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Preparation of High Performance Conductive Polyaniline Magnetite ($PANI/Fe_3O_4$) Nanocomposites by Sol-Gel Method

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The conductivities of polyaniline magnetite ($PANI/Fe_3O_4$) nanocomposites prepared by sol-gel method were measured by standard van der Pauw DC 4-point probe method. $PANI/Fe_3O_4$ conductivity was measured as a function of wt % (5, 10, 15, 20 and 25 wt %) of Fe_3O_4 nanoparticles. It was observed that the conductivity of polyaniline containing certain percentage of Fe_3O_4 nanoparticles is slightly lower than the bulk PANI nanotubes and drastically decreases with increase of wt % Fe_3O_4 nanoparticles. The high conductivities of $PANI/Fe_3O_4$ nanocomposites was observed due to high concentration of dopant (oxidants) used in the polymerization process and the optimization of these composites allows this being use as a parameter for the production of nanofibers. Fourier transform infrared spectra, field emission scanning electron microscope, X-ray diffraction and ultraviolet-visible absorption spectra are used to characterize the phase structure, morphologies and functional group of the $PANI/Fe_3O_4$ composites samples. Fourier transform infrared analysis indicates the presence of PANI containing Fe_3O_4 nanoparticles and the field emission scanning electron microscope (FESEM) results has proven that the formation of nanofibers in the $PANI/Fe_3O_4$ nanocomposites. The crystalline phase of $PANI/Fe_3O_4$ nanocomposites studied by X-ray diffraction indicated that the Fe_3O_4 nanoparticles was present in the PANI matrices.

Keywords: Polyaniline, Fe_3O_4 nanoparticles, Composites, Conductivity, FESEM.

INTRODUCTION

In the last two decade, there is an extensive increase in demand of nanocomposites materials; such materials with a unique combination of physical and chemical properties are now available [1,2]. The nanocomposite based on conducting polymers is one of the major experimental research areas due to their flexible electrical conductivity as a result of doping [3-5]. Nowadays, a number of metal or metal oxides are blended into the conducting polymers to form composites. Once the metal nanoparticles with certain magnetic properties are incorporated into electrical conducting polymers formed a novel material that has electric and magnetic properties [6,7]. Electroactive polymers are probably essential as the matrix for hosting metals nanoparticles because it served as the interface for easy transfer of charge. However, the optical properties of these materials drastically change from the corresponding bulk material [8-10].

Among these electroactive polymers, polyanline (PANI) gains much attention in recent time, due to its environmental stability, easy control electrical conductivity and interesting redox properties [11,12]. Polyanline is suited for covalent bond of the molecule due to its stable nature of optical and electrical properties and easy processibility. Moreover, the electrical properties can significantly be enhanced *via* doping. Polyanline has three possible oxidation states this include leucoemeraldine base (fully reduced), emeraldine (partly oxidized) and pernigraniline (fully oxidized). Among them, only protonated emeraldine is electrically conductive, while, during doping process leucoemeraldine and pernigraniline shows poor conductivity. Therefore, PANI is thermally, electrochemically and environmentally stable in both air and solution. Now blending PANI with certain materials that have magnetic properties can form composites with suitable electric and magnetic properties [13,14].

Some quantity of inorganic nanoparticles with good magnetic properties are added into PANI matrices to form a composites that has electric and magnetic properties, among them, Fe_3O_4 nanoparticles have been selected because it has potential application in making soft magnetic material, magnetic recorder, magnetic colour imaging and drugs delivery [15,16]. Polyanline having conductive layer blending it with Fe_3O_4 nanoparticles that has magnetic layer formed a composites with novel electric and magnetic properties. These properties are observed because of the chemical interaction of inorganic oxide macromolecule surface and physical interaction of organic materials [12]. PANI/Fe_3O_4/Au composites were synthesized *via* suitable ultrasonic irradiation method, the analysis results show that the particles are quite very small with efficient electric and magnetic properties suitable for sensing application [17].

