

Fabrication and Characterization of Electrospun PANi/Fe₃O₄/PVA Nanofiber Composites for Telecommunication Engineering

Zakiyyu Ibrahim Takai¹, Mohd Kamarulzaki Mustafa^{2,3}, Farida Mustapha Umar¹, Abbas Umar Farouk¹, Aisha Idris⁴, Auwal Ibrahim Usman⁵, Najib Hamisu Umar⁶

¹Department of Physics Faculty of Science Yusuf Maitama Sule University, Kano, Nigeria

²Microelectronic and Nanotechnology-Shamsuddin Research Centre (Mint-SRC) Universiti Tun Hussein Onn Malaysia (UTHM)

³Department of Physics and Chemistry, Faculty of Applied Sciences and Technology, Universiti Tun Hussein Onn Malaysia, Pagoh Educational Hub, 86400, Muar, Johor, Malaysia.

⁴Department of Biological Science, Faculty of Science, Federal University Dutse, Jigawa State, Nigeria

⁵Department of Science National Infrastructure Agency for Science and Engineering Infrastructure

⁶Department of Engineering National Infrastructure Agency for Science and Engineering Infrastructure

*Corresponding author E-mail: zitakai@yumsuk.edu.ng

(Received 09 May 2024, Accepted 25 June 2024, Published 05 July 2024)

Abstract

The synthesis of conducting polymers has opened new possibilities for the research community for their applications towards microwave absorption in telecommunication engineering. Polyaniline has been one such conducting polymer whose synthesis does not require any special equipment or precautions. Such conductive polymer generally, show highly reversible redox behaviour with a noticeable chemical memory and hence have been considered as prominent new materials for the fabrication nanofibers that could be used in many applications particularly for telecommunication engineering. The current work presents a successful fabrication of Polyaniline / Magnetite / Polyvinyl alcohol (PANi/Fe₃O₄/PVA) nanofibers composites using electrospinning methods. Field Emission Scanning Electron Microscope (FESEM) was employed to investigate the composites nanofiber's morphology and TEM was also used to confirmed the present of Fe₃O₄ nanoparticles in the composites nanofiber, where the nanofibers diameter were investigated and found to be approximately 141 nm as estimated by image-J computer software analysis. Fourier-Transformation Infrared Spectroscopy (FTIR) study confirmed the presence of both Fe₃O₄ nanoparticles and PANi in the nanofiber structure. The X-ray diffraction (XRD) pattern of Fe₃O₄ nanoparticles and PANi/Fe₃O₄/PVA nanofiber materials shows the characteristic behaviour of both amorphous and crystalline phase by indication of the peaks at $2\theta = 23.12^\circ$, 24.14° and 35.13° in the prepared materials.

Keywords: PANI; Fe₃O₄ nanoparticles; PANi/Fe₃O₄/PVA nanofiber; Electrospinning technique.

INTRODUCTION

A variety of processing techniques have been explored to manufacture conducting polymer nanofibres materials in recent years [1]. The potential of conducting nanofibers are huge as more electronic devices can be incorporated within textile nanofibers to served for many application. The “smart textile or conducting nanofiber composites materials” term is referred to conducting nanofibers where the possible applications could be toward medical, sensors, communication and military applications. The current processing technique includes phase separation, hard templates, self-assembly, soft templates, interfacial polymerisation, seeding, rapidly mixing and electrospinning [2-4].

Electrospinning technique is found to be the most efficient method in producing suitable continuous polymers nanofiber textile materials with good fibers diameter range of 500 nm to 5 μm [5]. These fiber ranges of 500 nm to 5 μm are having large surface to volume ratio, are even potential for cell separation or membrane applications[6].

Electrospinning technique started by electrohydrodynamic process, where a liquid droplet is electrified to generate stream jet, followed by stretching and elongation of fibers. The parametric condition such as flow rate, tips to collector distance will significantly alter the nanofiber textile quality [7]. In general, the main parameters to be controlled in producing high quality nanofibers include types of polymers, surface tension of the liquid droplets, polymer and composite materials concentration, solution properties including viscosity, polarity and conductivity [8]. Further adjustment in refining Electrospinning condition include tips to collector distance, applied external voltage and droplets flow rate [9].

