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Improving the electrochemical behavior of sustainable polyaniline titanium dioxide composite by intercalation of carbon nanotubes

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Abstract: Polyaniline titanium dioxide (PANI-TiO₂) composite had improved electrochemical behavior by intercalation of varied carbon nanotubes (CNTs): from 0 wt% to 30 wt%, compared to aniline. Those composites were coated on the titanium substrate to form PANi-TiO₂-CNTs/Ti by using a binder agent. Material properties were characterized by IR spectra, X-ray diffraction and thermal analysis. The scanning electron microscope (SEM) images explained that the composite existed in uniform fibers with diameter of about 100 nm if content of CNT was 20–30 wt%. Electrical conductivity measurement, electrochemical impedance spectroscopy (EIS) and cyclic voltammograms showed their excellent electrical performance: electrical conductivity reached approximately 10⁻¹ S/cm, and the resistances of those composite layers were reduced from 18 to 27 times compared to that without CNTs. Warburg diffusion was found by EIS analysis with coefficient data ranging from 10⁻¹⁵ to 10⁻¹⁶ cm²/s only for composites containing CNTs on which also no adsorption resistance occurred. The potentiodynamic and galvanodynamic polarizations explained that the composite contained 20% CNTs, exhibiting a better cyclic voltammetry (CV) diagram because of higher responding current density and a better performance because of yielding a higher current density (300 μA/cm²) at 250 mV versus silver/

silver chloride reference electrode in comparison with the other obtained composites in this study, respectively.

Keywords: anode materials for MFC; cyclic voltammetry; dynamic current polarization; electrochemical impedance spectroscopy; PANi-TiO₂-CNTs nanocomposite.

1 Introduction

Currently, organic-inorganic hybrid materials obtained from conducting polymers like polyaniline (PANI) and inorganic oxides such as TiO₂ [1, 2], MnO₂ [3], V₂O₅ [4], and PbO₂ [5] have been regarded as mostly having potential practical applications in many fields such as microbial fuel cells (MFC) [6], solar cells [7], sensors [8], electrocatalysis [9], etc. Among them, PANi-TiO₂ is a friendly environmental composite which is used widely to fabricate MFC because of characterization properties of PANi and TiO₂. PANi is known as a stable environmental conductive polymer. TiO₂ is a semiconductor with high photocatalytic activity, has a durable structure and is non-toxic. Therefore, it is used as a biocompatible stable anode material for MFC. However, the conductivity of PANi-TiO₂ composite is low. Improving properties of this composite is attracting the attention of scientists. Studies showed that the presence of carbon nanotubes (CNTs) in components of composites improved not only their mechanical strength and toughness, but also their electrical conductivity and stability under fuel cell operation conditions [10] because of the super-conductive CNTs.

As reported in [11] MFC convert chemical energy to electrical energy owing to electrons produced by bacteria from substrates such as brewery wastewater [12] or food and agriculture wastes [13]. Among these, brewery wastewater is non-toxic, but its chemical oxygen demand concentrations are high in comparison with other domestic or industrial wastewaters [14, 15]. Additionally, it becomes a potential fuel substrate for MFC because it contains a high concentration of carbohydrate and a low one of ammonium nitrogen [16]. Therefore, it was used as the

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electrolyte in our research for electrochemical characterization of composite electrode materials.

The aim of this research was intercalation of CNTs into PANi-TiO₂ matrix by chemical polymerization to improve their electrochemical behaviors in brewery wastewater. The study also examined the effect of CNTs content on material properties.

2 Materials and methods

2.1 Materials

Some agents such as ammonium persulfate ((NH₄)₂S₂O₈), chlorhydric acid, acetone and methanol were provided by Merck (Germany), dodecylbenzenesulfonic acid (70 wt%) was provided by Sigma-Aldrich (USA), and aniline by Kanto Chemical (Japan). Aniline and titanium electrodes were pretreated as reported in our previous research [17] before use. Sol-gel TiO₂ and CNTs were provided by the Institute of Applied Physics and the Institute of Materials Science, both of which belong to Vietnam Academy of Science and Technology (VAST) of Vietnam. The chitosan was provided by our Institute of Chemistry, and acetic acid by Merck (Germany). Thales software belonging to electrochemical equipment IM6 was provided by Zahner-Elektrik GmbH & Co KG, Germany.

