International Journal of ChemTech Research
CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555
Vol.10 No.6, pp 315-320, 2017

Synthesis and Characterization of Polyaniline-Ferric Ammonium Sulphate Nanocomposites

Dipika Khangate, Pramod Kulkarni*

Department of Chemistry , Hutatma Rajguru Mahavidyalaya Rajgurunagar, Pune MS India

Abstract : Polyaniline based nanocomposites containing Ferric ammonium sulphate nanocomposites were synthesized by using potassium persulphate as oxidizing agent for polymerization. Polyaniline-Ferric ammonium sulphate nanocomposites were synthesized using 0.1M aniline dissolved in 50mL 1M HC1, 0.01M ferric ammonium sulphate and 0.25M potassium persulphate as oxidizing agent. The prepared samples were characterized using some techniques such Fourier transforms infrared (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM). X-ray diffraction (XRD) and Fourier transform infrared (FT-IR) spectra studies indicated the presence of Fe-polyaniline bond. XRD spectra indicate the amorphous nature of this nanocomposites.
Keywords : Polyniline, Ferric ammonium sulphate, Polymerization, aniline hydrochloride.

Introduction:

In last few years, developments of conducting polymer-inorganic nanocomposites attracted many researcher due to their wide spread of potential applications and high absorption in the visible part of the spectrum and high mobility of the charge carriers. Polyaniline is the most attractive conductive polymers because of the presence of the reactive -NH- groups in polymer chain [1] and used in broad applications such as supercapacitors [2], batteries [3, 4], sensors [5], electronic devices [6], corrosion protection in organic coatings [7, 8]. Polyaniline has attracted considerable attention because of its physical and chemical properties, good electrical conductivity and mechanical properties, high environmental stability, low cost, light weight, flexibility, easy control of doping level, diverse color changes corresponding to oxidation level, facile fabrication and possibility of both chemical and electrochemical synthesis [9-13].

Conducting polymers are conjugated chain of organic compounds that exhibit high electrical conductivity similar to metals due to the presence of large carrier concentrations of extended π-electrons, known as polarons, which allow charge mobility along the backbone of the polymer chain. Their electrical conductivities are comparable with metals but polymers have many advantages, such as being light-weight, resistance to corrosion, flexibility, and low cost. Conducting polymers are finding numerous applications in television sets, cellular telephones, displays, light, emitting diodes, solar cells, batteries, actuators, sensors, electromagnetic shielding and microelectronic devices [14].

Metals and semiconducting nanostructure materials are utilized as stabilizers or capping agents of these conducting polymers. In last decades, several reports have been published on the synthesis of polyaniline nanocomposites with the inorganic nano-scale such as Fe@C magnetic [15], TiO_2 [16], Ag[17], Ni[18], CdS[19], MnO_2 [20], magnetite[21], Fe[22], Au[23], Pd[24], PbS[25], CeO_2 [26], Mn_3O_4 [27], Sb_2O_3 [28] have been reported. To prepare these nanoscale composite materials effectively, numerous methods have been employed such as physical mixing [29], the sol-gel technique [30], in-situ chemical polymerization in an aqueous solution with the presence of polymer, monomer and inorganic particles[31], emulsion technology [32], sonochemical process [33], radiation technique[34]. In this paper, we reported the new polyaniline nanocomposites with ferric ammonium sulfate as dopant. For polymerization of aniline, ammonium persulphate used as oxidizing agent and this oxidizing agent is hazardous compare to potassium persulphate.

In this paper PANI nanocomposites with ferric ammonium sulphate were synthesized by in situ oxidative polymerisation of monomer aniline with potassium persulphate and followed by addition of ferric alum to afford the nanocomposites. The products were characterized by FTIR spectra, X-ray diffraction, electron microscopy (SEM).

2. Experimental

2.1 General :

Aniline, Ferric Ammonium Sulphate and Potassium persulphate were purchased from Merck. Aniline was distilled before to use. All supplementary chemicals were of analytical grade and solutions were prepared with distilled Water. FTIR characterization was done using a JASCO FTIR-6100 spectrometer. The XRD result was obtained using D8 advance diffractometer, Bruker AXS. SEM images were taken on FEI Nova Nano SEM 450 analytical scanning electron microscopes.

2.2 Synthesis of PANI-Ferric Ammonium Sulphate Nanocomposities:

The PANI/Ferric Ammonium Sulphate nanocomposite was prepared by an in-situ chemical oxidation polymerization of aniline using Potassium persulphate as an oxidant in presence of Ferric ammonium sulphate at room temperature in air. Aniline (0.1M) was dissolved in 50 mL of HCl (1M) and stirred at room temperature. 0.01M solution of Ferric ammonium sulphate was added in the above solution with stirring. As an oxidizing agent, 20 mL of potassium persulphate(0.25 M) was then slowly added drop wise to the mixture with a constant stirring over a period of 1 h. The reaction was then left at room temperature for 24 h. The product obtained was collected by filtration and washed several times by distilled water and acetone until the filtrate was colorless. The product was dried at 60ºC for 24h.