The continuous nanofiber textile production of conducting Polyaniline through aqueous solution using electrospinning was formerly consider as impossible because of the higher repulsive force between ionogenic groups and their capability to form intermolecular interaction [10-11]. PANi is regarded as one of the most extensively studied conducting polymers owing to its ease synthesis process and higher electrical conductivity[13]. New strategy in tuning PANi physical and chemical properties are through incorporating of doping materials. PANi can be doped with organic or inorganic dopants to increase its physical, chemical and mechanical properties [14].

Another strategy of changing Pani nanotubes properties is through the incorporation of inorganic nanomaterial and polymeric matrix such as poly (vinyl pyrrolidone) (PVP), polylactide (PLA), poly (vinyl alcohol) (PVA) and poly (ethylene oxide) (PEO) [15-16]. PVA is considered as the most suitable host for PANi/inorganic molecular matrix due to its higher heat resistance, wider surface area, thermoplasticity, low toxicity and low-dimensional nature [17-18]. In the current work PANi/Fe₃O₄/PVA nanofiber composites are prepared through electrospinning. The chemical, physical, morphology and optical property are analysed and investigated via FT-IR spectra, X-ray diffraction spectra (XRD), Field Emission Scanning Electron Microscope (FESEM), and Ultraviolet UV-visible (UV-vis). The image-J computer software was employed to investigate the nanofiber

composites diameter. Furthermore, based on the analysis the composites showed suitable properties that could be used for microwave absorption application.

Methodology

Preparation of Electrospinning Solution

The spinning solution was prepared as follows, PVA (10 % w/v) were dissolved in distilled water at 50 °C using magnetic stirrer overnight to obtain aqueous solution of PVA. A separate container of 15 % w/v of the prepared PANi/Fe₃O₄ was dispersed in DMSO solvent (15 ml) at room temperature using reflux for 0.5 h, and then transfer the solution of PANi/Fe₃O₄ in DMSO into the prepared PVA in order to achieve the homogeneous solution of PANi/PVA composites in DMSO. Magnetic stirrer is used throughout the solution process of PANi blended homogeneously with PVA solution to form PANi/Fe₃O₄/PVA nanocomposites solution that later electrospinning process was taken to produce nanofiber composite fibers.

Nanofiber production using Electrospinning techniques

The electrospinning set-up is used in the preparation of PANi/Fe₃O₄/PVA composite nanofiber, along the way, the PANi/Fe₃O₄/PVA composite solution (10 ml) was filled in the syringe the solution was pumped via the needle in the electrospinning set-up. The electrospinning machine was connected to the higher DC voltage. Therefore, the major syringe pump role is to monitor and control the constant motion (flow rate) of the polymer melt solutions. The interval between the tips to collector is considered as electrospinning distance. The electrospun nanofiber textile materials are later collected on aluminum foil. The electrospun nanofiber textile materials would be later dried under ambient conditions to remove the solvent as illustrated in Figure 1.

