of Sn/TiO<sub>2</sub> in the PANI matrix will be very beneficial for the improvement of the physical and chemical properties of the polymer's conductivity. [26].



**Figure 4.** FT-IR PANI/Sn/TiO<sub>2</sub> nanocomposite.

The conjugated systems such as PANI, meaning have a degree of specificity. These systems absorb the UV at a higher wavelength and in higher intensity. The models were examined in DMF solvent (due to complete solubility), with the weight ratios, 5%, 10%, 15%, 20% and, 25% of Sn<sup>2+</sup>/TiO<sub>2</sub>, respectively. The wavelength between the ranges is about 360 nm and 626 nm. Compared with the polyaniline form of emeraldine base. The peak at 360 nm was pointed to the ( $\pi$ - $\pi^*$ ) transition in the benzenoid ring. While the peak at 626 nm was related to a benzenoid to quinoid transition [27]. The intensity of the wavelength increases with increasing the concentration of nanomaterials, which are composited with polyaniline. The value spectrum of UV-Vis of polyaniline included two bands at ~320 and 626 nm due to the stimulation of the benzenoid and quinoid parts in the polyaniline [28]. The electrons transition in the PANI emeraldine salt:

From ( $\pi \rightarrow \text{polaron}$ ), ( $\text{polaron} \rightarrow \pi$ ), and, ( $\pi \rightarrow \pi$ ). The band at 320 nm is pointed to the transition ( $\pi \rightarrow \pi$ ) band centered on the benzenoid rings related with the extent of ( $\pi$  orbitals), on the polymer backbone (band-gap excitation).

Furthermore, the blue-shifted of these bands are depending on the concentration of the TiO<sub>2</sub>. The shift pointed to redistribution for density of polaron in the band gap of PANI Emeraldine,

because of the impact of TiO<sub>2</sub> nanoparticles. The peaks of absorption of ( $\pi \rightarrow \text{polaron}$ ), are variations. The intensity ratios at (A1.236/A1.319/A1.413/A1.587/A1.800) of the composites are higher than PANI (A0.968), as shown in (Table 1) indicating that the composite level of Sn/TiO<sub>2</sub> with PANI, is higher than pure PANI [29]. The results from the value of UV-Vis absorption spectra mean that the Sn/TiO<sub>2</sub> has confirmed effects on the polymer matrix.

**Table 1.** UV/Vis wavelength values in nm for PANI/Sn/TiO<sub>2</sub> nanocomposite.

No	Sample	wave-length	Abs.	Type	Ratio Sn/TiO <sub>2</sub>
1.	PANI	626	0.968	Peak	0 %
		322	1.301	=	=
		434	0.206	Valley	=
2.	PANI/Sn <sup>2+</sup> /TiO <sub>2</sub>	626	1.236	Peak	5 %
		320	1.346	=	=
		432	0.370	Valley	=
3.	PANI/Sn <sup>2+</sup> /TiO <sub>2</sub>	626	1.319	Peak	10 %
		316	1.378	=	=
		434	0.396	Valley	=
4.	PANI/Sn <sup>2+</sup> /TiO <sub>2</sub>	626	1.413	Peak	15 %
		320	1.361	=	=
		432	0.399	Valley	=
5.	PANI/Sn <sup>2+</sup> /TiO <sub>2</sub>	626	1.587	Peak	20 %
		318	1.367	=	=
		436	0.506	Valley	=
6.	PANI/Sn <sup>2+</sup> /TiO <sub>2</sub>	626	1.800	Peak	25 %
		312	1.357	=	=
		430	0.491	Valley	=

The X-ray diffraction patterns results showed the average sizes of crystalline materials. Figure 5, display the XRD spectra of PANI/Sn<sup>2+</sup>/TiO<sub>2</sub>, the peaks in the XRD patterns can be indexed the peaks at  $2\theta=25.32$ ,  $38.35$ , and,  $48.76$  for the anatase phase of TiO<sub>2</sub>, and the peaks at  $2\theta=36.15$ ,  $54.08$ ,  $63.03$  for Sn<sup>2+</sup>/TiO<sub>2</sub>. While the presence of peaks at  $2\theta = 20.22$  and  $69.29$  indicated to the polymers. The crystalline size can be determined from the classical Scherrer equation (1) [30].

$$D = K\lambda / (\beta \cos \theta) \quad (1)$$

It was found for PANI/Sn/TiO<sub>2</sub> (11.11 nm). Where (D) is the crystallite size, K = 0.89 is the shape factor, ( $\lambda$ ) is the X-ray wavelength, ( $\beta$ ) is the full width at half of the diffraction peak, in the radians (Equation 1), and ( $\cos \theta$ ) is the cosine of the Bragg angle ( $\theta$ ). [16] and [31].