1. Results and Discussions

3.1 FTIR:

FTIR spectra of PANI/ferric ammonium sulphate composites has been shown in Fig. 1. Characteristic bands of PANI have been shown by 577 cm^{-1}, 666 cm^{-1}, 733 cm^{-1}, 792 cm^{-1}, 1111 cm^{-1}, 1233 cm^{-1}, 1292 cm^{-1}, 1436 cm^{-1}, 1477 cm^{-1}, 1556 cm^{-1}. The bands at 733 and 792 cm^{-1} are due to C-H out of plane bending vibration and para-disubstituted aromatic rings, respectively. The peak appearing at 1292 cm^{-1} corresponds to C-N stretching of secondary amine in polymer main chain, and the band observed at 1111 cm^{-1} is attributed to in plane bending vibration of C-H mode. The bands at 1556 cm^{-1} and 1477 cm^{-1} attributed to C=C stretching deformation of quinonoid and benzenoid units of PANI respectively. The bands at 577 cm^{-1} indicates stretching vibration between M-N. The bands at 3812 cm^{-1} , 3384 cm^{-1} , 3217 cm^{-1} , 2360, 2166, 2056, 1981, 1756 are due to ferric ammonium sulphate. The bands at 491, 446, 416 are due to ferric atom.

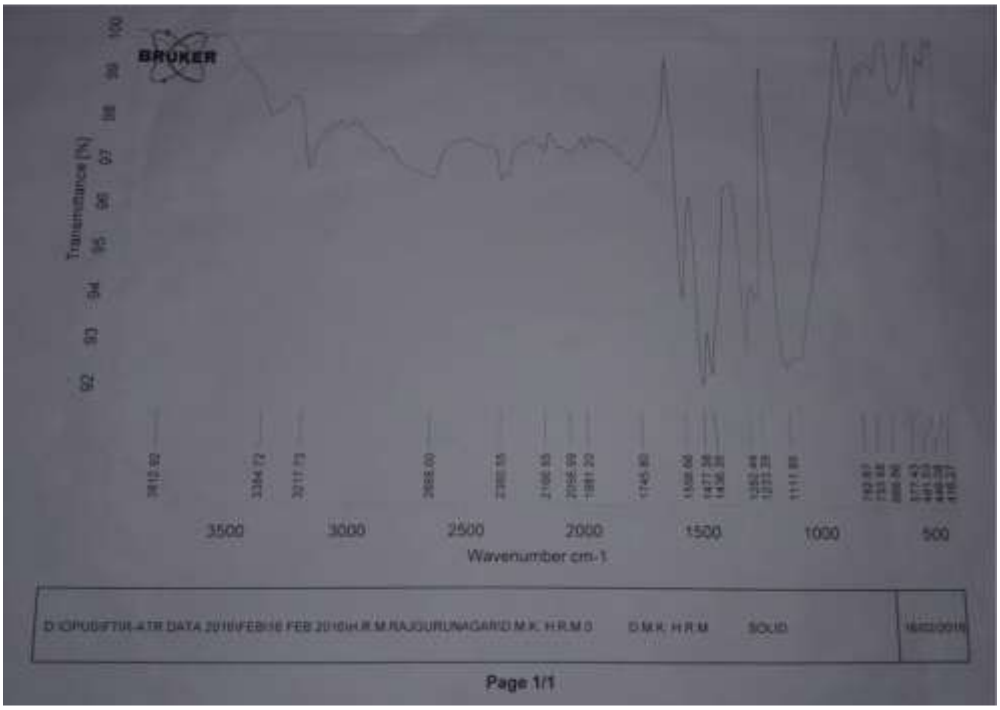

Fig. 1. FTIR spectra of Polyaniline -ferric ammonium sulphate

3.2 XRD Analysis:

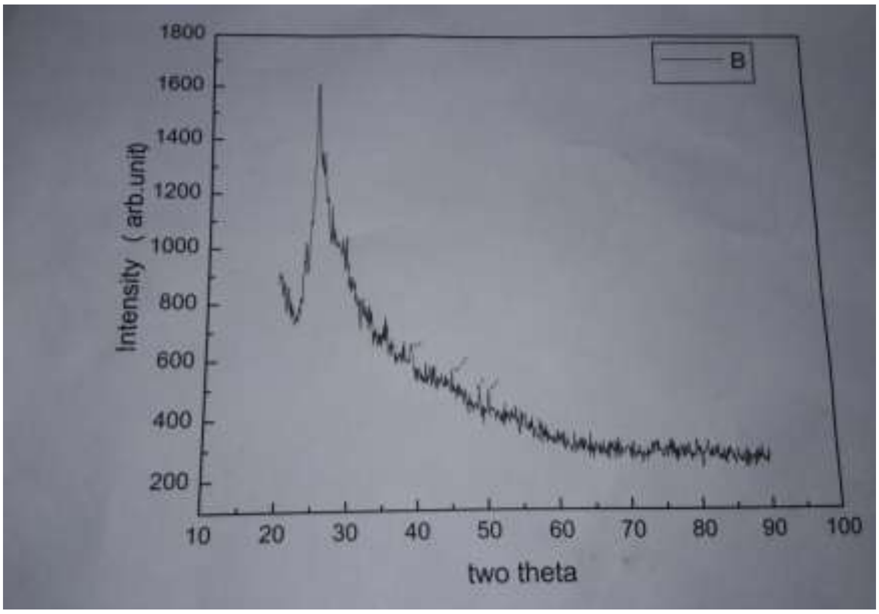

Fig. 2. XRD of polyaniline-ferric ammonium sulphate nanocomposities

The XRD patterns of the PANI/Ferric ammonium sulphate composites are shown in figure 2. Figure shows that XRD pattern of composites is amorphous in nature. The peak at $2\theta=25^{\circ}$ is characteristics peak of PANI which retains in the composite. The heights of peak at $2\theta=$ 39.34, 44.75, 47.67, 50.45 are corresponds to ferric ammonium sulphate.