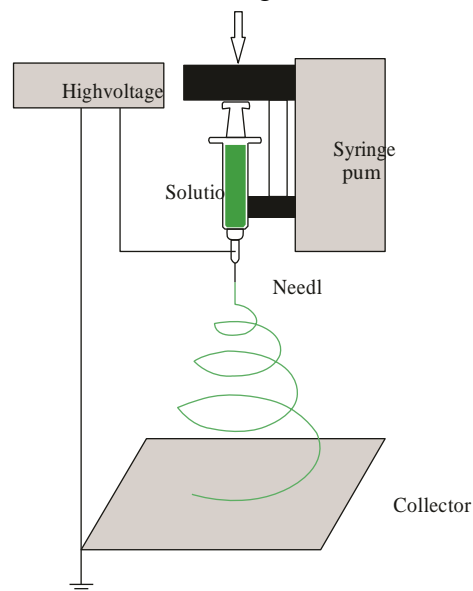


Figure 1: Schematic diagram of an electrospinning setup

Characterization

The Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA nanofiber composites textile structural pattern was investigated using Bruker (D8 FOCUS), X-ray diffraction pattern (XRD) with $\text{Cu K}\alpha$, and λ radiation wavelength. Moreover, the Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA composites nanofibers textile chemical bonding structure were analyzed by Fourier Transformation Infrared Spectra (FTIR) and The Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA absorption spectral analysis and PVA nanofibers textile composites were further study by means of Ultraviolet UV-visible (UV-vis). Furthermore, the internal structure and morphology of Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA nanofibers textile composites was analysed by means FESEM (XL-30) and image-J were used to study the average diameter of the PANi/PVA and poly vinyl alcohol nanofiber mixtures materials.

Results and Discussion

XRD analysis of Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA composites nanofibers

It was observed that most polymeric materials are not completely crystalline materials due to their chain arrangement which indicated that the polymers has two phase crystalline and amorphous phase [19]. Presence of amorphous phase revealed the existence of characteristic of amorphous pattern in several form, the amorphous phase of Fe_3O_4 nanoparticles and crystalline phase of PANi/ Fe_3O_4 /PVA nanofiber composites textile were study using deviation of x-ray spectrum analysis (XRD) the outcomes were revealed in Figure 2.

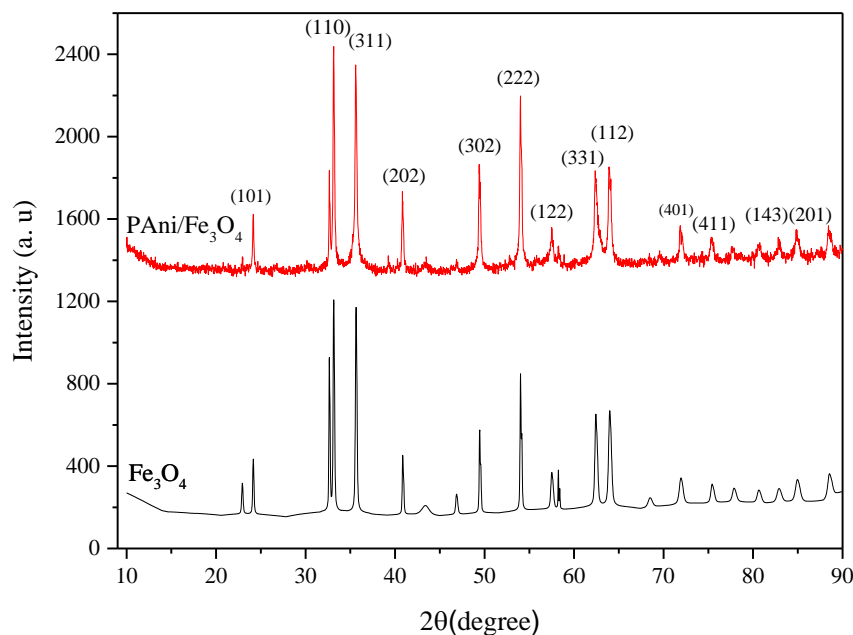


Figure 2: XRD study of Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA spun at 16 kV, 10 ml, 9 cm tips to collector distance and 0.35 ml/h.