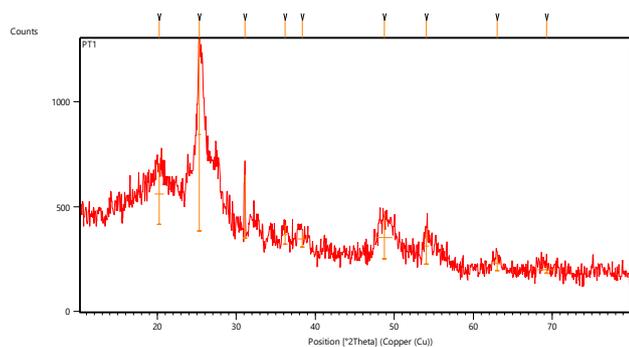


Figure 5. XRD of PANI/Sn/TiO<sub>2</sub>.

Figure 6 of SEM image, in scale 200 nm with magnified 20 KX, shows the Sn/TiO<sub>2</sub> particle is incorporated into the surface of the PANI matrix. The SEM results showed that the PANI/nanoparticles exhibited some extent of agglomeration because of the interaction between the Sn/TiO<sub>2</sub> nanoparticles. This image shows the regular particles are compressed on each other. Most particles form arrangements with compact structures that showed the crystalline structure, where the granular structure or cluster, and the micrometer-sized particles were observed on the surface of PANI/Sn/TiO<sub>2</sub>, confirming emeraldine salt formation, in good agreement with those reported by others [24]. The microstructural regular feature was formed by the secondary growth stages of PANI, during the initial growth phase of the chain of the polymer [32].

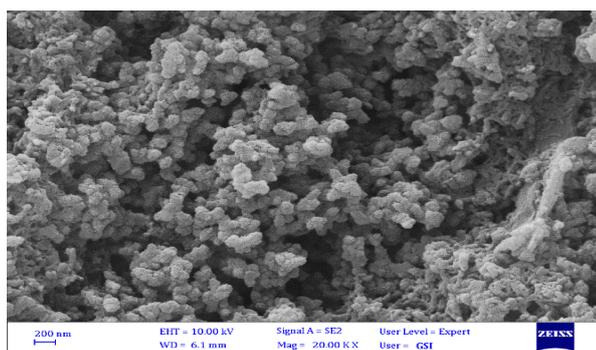


Figure 6. SEM image of PANI/Sn/TiO<sub>2</sub>.

It was Investigated that the surface topography of (PANI/Sn/TiO<sub>2</sub>) by AFM. Moreover, Figure 7, illustrates the images of two and three-dimensional (2D and 3D) also demonstrated the grain size distribution, where the average diameter is 88.72 nm. See Figure 7.

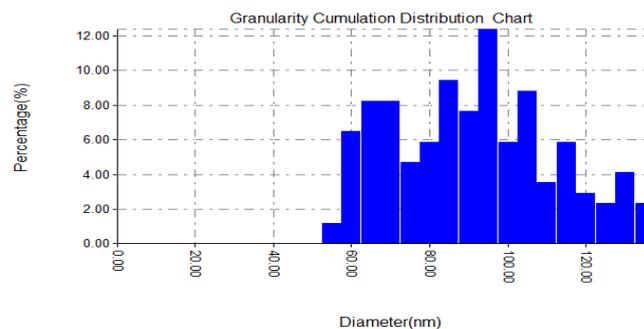


Figure 7. AFM chart of PANI/Sn/TiO<sub>2</sub>.

The (3D), images in figure 9, it display the bumpy shape with a large number of valleys. These structures make a rough surface. The (2D) images in figure 8, display quasi-uniform stacked particles. The AFM image observation perhaps for the restructuring of molecular structure, to be more suitable for ions diffusing into the matrix of the polymer [33].

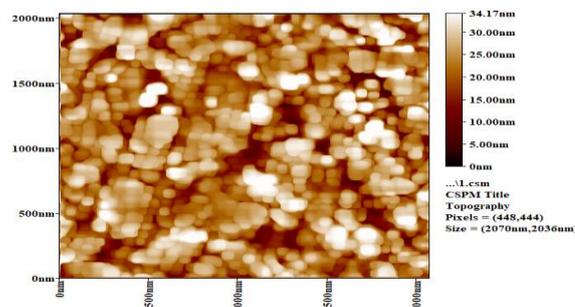


Figure 8. AFM (2D) of PANI/Sn/TiO<sub>2</sub>.