2.2 Fabrication of PANi-TiO₂-CNTs composite

The PANi-TiO₂-CNTs nanocomposites were prepared by chemical polymerization of 0.1 M aniline in 1 M chlorhydric solution containing TiO₂, CNTs and surface-active agent (0.015 M dodecylbenzenesulfonic acid) under stirring at temperature of about 0–5°C and dropping oxidative ammonium persulfate solution (molar ratio to aniline is 1 : 1). CNT content ranging from 0 wt% to 30 wt% was used to consider its effect on material properties, keeping the mass ratio of titanium dioxide to monome at 1 : 6. The reaction time was a full day, with stirring in the initial 8 h. After finishing the polymerization process, the product was obtained by firstly filtrating, cleanly washing with distilled water and finally spreading it by mixed solvent of acetone/methanol (mass ratio 1 : 1) for removing the rest of the monome. It was dried under vacuum at a temperature of 50°C for 2–3 h.

Electrodes were prepared by covering the paste of obtained composites onto pretreated titanium substrate mentioned above for electrochemical measurements. The chitosan acetic acid solution (1%) was used for fabricating this paste and the fabricated electrodes were dried at 120°C for 2 h. The chemical oxygen demand concentration in substrate electrolyte (brewery wastewater) was 3555 mg/l provided for electrochemical measurements.

2.3 Detection method

Characteristic groups in the molecular of PANi-TiO₂-CNTs composite were determined by IR spectra on a Nicolet Impact 410 FTIR Spectrometer (Germany). The morphological structure of materials was

analyzed by a scanning electron microscope (SEM) and transmission electron microscope (TEM) on FE-SEM Hitachi S-4800 (Japan) and a Jeol 200CX (Japan), respectively. The chemical structure was determined by X-ray diffractometer D8-Advance Bruker (Germany). The thermal stability was analyzed by a thermal detector (Labsys Evo, France) until 1000°C, with a heat flow of 10°C/min under nitrogen gas.

The electrical conductivity measurements of materials which were pressed in a cylinder form with thickness of 3 mm and diameter of 3 mm were carried out by cyclic voltammetry (CV) at scan rate of 100 mV/s using a two-point-electrode method without electrolytes on the electrochemical equipment IM6 (Zahner Elektrik GmbH & Co KG).

The conductivity δ (mS/cm) can be calculated by the following equation [18]:

$$\delta = \frac{(\Delta I * d)}{\Delta E} \quad (1)$$

where ΔE is the difference of potential (mV), ΔI is the difference of responding current (mA) and d is thickness of sample (cm).

Electrochemical impedance spectroscopy (EIS) analysis, CV, and galvanodynamic polarization were carried out on IM6 equipment using a three-electrode cell in brewery wastewater. A platinum plate was used as the counter electrode, PANi-TiO₂-CNTs/Ti composite electrode (diameter of 6 mm) as the working electrode and silver/silver chloride (saturated KCl) as the reference electrode.

EIS diagrams were taken at open circuit potential in the frequency area of 100 kHz to 10 mHz (amplitude of 5 mV). The Thales simulation software was used for fitting experimental data, to find out suitable electrical circuit schema.

Equation (2) demonstrates a Warburg diffusion coefficient D , where n is exchange electron number, ν is reaction order, F is Faraday constant, R is Boltzman gas constant, A is electrode surface area, T is absolute temperature, C is oxidant/reductant concentration on the electrode material ($C=1$) and Warburg constant σ obtained by fitting [19].