Furthermore, Figure 2 indicates the presence of Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA composites nanofibers materials on X-ray diffractometry ranging from 10° - 90° . The structural phase were found to match with the joint committee on powder deviation standards file (JCPDS: 97-5869) to the phase pattern shown on each nanofiber composites materials. Furthermore, the XRD pattern of Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA nanofiber mixtures materials contain crystalline amorphous phase pattern with certain peaks appear at $2\theta = 23.12^\circ, 24.14^\circ, 35.13^\circ, 37.14^\circ, 41^\circ, 43^\circ, 53.3^\circ, 57.42^\circ, 64.3^\circ, 65.4^\circ, 73.1^\circ$ and 79.8° assigned to (101),(110), (311), (202), (302), (222), (122), (331), (112), (401), (501), (411), (143), and (201) which indicate the presence of PANi phase. Nevertheless, the peaks appear at $24.14^\circ, 35.13^\circ$ which is perpendicular to the PANi matrix and Fe_3O_4 respectively ascribed to the parallel periodicity [20].

FTIR analysis of Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA nanofibers composites

The FTIR spectra are one of the major investigative technique used to study the chemical bonding analysis in a materials. However, these instruments were utilized to analyse the prepared Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA nanofiber composites materials as well as determine the presence of functional group in the polymers materials. Furthermore, Figure 3 revealed that the infrared spectrum of the synthesized polyvinyl alcohol and polyaniline /polyvinyl alcohol nanofiber compounds materials electrospun at certain parameter which include high DC applied voltage tips to collector distance and flow rate respectively. However, the weak intensity peaks were demonstrated at $2918, 2942, 3284,$ and 3299 cm^{-1} assigned for vibrating and stretching of the O-H group found in the poly vinyl alcohol nanofiberous materials and vibration stretching of amine group found in mixtures materials of polyaniline/ poly vinyl alcohol nanofibers as shown in the Figure 2.

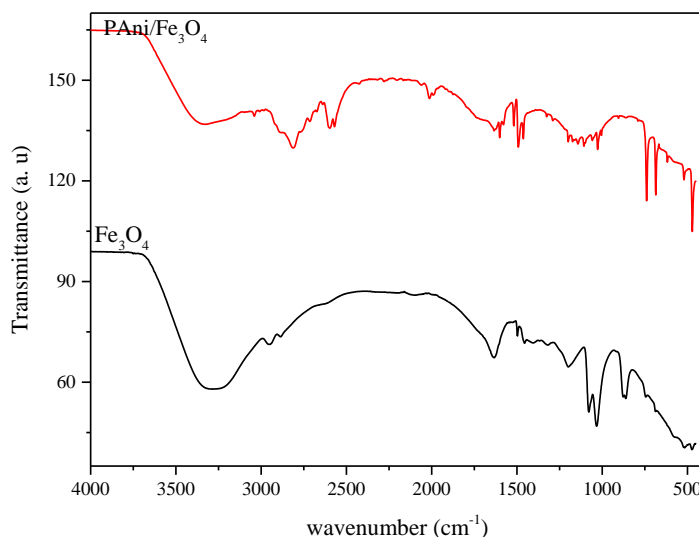


Figure 3: FTIR spectrum of Fe_3O_4 nanoparticles and PANi/ Fe_3O_4 /PVA nanofibers composites spun with 16 kV, 10 ml, 9 cm tips to collector distance and 0.35 ml/h.

The bands exist between 1000 to 1656 cm^{-1} , in the entire FTIR spectrum of the prepared PANi/Fe₃O₄/nanofibers composites with some weak bands for PVA nanofiber materials at 3000 to 1450 cm^{-1} are indeed due to of the alkane and amine groups, vibrations stretching which further revealed more bond at 481, 592 and 847 cm^{-1} that derivatives of Nitrogen, oxygen, carbon and hydrogen vibrating stretching [17, 18]. Additionally the peaks appear at 1401 to 1000 cm^{-1} indicated that C=O vibrating stretching and C-O symmetric within the nanofibers composites materials. Some peaks were further revealed at 581 cm^{-1} in the FTIR spectra of the prepared nanofiber composites, these bond are assigned to stretching vibration of F-O and C-H group that are usually presence in the PANi, PVA and Fe₃O₄ nanomaterials [19-20]. Several vibrating bond were found ranging from 1401 - 847 cm^{-1} which might be correspond to the CH₃ group stretching vibration that exists in the PANi and PVA nanotubes, this can simply revealed the absence of H₂O molecules in the composites [21].