Recently, *in situ* chemical polymerization method were employed to synthesized Fe_3O_4/PANI and the samples were characterized by FESEM, vibrating sample magnetometer (VSM), 4-point probe and FTIR spectra, the analysis result shows a very good electric and magnetic properties used for electrochemical sensor. Therefore, this is the most efficient method for direct polymerization of aniline monomer in aqueous solution in the presence of dissolved iron oxide nanoparticles [18].

PANI/Fe_3O_4 nanocomposites were prepared by *in situ* chemicals polymerization methods in the aqueous solution of aniline monomer contain certain percentage of Fe_3O_4 nanoparticles as a materials [19]. Incorporating inorganic components into PANI has found to be suitable techniques for obtaining nanocomposites that has a wider range of application in science and engineering.

In this research, PANI/Fe_3O_4 was synthesized using a suitable techniques that usually allows synthesis of PANI/Fe_3O_4 nanocomposites by sol-gel method in the aqueous solution of aniline monomer, ammonium peroxydisulfate (APS), hydrochloric acid and certain percentage of aniline dimer-COOH capped in Fe_3O_4 nanoparticles. The polymerization aniline was started by adding the oxidant, the synthesized Fe_3O_4 nanoparticles was deposited on the surface of PANI matrices. The molecular structure of the PANI/Fe_3O_4 nanocomposites was characterized by Fourier transformation infrared spectra (FITR), Ultraviolet-visible absorption spectra, field emission scanning electron microscope (FESEM). The result shows that PANI containing 5, 15 and 25 wt % Fe_3O_4 nanoparticles have similar morphologies. The conductivity of the composites was measured by 4-point probe method, it was observed the conductivity of PANI nanotubes contain 5, 15 and 25 wt % of Fe_3O_4 nanoparticles are almost similar.

EXPERIMENTAL

All the chemicals were purchased from the Sigma Aldrich and used without further purification. This includes aniline monomer, hydrochloric acid, ammonium peroxydisulfate [$(NH_4)_2S_2O_8$], succinic anhydride, N-phenyl-1,4-phenylenediamine, diethyl ether, ethylene glycol, acetone, iron(III) chloride, iron(II) chloride, dichloromethane, ammonium hydroxide, methanol and ethanol.

Synthesis of aniline dimer-COOH: The reaction was performed in the 200 mL necked round bottom flasks under magnetic stirrer. 4.5 g of succinic anhydride and 0.821 g of N-phenyl-1,4-phenylenediamine was dissolved into a 25 mL of dichloromethane under magnetic stirrer for 5 h. When the reaction rich certain period of time a white precipitate were obtained, at the end of the reaction. The precipitate was filtered and washed with diethyl ether and dried in the vacuum for 12 h.