$$D = \frac{\nu R^2 T^2}{n^4 F^4 A^2 C^2 \sigma^2} \quad (2)$$

3 Results and discussion

3.1 Thermogravimetric analysis

Firstly, the thermogravimetric diagrams (Figure 1) illustrated that the physical dehydration of PANi-TiO₂ at a temperature of 200°C was about 7 wt%, higher than that of PANi-TiO₂-CNTs (5–6 wt%) at the same temperature. Decomposition temperature was 293°C for PANi-TiO₂, less than that of PANi-TiO₂-CNTs which was about 12–15°C. However, no difference was found at CNTs content of 10 wt% and 20 wt% (Table 1). The weight of PANi-TiO₂ was lost at 800°C about 4.4% and 10% respectively more than that of PANi-TiO₂-CNTs in case of CNTs content of 10–20 wt% and 30%. Generally, the thermogravimetric analysis behavior was similar for all CNT%, because they are not thermally degraded, indicating they do not cause any

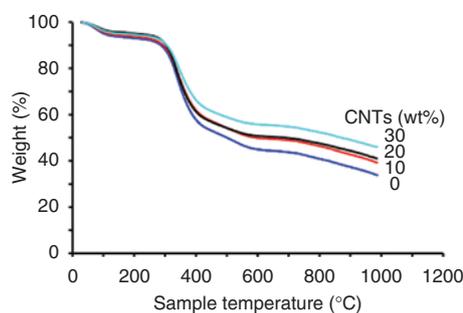


Figure 1: The influence of carbon nanotubes (CNTs) on the thermogravimetric plots of materials.

particular thermal behavior. The more CNTs content, the less organic material to degrade. This is explained by the fact that PANi-TiO₂-CNTs was more thermally stable than PANi-TiO₂ owing to less PANi degraded by CNTs intercalation into material matrix.

3.2 Electrical conductivity measurement

Figure 2A shows the typical current-voltage curves for an ohmic material of PANi-TiO₂-CNTs composites with CNTs of 0%, 10%, 20%, and 30%. The result showed that current density increased when CNTs content increased. The conductivity of materials was obtained from these diagrams according to Eq. (1) and calculated data are given in Figure 2B. The electrical conductivity of composites lightly increased from 46.5 mS/cm to 48.3 mS/cm with CNTs amount from 0% to 10%. Then, it increased to 69.8 mS/cm and 77.4 mS/cm with CNTs of 20% and 30%, respectively. This increase was due to high conductivity of CNTs which intercalated into the composite matrix. However, the non-linearity with CNTs amount is probably due to different percolation pathways depending on CNT percent. Phase segregation probably occurs at various degrees depending on CNT%, which does not, however, appear on SEM images. The biggest increasing slope in the curve was found by CNTs from 10% to 20%.

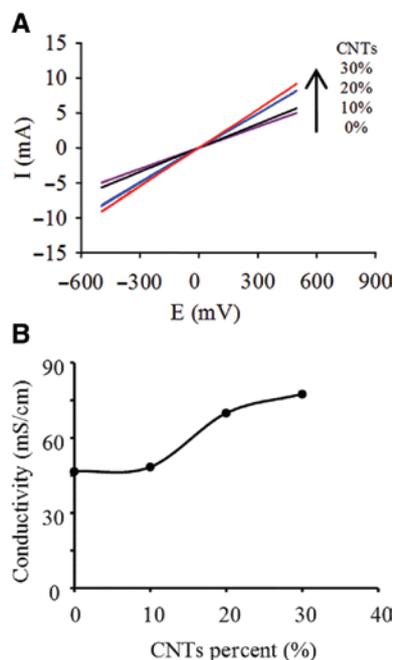


Figure 2: The influence of carbon nanotubes (CNTs) on current-voltage curves for an ohmic material of polyaniline titanium dioxide (PANi-TiO₂-CNTs) composites (A) and their electrical conductivities (B). Scan rate: 100 mV/s.

3.3 EIS study

The impedance measurements were focused to find the electrochemical mechanism occurring on the surface of composite electrodes in brewery wastewater through simulation software of Thales. Figure 3 presents Nyquist plots measured at open circuit potential, where measuring points were symbols and fitting lines were solid ones. It showed that the fitting lines were coincided with measuring data. Two electrical equivalent circuits (EEC) were found in Figure 4, and among them the first one (Figure 4A) illustrated six elements belonging to electrodes containing different CNTs content and the second one (Figure 4B) showed seven elements belonging to those without CNTs intercalation. Table 2 provides data of electrochemical impedance parameters obtained from

Table 1: Thermogravimetric analysis (TGA) data of composites with carbon nanotubes (CNTs) content varied from 0 wt% to 30 wt%.