UV-visible analysis of Fe₃O₄ nanoparticles and PANi/Fe₃O₄/PVA composites nanofibers

In the current research, the UV-visible spectroscopy was used to study the optical absorption analysis of the prepared Fe₃O₄ nanoparticles and PANi/Fe₃O₄/PVA nanofiber composites materials. The optical absorption spectroscopy result was recorded in range of 200-900 nm wavelength through shimadzu UV-1800 spectroscopy. However, the UV-vis spectrum of the prepared Fe₃O₄ nanoparticles and PANi/Fe₃O₄/PVA nanofiber composites electrospun at some parameter that include high DC voltage, tip to collector distance and flow rate, the fabricated nanofiber result was revealed in the Figure 4. The higher intense bond found at 670 nm correspond to π - π^* benzonoid ring transition. This shoulder-like structure revealed the peaks at 400-700 nm presence of emeraldine salts phase of the doped PANi and PVA in the composites materials [22-23].

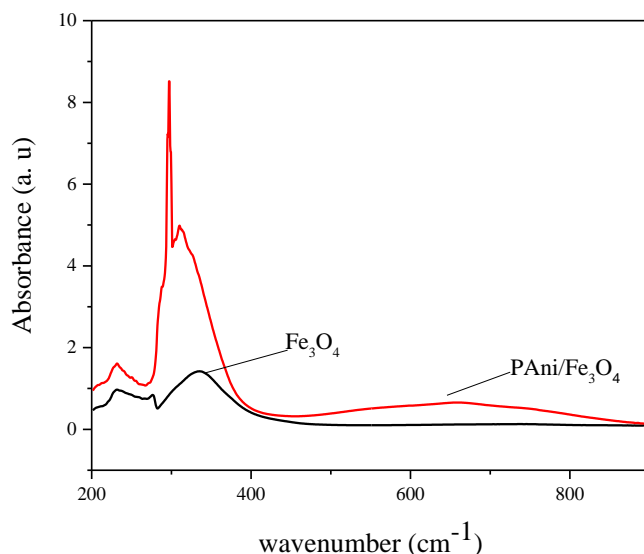


Figure 4: UV-vis analysis of Fe₃O₄ nanoparticles and PANi/Fe₃O₄/PVA nanofibers composites spun with 16 kV, 10 ml, 9 cm tips to collector distance and 0.35 ml/h.

Generally, the nanofiber composites materials with certain quantity of polyaniline revealed nearly the same peaks in the resulting UV-vis spectra 200-700 nm [24], this indeed is attributed to the higher component of poly vinyl alcohol within the nanofiber composites materials and interaction of photons of light with conducting electron in the PANi matrix which result in the high absorption, however the nanofiber composites are quite transparent in the visible region [25]. The structure found in the resulting spectra of the nanofiber indicate very high absorption (400-700 nm).

Surface morphology study of Fe₃O₄ nanoparticles and PANi/Fe₃O₄/PVA nanofibers composite

The electrospinning technique was employed in the fabrication of nanofiber composites materials, in the process PVA are used to stabilized the formation of electrospinning solution. Compared to other materials its low cost nontoxic materials and easy toward the realization of good solution for electrospinning set-up. The morphology of the synthesized Fe₃O₄ nanopaticles and PANi/Fe₃O₄/PVA nanofiber mixtures were confirmed using Field Emission Scanning Electron Microscope (FESEM), the result was revealed in the Figure 5 (a & b). As usual the Fe₃O₄ nanopaticles, as well as Polyaniline/Magnetite/Poly(vinyl alcohol) nanofiber solution, were achieved after loading 10 ml rotation solution and electrospun applied voltage at 16 kV, the homogeneous nanofiber with large diameter as well as smooth surface were obtained at PVA nanofiber composites materials, but when PANi/Fe₃O₄ were incorporated into PVA solution, a slight decrease of nanofiber diameter was observed, this observation was in agreement with recent study presented by [26-27] as shown in Figure 5.