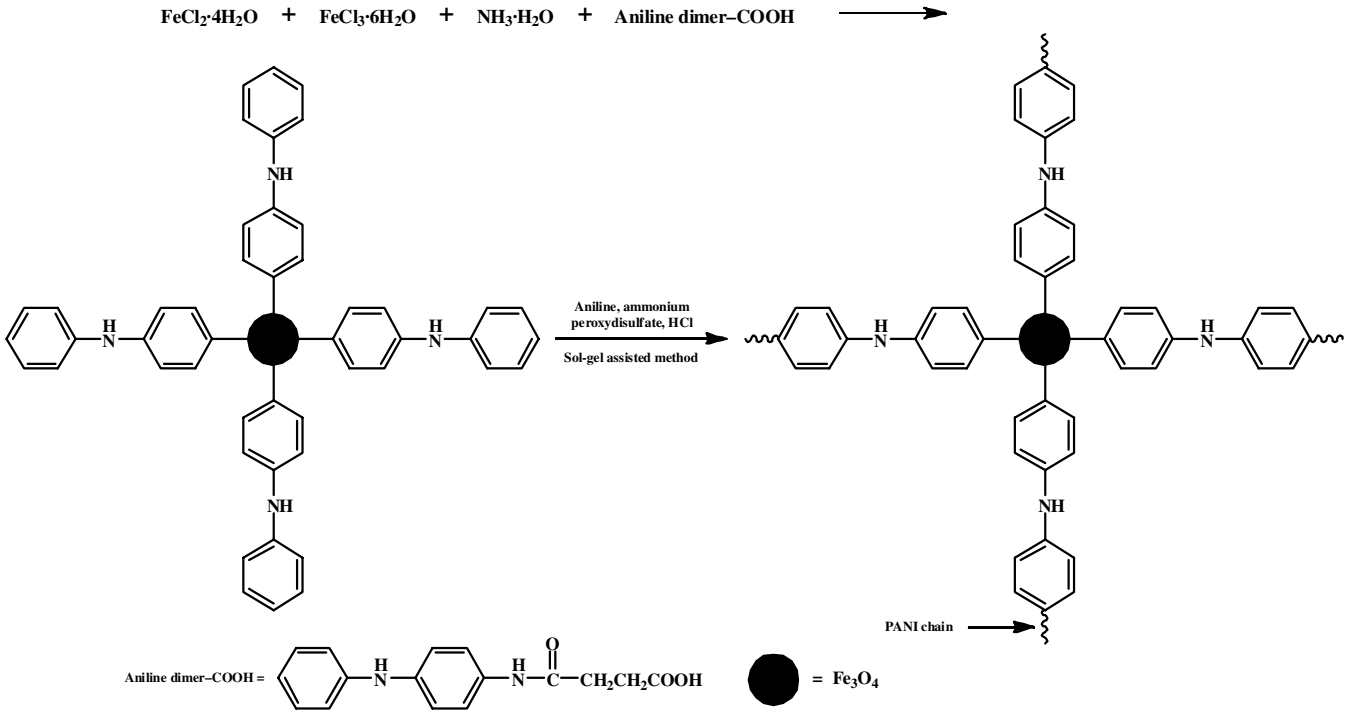
Preparation of magnetite (Fe_3O_4) nanoparticles: The magnetite nanoparticles was prepared as followed 4.7 g of ferric(III) chloride ($FeCl_3 \cdot 6H_2O$) and 1.72 g of ferrous(II) chloride ($FeCl_2 \cdot 4H_2O$) was dissolved in 60 mL deionized distilled water under vigorous magnetic stirrer. As the reaction was heated to a certain temperature of 60 °C, 10 mL of ammonium was added. Subsequently, 400 mg of aniline dimer-COOH 4 mL of acetone was also added into the above reaction and allowed proceed for 1.5 h at 90 °C under magnetic stirrer to produce a stable water base suspension. When the reaction cooled to room temperature, the sample was filter, washed and dried in the oven for 6 h.

Preparation of PANI/Fe_3O_4 nanocomposites: Polyaniline contains different wt % of Fe_3O_4 nanoparticles was prepared by sol-gel method. In a general preparation method, 0.4 mL aniline monomer was mixed with 10 mL of HCl. Then, 5 to 20 wt % of Fe_3O_4 nanoparticles was dissolved in 60 mL of ethylene glycol under magnetic stirring for 30 min at 50 °C to obtained sol. Subsequently, 10 mL aqueous solution of ammonium peroxydisulfate was added to the above mixture to form PANI/Fe_3O_4 nanocomposites. This reaction mixture was kept under magnetic stirring for 5 h at certain temperature range from 80 to 90 °C until gel was formed. The obtained gel was age for 24 h and later deposited on glass for certain analysis. The preparation of Fe_3O_4 nanoparticles capped aniline dimer-COOH, growth on the surface of PANI nanotubes with assisted sol-gel method is shown in **Scheme-I**.