CNTs content (wt%)	Physical dehydration (%)	Decomposition temperature (°C)	Weight loss of material at 800°C (%)	Weight loss of material at 1000°C (%)
0	7.0	293	51.90	59.20
10	6.0	305	47.47	54.70
20	5.0	305	47.55	54.19
30	5.4	308	42.25	48.77

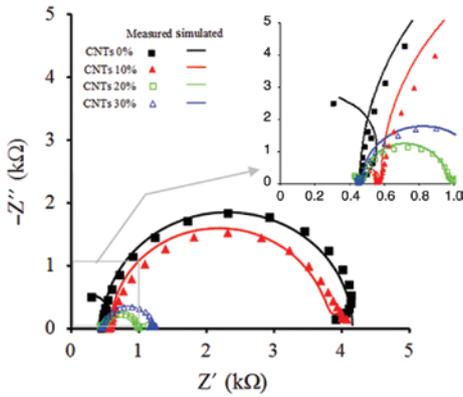


Figure 3: The influence of carbon nanotubes (CNTs) on Nyquist plots of materials in substrate electrolyte with chemical oxygen demand (COD) concentration of 3555 mg/l.

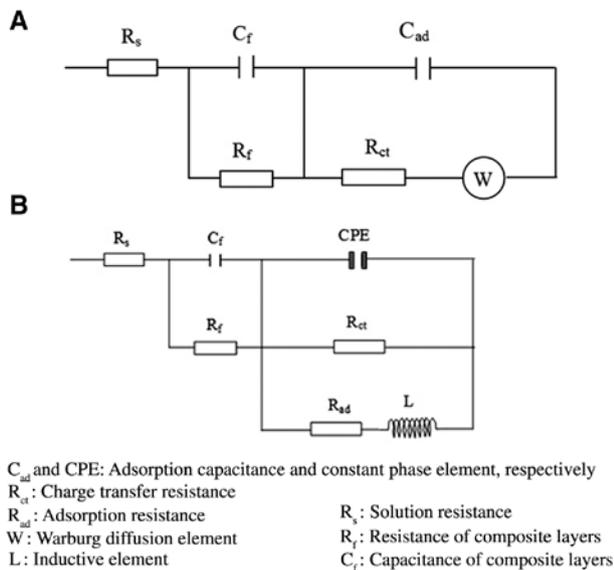


Figure 4: The electrical schema of polyaniline titanium dioxide carbon nanotubes (PANi-TiO₂-CNTs) (A) and PANi-TiO₂ (B) simulated from Figure 3.

simulation. An important contribution of CNTs intercalation was its effect on capacitance (C_f), resistance (R_f) and electrochemical process because of adsorption

capacitance (C_{ad}) and Warburg diffusion (W) appeared in EEC (A). R_f ranged from 137 Ω to 200 Ω and C_f from 4.0 nF to 4.8 nF for PANi-TiO₂-CNTs. Both of them were significantly smaller than that for PANi-TiO₂ (3.701 k Ω and 50.84 μ F, respectively) because no adsorption resistance was found in EEC (A). This is explained by the fact that an advantage for the electrochemical process occurred on the electrode surface owing to the CNTs intercalation into material matrix. Conversely, an inhibition of electrochemical reactions on the surface of composite without CNTs can be evaluated owing to appearance of adsorption resistance (R_{ad} = 19.98 Ω) that was found on EEC (B). An inductance element was also observed that probably indicated a pseudo-inductive electrochemical process assigning a relaxation effect, which affects the conductivity.