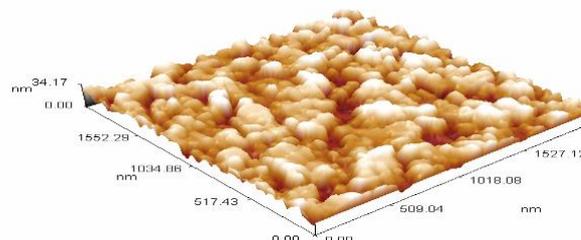
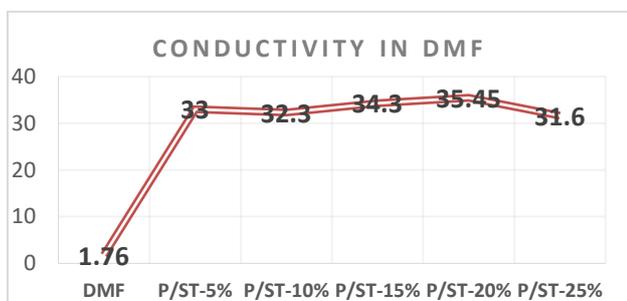


Figure 9. AFM (3D) of PANI/Sn/TiO<sub>2</sub>.

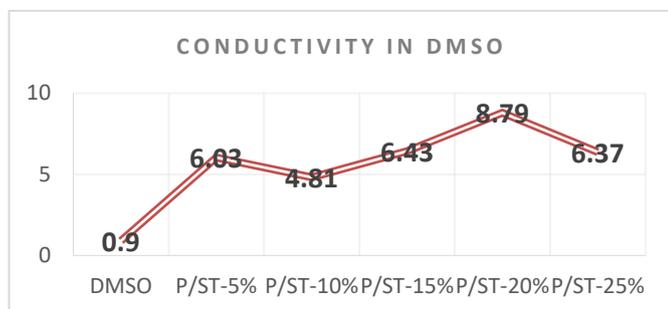
The conductivity of the prepared materials, which are soluble in DMF or DMSO, was carried out by an electrical conductivity device (EC.71), at room temperature. It notices that the obvious variation in the electrical conductivity of the prepared materials when the nanomaterial is gradually increased. However, gives the higher conductivity at a ratio of 20% (w/w %) of Sn/TiO<sub>2</sub> nanocomposite, in both DMF and DMSO solvents. As shown in Tables (2 and 3), and figures 10 and 11.

**Table 2.** Conductivity in DMF.

No.	sample	EC.	unit	Notes
1.	0.1 M KCl	12.73	mS.cm <sup>-1</sup>	at room temperature 0.005g in the 20mL DMF
2.	DMF	1.76	μS.cm <sup>-1</sup>	
3.	P/ST-5%	33.00	μS.cm <sup>-1</sup>	
4.	P/ST-10%	32.30	μS.cm <sup>-1</sup>	
5.	P/ST-15%	34.30	μS.cm <sup>-1</sup>	
6.	P/ST-20%	35.45	μS.cm <sup>-1</sup>	
7.	P/ST-25%	31.60	μS.cm <sup>-1</sup>	

**Figure 10.** Conductivity of PANI/Sn/TiO<sub>2</sub> in DMF.**Table 3.** Conductivity in DMSO.

No.	Sample	EC.	Unit	Notes
1.	0.1 M KCl	12.78	mS.cm <sup>-1</sup>	at room temperature 0.005g in the 20mL DMSO
2.	DMSO	0.90	μS.cm <sup>-1</sup>	
3.	P/ST-5%	6.03	μS.cm <sup>-1</sup>	
4.	P/ST-10%	4.81	μS.cm <sup>-1</sup>	
5.	P/ST-15%	6.43	μS.cm <sup>-1</sup>	
6.	P/ST-20%	8.79	μS.cm <sup>-1</sup>	
7.	P/ST-25%	6.37	μS.cm <sup>-1</sup>	

**Figure 11.** Conductivity of PANI/Sn/TiO<sub>2</sub> in DMSO.

## CONCLUSION

The functionalized nanocomposites of PANI - (Sn<sup>2+</sup>/TiO<sub>2</sub>) were successfully formulated via the situ oxidation method by fabrication of (Sn/TiO<sub>2</sub>) using the sol-gel method. The FTIR provides the incorporation of (Sn/TiO<sub>2</sub>) in the (PANI) matrix, showing an advantage with an enhancement of the physical and chemical properties of the polymer's conductivity. The PANI and pure metal-oxide, with existing oxide material, are observed by the XRD pattern. Therefore, it was determined that the existing nanocomposite between (PANI) / (Sn<sup>2+</sup>/TiO<sub>2</sub>), produces electrically conductive materials and

that the most conductive was at (20 w/w %) a percentage of the nanomaterial (Sn<sup>2+</sup>/TiO<sub>2</sub>). It was found through the results of the XRD, SEM, and AFM examinations that the size of the crystals of the substance (PANI/Sn<sup>2+</sup>/TiO<sub>2</sub>), and the average size of nanoparticles of the substance (PANI/Sn<sup>2+</sup>/TiO<sub>2</sub>) is high, which indicates that the properties are better for conductivity.

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