Measurement: Fourier transformation infrared spectrum (FTIR) (Perkin Elmer Spectrum 100 FTIR Spectrometer) are used to observe the molecular structures of polyaniline magnetite (PANI/Fe_3O_4) nanocomposites. The crystalline phase structure of the PANI/Fe_3O_4 were examined by X-ray diffraction spectrum with a current and voltage radiating at 40 mA and 45 Kv, respectively at a Kα radiation wavelength of (λ = 0.154 nm) with phase diffraction angle (2θ) in range of 20 to 90°. The morphologies of PANI nanotubes contain different level of Fe_3O_4 nanoparticles were investigated by field scanning electron microscopes (FESEM) (JEOL JSM-7600F). The ultraviolet-visible absorption spectra were measured using (SHIMDZU 1800 UV-visible series). The conductivity of PANI/Fe_3O_4 nanocomposites was measured by standard van der Pauw DC Four-probe method, PANI nanotube containing Fe_3O_4 nanoparticles was deposited on the glass and place in the 4-point probe apparatus. The voltage was provided and the corresponding electric current was obtained. Therefore, these relations could be used to obtain the electrical conductivity of a particular sample:

$$\delta\ (s/m) = (2.44 \times 10/S) \times (I/E) \quad (1)$$

where δ is the conductivity, S is the sample side area, I is the current passing through the outer probe, E is the voltage across the outer probe [13].



Scheme-I: Schematic representation of preparation PANI/Fe_3O_4 nanocomposites by sol-gel method

RESULTS AND DISCUSSION

The FTIR spectra were used to estimate the molecular structures of PANI/Fe_3O_4 nanocomposites. Fig. 1 shows the characteristic peaks of PANI containing five different levels of Fe_3O_4 nanoparticles. The peak of the pure Fe_3O_4 nanoparticles appear at 475 cm^{-1} and 694 cm^{-1} is assigned to the Fe-O stretching vibration [13,14]. It is equally observed that the characteristic peak of PANI/F_3O_4 nanocomposites appeared at 3322 cm^{-1} (N-H stretching), 1517 cm^{-1}, 1545 cm^{-1} (C=C vibration stretching and deformation of benzenoid ring and quinoid respectively) [20,21]. At peak 1308 cm^{-1} represent for (C-N vibrating stretching of aromatic secondary amine), 1118 cm^{-1} as well as 900-800 cm^{-1} assigned for (C-N are out of plane deformation in 1,4-distributed benzene ring) which signified the presence of PANI contain Fe_3O_4 nanoparticles [13,22].

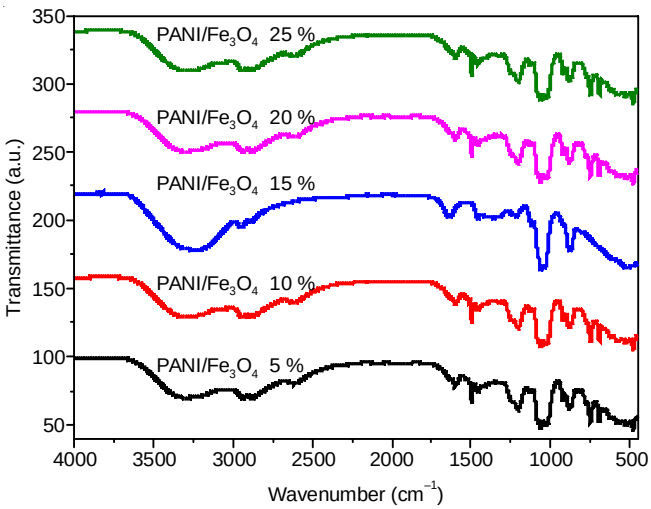


Fig. 1. FTIR spectra of PANI/Fe_3O_4 nanocomposites

However, blending of Fe_3O_4 nanoparticles leads to the shift of many IR bands in the PANI matrix. This can be assigned to fact that some interaction of PANI and Fe_3O_4 nanoparticles channelled to the formation of H-banding between the proton within N-H and oxygen atom on the Fe_3O_4 surface [23].

The structural pattern of the PANI/Fe_3O_4 nanocomposites is obtained at the peak 2θ = 11.26°, 27.3°, 32.43°, 35.45°, 61.26°, 63.05° and 68.08°. The obtained results are in agreement with what was found in the synthesized Fe_3O_4 reported [24]. The broad peaks at 2θ = 20-30° in the XRD analysis curve of PANI indicated the prepared PANI without Fe_3O_4 nanoparticles is amorphous [23,25]. Nevertheless, the major peaks of PANI/Fe_3O_4 nanocomposites are similar those in Fe_3O_4 nanoparticles [26,27].