3.3 SEM Analysis:

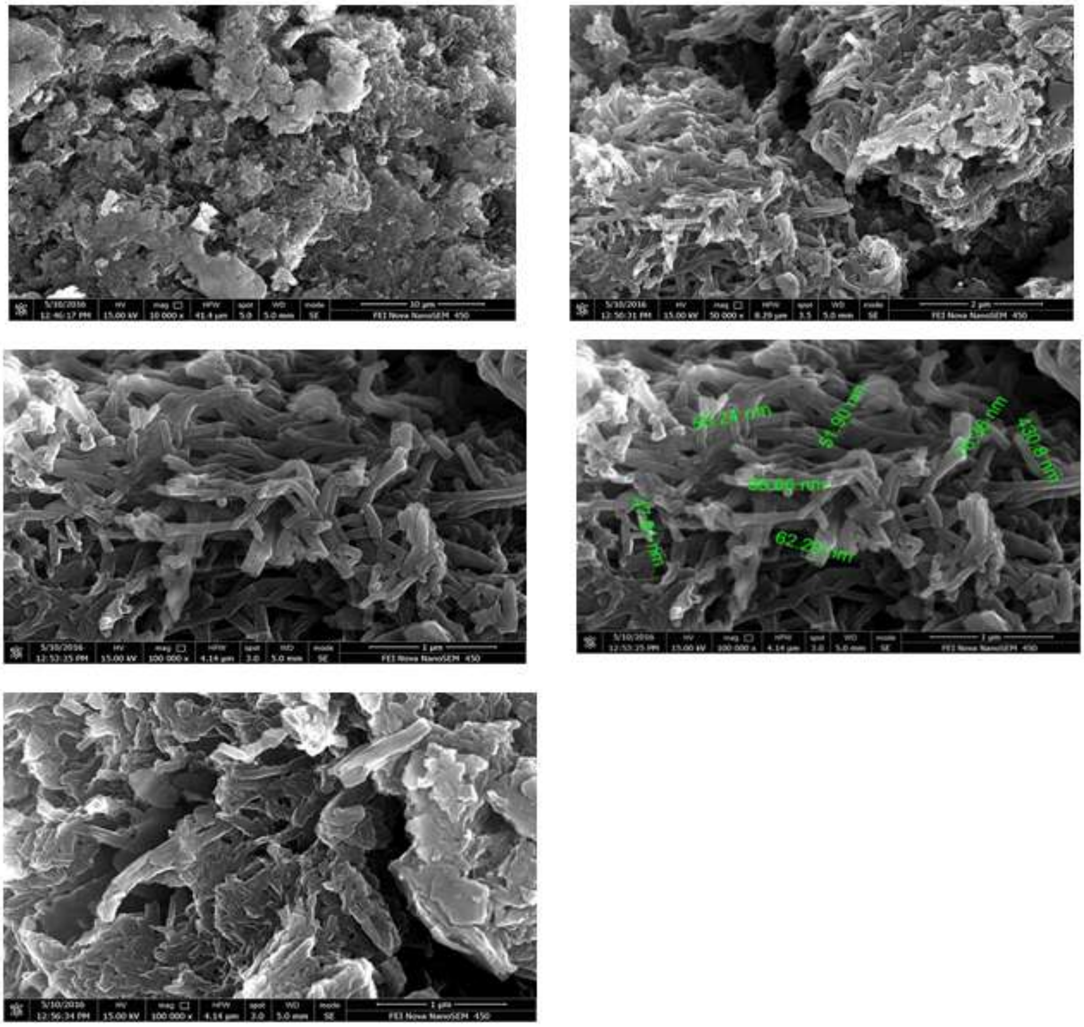

Fig. 3. SEM images of polyaniline-ferric ammonium sulphate nanocomposities

The SEM micrographs were used to investigate the morphology of polyaniline-ferric ammonium sulphate nanocomposites. PANI-ferric ammonium sulphate shows agglomerated structure. The shape of the nanocomposites is rod shaped. Ferric ammonium sulphate nanoparticles with average diameter sizes of about 57.88 nanometers have been distributed in the polymer matrix and diameter with high size is due to polyaniline nanoparticles. The SEM images of polyaniline –ferric ammonium sulphate is shown in figure 3.

2. Conclusion

In the present paper, we described novel nanocomposites of polyanline-ferric ammonium sulphate by in situ polymerization of aniline using potassium persulphate. The potassium persulphate is substitute for ammonium persulphate to polymerization of aniline to polyaniline. The applications of this nanocomposites is under study in our laboratory. This method has several merits over other reported method such as insitu synthesis of polyaniline nanocomposites, yield is good and eco-friendly conditions.

Reference

1. Mostafaei, A. Zolriasatein, "Synthesis and characterization of conducting polyaniline nanocomposities conataining ZnO nanorods", Progress in Natural Science: Materials International , 2012, 22, 273-280.

2. Li N., Xao Y. , Xu C., Li H. , Yang X., "Facile Preparation of polyaniline nanoparticles via electrodeposition for supercapacitors", Int. J. Electrochem Sci , 2013, l.8, 1181-1188.
3. Devendrappa H., Subba Rao U. V., Ambika Prasad M. V. N., " Study of dc conductivity and battery application of polyethylene oxide/polyaniline and its composites", Journal of Power Sources, 2006, 155, 368-374.
4. MacDiarmid A. G., Yang L. S., Huang W. S., Humphery B. D., "Polyaniline: Electrochemistry and application to rechargeable batteries", Synthetic Metals, 1987, 18, 393-398.