Furthermore, the addition of PANi/Fe₃O₄ into PVA nanotubes causes the net electric charge density in the solution to increasing which indicates the production of nanofiber with a smaller diameter [28-29]. The existence of PANi in the composites solution indicates a related effect with salts added to the electrospinning solution. The addition PANi alters the viscosity of the solution but also increases the dielectric constant as well as the electrical conductivity concerning the spinning solution, favouring the nanofiber production with a shallow diameter, as shown in Figure 5. However, presence of Fe₃O₄ nanoparticles in the composites nanofiber was further revealed in the TEM results as shown in Figure 5 (b) which indicate that the nanofiber composites is indeed containing the magnetite nanoparticles.

Finally, the image-J computer software analysis was employed to estimate the diameter of PANi/Fe₃O₄/PVA nanofiber composites materials, the results was revealed in Figure 5(c). It was found from image-J result that the diameter of the composites was approximately 141 nm. Although, presence of PANi in the composites reduce the diameter, as a result the diameter of PANi/Fe₃O₄/PVA nanofiber composites was found to be 141 nm that is completely less than unmixed PVA nanofiber as reported by [30], suggesting that addition of PANi to PVA lower the diameter of the nanofiber materials [30-31].

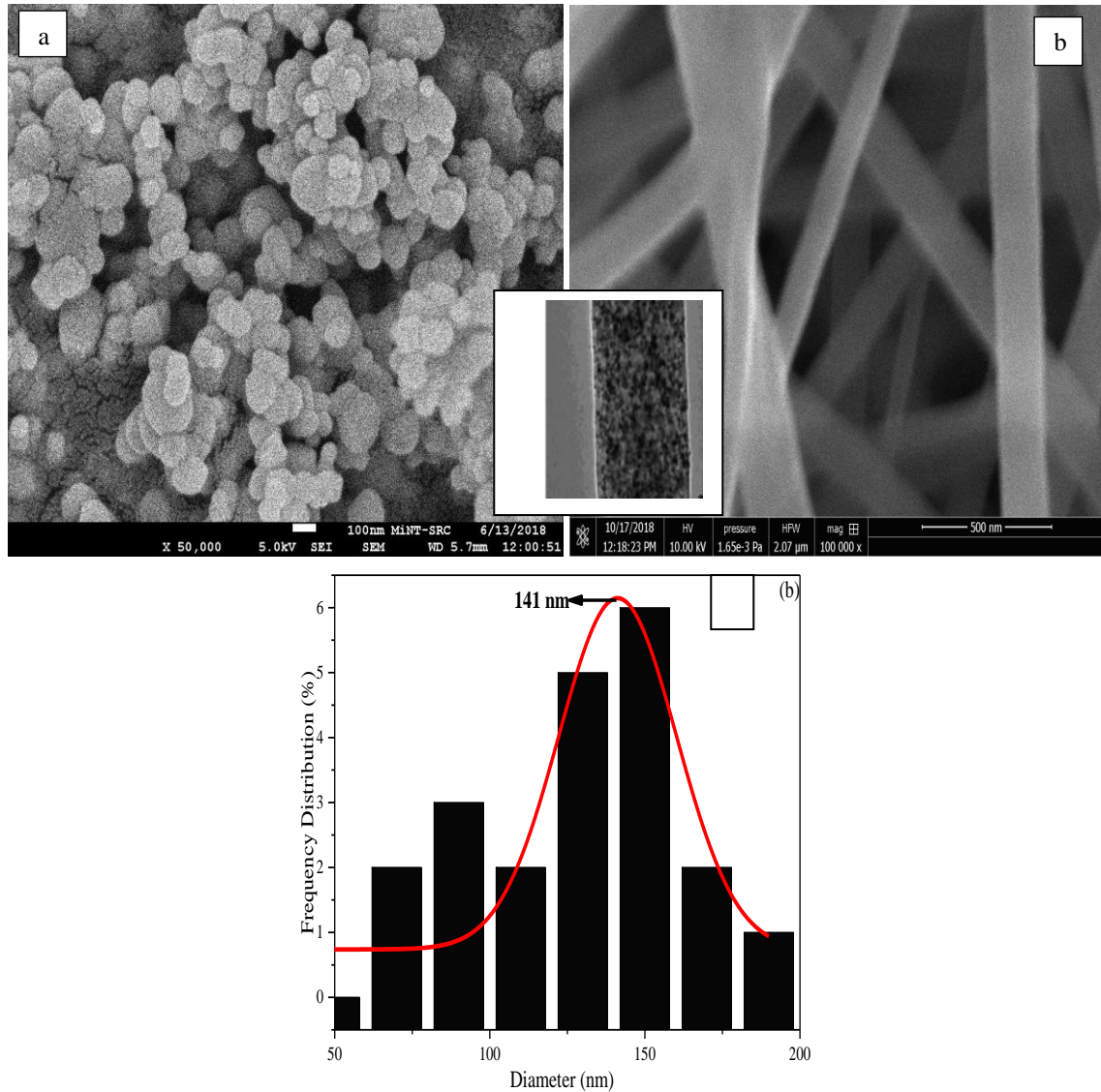


Figure 5: FESEM analysis of (a) Fe₃O₄ nanoparticles (b) PANi/Fe₃O₄/PVA (c) diameter distribution of PANi/Fe₃O₄/PVA nanofibers composites spun at 16 kV, 10 ml, 9 cm tips to collector distance and 0.35 ml/h

Conclusion

In summary, the structural pattern of the Fe₃O₄ nanoparticles and PANi/PVA nanofiber composites were investigated using XRD, the crystalline phase were recorded on the diffractometer with 2θ to 90°. The X-ray diffraction (XRD) pattern of PANi, PVA and Fe₃O₄ nanofiber materials shows the characteristic behaviour of both amorphous and crystalline phase