From Scherer's equations:

$$D = \frac{K \cdot \lambda}{\beta \cdot \cos\theta} \quad (2)$$

The crystalline size (D) of the particles are estimated where the wavelength λ for X-ray is 0.154 nm, the shape factor k has a value of 0.89 if the shape is unknown, the average diameter of the crystal D in Å, α is the Bragg's angle in degree and β is the help-height of angle in radian. The broad peak at 2θ = 35.45° is chosen to estimate the crystalline size. The mean size of Fe_3O_4 nanoparticles is found to be 12 nm.

The morphologies of polyaniline magnetite (PANI/Fe_3O_4) nanocomposites are shown in Fig. 2. The composites morphologies are measured as a function of wt % of Fe_3O_4 nanoparticles. PANI nanotubes contain 5, 15, 25 wt % of Fe_3O_4 nanoparticles are measured by field emission scanning electron microscope (FESEM), the image (Fig. 3) shows both the samples posses the same morphologies of the nanofibers. It was observed that the diameter is similar in all the samples.

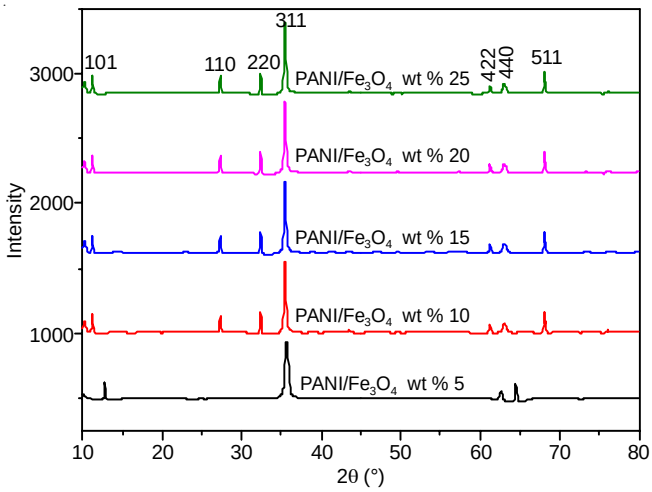


Fig. 2. XRD image of PANI/Fe_3O_4 nanocomposites

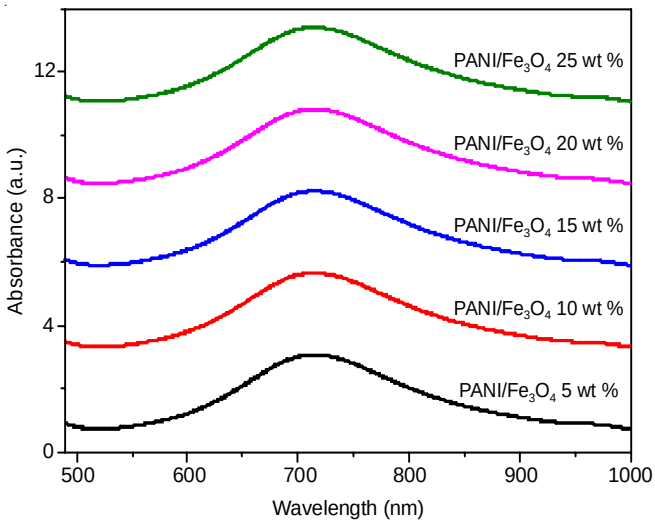


Fig. 4. UV-visible analysis of PANI/Fe_3O_4 nanocomposites

The spectra of resulting PANI/Fe_3O_4 nanocomposites dispersed in water were measured by UV-visible so as to assign the essential bands. For this, we estimated the UV-visible spectra of the pure PANI and PANI/Fe_3O_4 nanocomposites samples, which were prepared *via* sol-gel method. It was observed that the characteristic peaks of PANI/Fe_3O_4 nanocomposites appear at about 700-900 nm (π-polaron-transition) (Fig. 4). However, similar result was reported [28,29] on PANI/Fe_3O_4 composites, it was also observed that increased of Fe_3O_4 contain in the PANI matrices the UV-visible absorption of the π-polaron transition is at high wave length 700-900 nm (this indicate that there is significant interaction between PANI and Fe_3O_4 nanoparticles dispersed with assisted sol-gel method).