The given data for diffusion element probably caused by bacteria and organic species from brewery wastewater used as substrate electrolyte in this study, forming a biofilm on the electrode surface, rang from 10^{-15} cm²/s to 10^{-16} cm²/s, indicating that the diffusion process was low, but, it seems to be faster than that reported in [20]. The fastest one was found for the composite containing 20% CNTs owing to the biggest D (3.23×10^{-15} cm²/s) caused by the smallest charge transfer resistance (R_{ct}) (495 Ω).

3.4 CV study

The CV diagrams of composites in Figure 5 contain different CNTs content and show the increase of responding current when CNTs amount was bigger than 10 wt%; among them, the highest one obtained was in the case of 20 wt% CNTs. The higher the responding current in the CV diagram is, the better electrochemical behavior the material obtains. This is explained by the fact that the composite with 20% CNTs has the best electrochemical activity and is useful for an electrocatalytic process, which will be considered in the next paragraph.

Table 2: Electrochemical parameters corresponding to Figure 3 calculated by electrical schema displayed in Figure 4.

CNTs (%)	R_s (Ω)	C_f (nF)	R_f (Ω)	CPE (nF)	C_{ad} (μ F)	R_{ct} (k Ω)	R_{ad} (Ω)	W		L (TH)
								σ ($\Omega \cdot s^{-1/2}$)	D (cm ² /s)	
0	0.3	50840	3701.0	0.3	–	0.459	19.98	–	–	901
10	379.5	4.323	199.9	–	51.70	3.169	–	79.60	1.39×10^{-16}	–
20	313.9	4.759	154.5	–	40.00	0.495	–	16.54	3.23×10^{-15}	–
30	328.3	4.022	137.0	–	49.86	0.710	–	24.32	1.49×10^{-15}	–

C_{ad} , Adsorption capacitance; C_f , capacitance; CNTs, carbon nanotubes; CPE, constant phase element; D, diffusion coefficient; L, inductive element; R_{ad} , adsorption resistance; R_{ct} , charge transfer resistance; R_f , resistance; R_s , solution resistance; TH, terahenry; W, Warburg diffusion element.

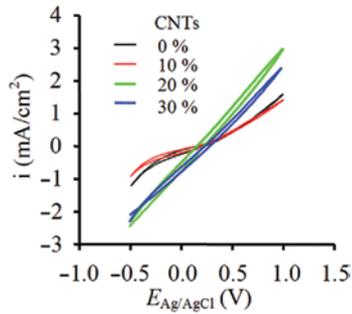


Figure 5: Effect of carbon nanotubes (CNTs) content on voltammograms of materials in substrate electrolyte with chemical oxygen demand (COD) concentration of 3555 mg/l at scan rate of 20 mV/s.

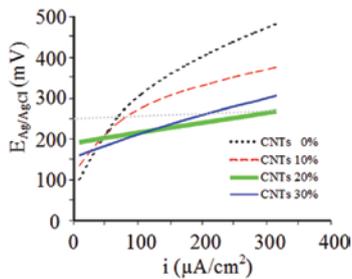


Figure 6: Effect of carbon nanotubes (CNTs) content on galvanodynamic polarization of materials in substrate electrolyte with chemical oxygen demand (COD) concentration of 3555 mg/l at scan rate of 5 μ A/s.

3.5 Galvanodynamic polarization

Galvanodynamic polarization is a characterization for considering the effect of electrode materials on their electrocatalytic activity and performance, especially with microbial fermentation products [21] such as brewery wastewater, used as an excellent substrate electrolyte for MFC [13]. The

higher the current density at the same potential observation reaches, the better electrocatalytic oxidation activity and performance the material has. It is demonstrated in Figure 6 that a different current density was observed at a response potential of 250 mV versus silver/silver chloride reference electrode, because of changing CNTs content. Composite with 20 wt% CNTs had a better performance than the other ones because it reached a higher current density (300 μ A/cm²) than the rest one (up to 200 μ A/cm²). This meant that the exhibition of this performance depended not only on the electrical conductivity of material, but also on suitable CNTs amount (20 wt%) in the composite that obtained the best electrochemical property through EIS and CV studies mentioned above.