by indication the peaks at 2θ = 23.12°, 24.14° and 35.13° respectively in the prepared nanocomposites materials. The FTIR analysis are used to study the functional group of the prepared materials, findings shows that several vibrating bond were found ranging from the results which could be correspond to the stretching vibration of

methyl group appearing in the poly aniline and poly vinyl alcohol nanotubes, this can simply revealed the absence of H₂O molecules in the composites. The bands appear at 1000 cm⁻¹, 1334 cm⁻¹, 1401 cm⁻¹, 1416 cm⁻¹, 1606 cm⁻¹, and 1656 cm⁻¹ take place in whole the FTIR spectrum of the prepared nanofibers composites are indeed because of the alkane and amine groups vibrations stretching which revealed the bond at 481 cm⁻¹, 592 cm⁻¹, and 847 cm⁻¹ due to presence of Fe₃O₄ nanoparticles, that are oxygen derivatives, carbon and hydrogen vibrating stretching. The morphology of the prepared PVA and PANi/PVA nanofiber composites were investigated using FESEM, it's observed that addition of PANi into PVA nanotubes the net electric charge density in the solution increase which lead to the production of nanofiber with smaller diameter. Furthermore, it was found from image-J result that the diameter of PVA nanofiber was found to be averagely to be 234 nm. Although, presence of PANi in the composites reduce the diameter, as a result the diameter of PANi/Fe₃O₄/PVA nanofiber composites was exhibited at 141 nm that is completely less than unmixed PVA nanofiber, suggesting that addition of PANi to PVA lower the diameter of the nanofiber materials.

Acknowledgments

This work was financially supported by Yusuf Maitama Sule University, Kano, Nigeria under Institutional Base Research Grand (IBR) funded by Tertiary Education Trust Fund (Tetfund). While preparation and characterization of the material was carryout at Microelectronic and Nanotechnology-Shamsuddin Research Centre (Mint-SRC) Universiti Tun Hussein Onn Malaysia (UTHM).

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