The conductivity of pure PANI nanotubes and PANI/Fe_3O_4 nanocomposites prepared by sol-gel method were measured by standard four-point probe apparatus. Fe_3O_4 nanoparticles present in the PANI matrix drastically reduce the PANI's conductivity. Table-1 shows the conductivity of PANI containing certain level of Fe_3O_4 nanoparticles. The conductivity of polyaniline (PANI) nanotubes is found to be 6018×10^{-1} S/cm, while the PANI/Fe_3O_4 nanocomposites (5, 10, 15, 20 and 25 wt % of Fe_3O_4 nanoparticles added to PANI nanotubes) indicated the conductivity values are found to be 3.81×10^{-3}, 3.25×10^{-4}, 2.4×10^{-2}, 2.30×10^{-3} and 1.84×10^{-2} S/cm, respectively, which is quite similar with reported literature [29,30]. The conductivity of PANI/Fe_3O_4 nanocomposites is decrease with increasing of Fe_3O_4 nanoparticles concentration, which can easily be attributed to the insulating behaviour of Fe_3O_4 nanoparticles in the charge transfer thereby lowering the conductivity. It is observed that the conductivities of PANI containing 5, 15 and 25 wt % of Fe_3O_4 nanoparticles are almost similar. This may result due to lowering of the concentration of doping in the bulk PANI matrix [31,32].