3.6 Morphological structure

The SEM images in Figure 7 show the existence of titanium dioxide in grain with size of about 20 nm (Figure 7A), while CNTs had the fiber form with diameter of about 100 nm (Figure 7B). The PANi-TiO₂-CNTs composites existed in fiber (Figure 7C–E) with uniform diameter which was bigger than that of CNTs alone. This meant that CNTs were covered by a thin PANi film. The SEM images are in agreement with the electrical conductivity mentioned above, that the thinner the PANi layer on the CNTs covers, the higher the electrical conductivity the composite obtains.

The composite with 30 wt% CNTs was chosen for taking TEM images which existed in different colors (Figure 8). The significantly light color belongs to PANi enclosing both of the big dark one owing to CNTs and the small dark one due to TiO₂. It was found that their size was in the nano range. Both SEM and TEM images showed evidence of successfully

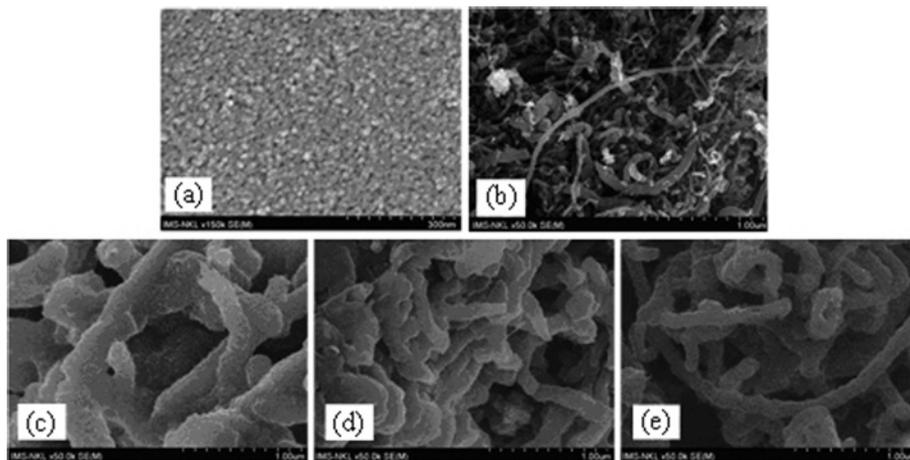


Figure 7: Scanning electron microscope (SEM) images of regarded materials: (A) titanium dioxide (TiO₂), (B) carbon nanotubes (CNTs), (C, D, E) composite with 10 wt%, 20 wt% and 30 wt% CNTs.

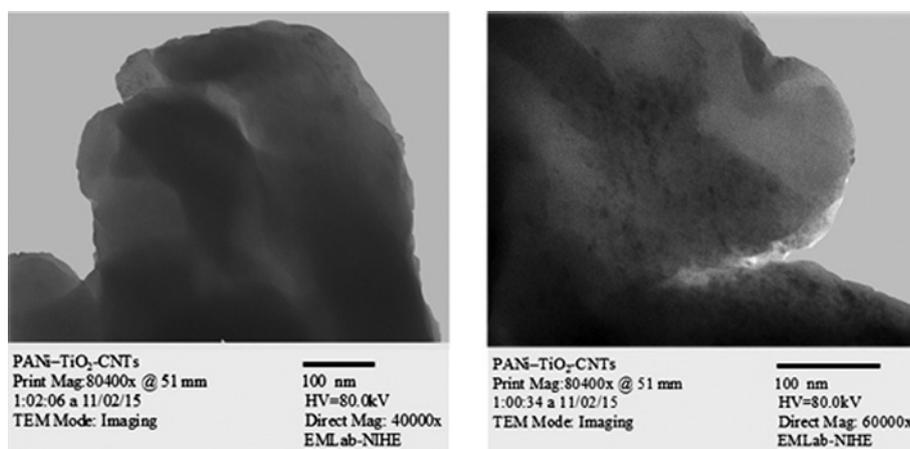


Figure 8: Transmission electron microscope (TEM) images of polyaniline titanium dioxide carbon nanotubes (PANi-TiO₂-CNTs) composite with 30 wt% CNTs.