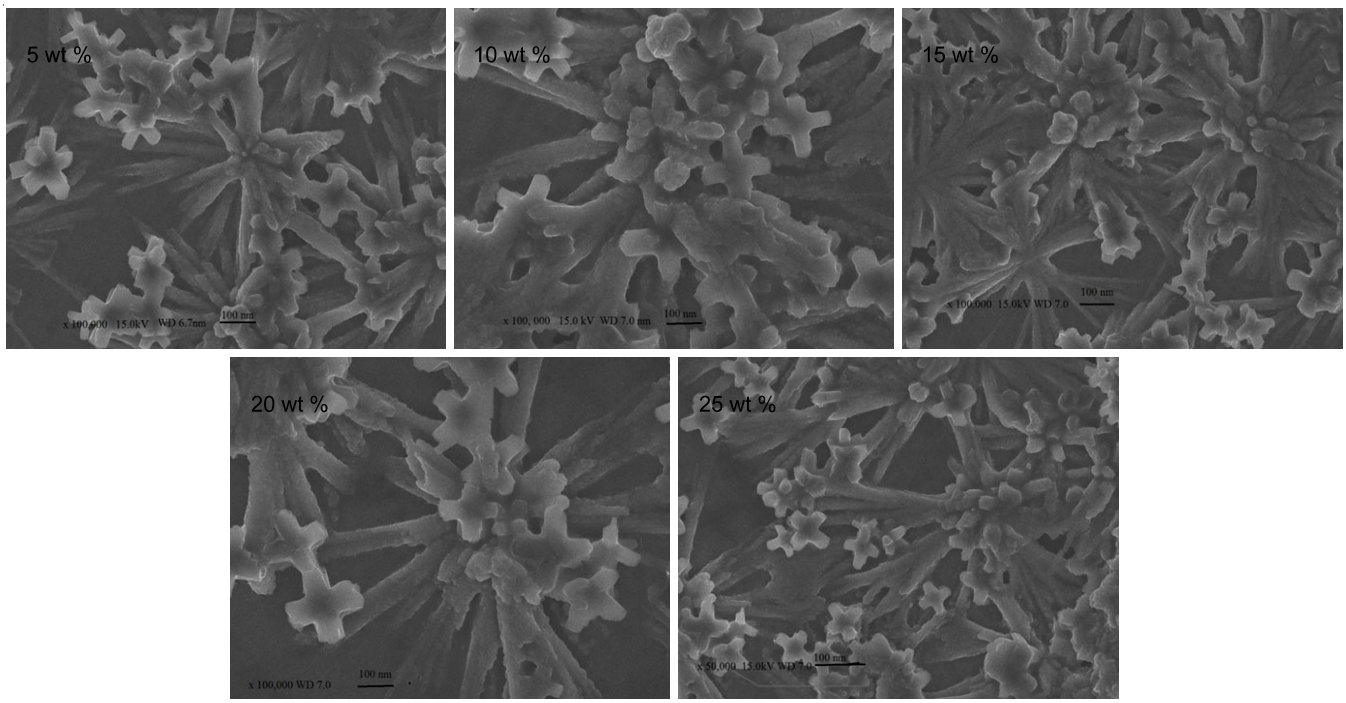


Fig. 3. FESEM image of PANI nanotubes containing 5, 10, 15, 20 and 25 wt % of Fe_3O_4 nanoparticles

TABLE-1
CONDUCTIVITY OF PANI CONTAIN
DIFFERENT LEVEL OF Fe_3O_4 NANOPARTICLES

Sample	Fe_3O_4 content (wt %)	Conductivity (s/cm)
PANI	–	6.32×10^{-1}
Fe_3O_4	–	–
PANI/Fe_3O_4	5	3.81×10^{-2}
PANI/Fe_3O_4	10	3.25×10^{-3}
PANI/Fe_3O_4	15	2.41×10^{-2}
PANI/Fe_3O_4	20	2.30×10^{-3}
PANI/Fe_3O_4	25	1.84×10^{-2}

In an effort to explicate good formation of PANI/Fe_3O_4 composites using sol-gel method, the aniline solution, ammonium peroxydisulfate, HCl and 5, 10, 15, 20 and 25 wt % of Fe_3O_4 nanoparticles were polymerized under magnetic stirring process and PANI/Fe_3O_4 nanotubes were observed with assisted sol-gel method. For this, we thought that it was attributed to the sol-gel techniques, but the mechanism of PANI nanofibers produce by interfacial polymerization were studied by Fayemi *et al.* [4] and PANI preferentially obtained as a nanofibers in the aqueous solution during chemical oxidation polymerization [33]. However, using sol-gel method secondary growth has been successfully prevented during early stage of polymerization, so that the nanofibers could be obtained. In our experiment, sol-gel techniques can effectively prevent large growth of the particles [29], hence the nanofibers were obtained. In the polymerization process of PANI nanotubes, Fe_3O_4 nanoparticles capped aniline dimer-COOH were bonded by the PANI matrices through polymerization graft, hence PANI/Fe_3O_4 nanocomposites were synthesized [29].

Conclusion

This study has demonstrated the preparation procedure of PANI/Fe_3O_4 nanocomposites, based on Fe_3O_4 nanoparticles with certain level of aniline dimer-COOH. The magnetite (Fe_3O_4) nanoparticles are added to aniline monomer and oxidant to growth the PANI chain on the surface of Fe_3O_4 nanoparticles, which lead to the formation of PANI/Fe_3O_4 nanocomposites. Through this technique secondary growth has been successfully prevented during polymerization. PANI/Fe_3O_4 nanocomposites with suitable electrical conductivity were successfully synthesized using chemical oxidative polymerization in the presence of Fe_3O_4 nanoparticles using sol-gel method. FESEM results confirmed that the magnetite nanoparticles were encapsulated in the PANI matrices and the conductivity of PANI nanotubes were measured as a function of Fe_3O_4 nanoparticles and observed that the conductivity decrease with increase of Fe_3O_4 nanoparticles. The structural properties and functional group of the composites were characterized by means of XRD, FTIR and UV-visible. Therefore, these composites was found to be conductive, hence it's very possible to be used as a conductive matrix for the development of nanofiber for sensing application.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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