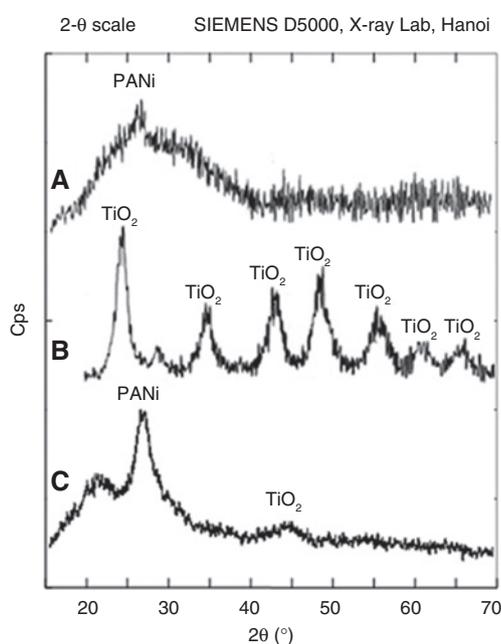


Figure 9: X-ray diffraction pattern of polyaniline (PANi) (A), titanium dioxide (TiO₂) (B), PANi-TiO₂-carbon nanotubes (CNTs) (30 wt%) (C).

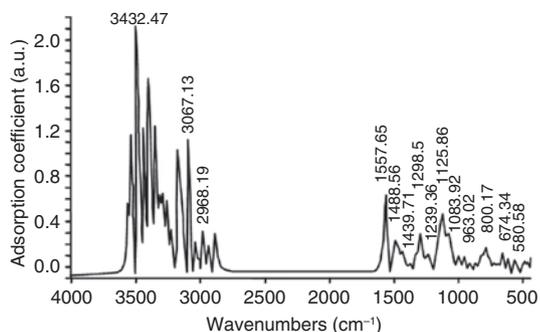


Figure 10: IR spectrum of polyaniline titanium dioxide carbon nanotubes (PANi-TiO₂-CNTs) composite.

Table 3: Vibration signals on IR spectrum of polyaniline titanium dioxide carbon nanotubes (PANi-TiO₂-CNTs) composite.

Wave number ν (cm ⁻¹)	Binding
3462	$\nu_{\text{N-H}}$
3067, 2968	$\nu_{\text{C-H aromatic}}$
1557	$\nu_{\text{C=C}}$, Benzoid
1488, 1439	Quinoid
1298, 1239	-N=quinoid=N-
1125	C-N ⁺ group

obtaining nanostructural PANi-TiO₂-CNTs composites by a chemical method.

3.7 Chemical structure

The chemical structure of materials was demonstrated by the X-ray spectra (Figure 9). The results showed the presence of two characteristic peaks of anatase TiO₂ at 2θ of 27° and 44°. In addition an amorphous part of PANi is presented.

There were two vibration signals found in Figure 10, at 1557 cm⁻¹ and 1488 cm⁻¹, belonging to benzoid and quinoid rings of PANi, respectively [22]. Additionally, there were other signals illustrating N-H stretching mode at 3462 cm⁻¹, -N=quinoid=N- stretching one at 1298 cm⁻¹ and 1239 cm⁻¹, C-N⁺ group at 1125 cm⁻¹, and C-H group at 3067 cm⁻¹ and 2968 cm⁻¹ (Table 3).

4 Conclusions

The nanostructured PANi-TiO₂-CNTs composite was successfully synthesized by a chemical method. The presence

of CNTs in the composite acts an important role in the electrochemical mechanism on which no adsorption resistance was found. The electrochemical impedance of the composite can be improved owing to the presence of CNTs intercalated into its matrix; among them, the case of 20% CNTs exhibits the highest responding current on the CV diagram. Its performance depended significantly on intercalated CNTs content into materials and the best one corresponding to current density of 300 $\mu\text{A}/\text{cm}^2$ at 250 mV versus silver/silver chloride reference electrode by galvanodynamic scanning belonged to that which contained 20 wt% CNTs.

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Bionotes



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