5. Sen T., Mishra S., Shimpi N. G., "Synthesis and sensing applications of polyaniline nanocomposities: a review", RSC Adv ,2016, 6, 42196-42222.
6. Bocchini S., Chiolerio A., Porro S., Accardo D., Garino N., Beitka K., Perrone D., Pirri C. F., "Synthesis of polyaniline-based inks, doping thereof and test device printing towards electronic applications", J. Mater. Chem. C , 2013, 1, 5101-5109.
7. Chen F., Liu P., "Conducting polyaniline nanoparticles and their dispersion for waterborne corrosion protection coatings", ACS Appl. Mater Interfaces, 2011, 3, 2694-2702.
8. Ansari R., Alikhani A. H., "Application of polyaniline/nylon composites coating for corrosion protection of steel", J Coat Technol Res , 2009, 6, 221-227.
9. Boeya Z. A., Sergeyey V. G., "Polyaniline: Synthesis, properties, and application", Polymer Science Series C , 2014, 56, 144-153.
10. Neelgund G. M., Oki A., "A facile method for synthesis of polyaniline nanospheres and effect of doping on their electrical conductivity", Polym Int. 2011, 60, 1291-1295.
11. Rosa V. A., Hugo R. B., Eduardo A. Y. , "Synthesis and Characterization of polyaniline and poly-ortho-methoxyanline, behaviour against Carbon Steel Corrosion", J Chilean Chemical Soc , 2003, 48, 35-40.
12. Kim B. H., Jung J. H., Hong S. H., Kim J. W., Choi H. J., Joo J., " Physical characterization of emulsion intercalated polyaniline-clay nanocomposite", Current Applied Physics ,2001, 1, 112-115.
13. Patil R. C., Radhakrishan S., "Conducting polymer based hybrid nano-composites for enhanced corrosion protective coatings", Progress in Organic Coatings, 2006, 57, 332-336.
14. Meftah M., Gharibshahi E., Soltani N., Yunus W. M. M., Saion E., "Structural, Optical and Electrical properties of PVA/PANI/Nickel Nanocomposites Synthesized by Gamma Radiolytic Method" Polymers, 2014, 6, 2435-2450
15. Fleaca C. T., Dumitrache F., Morjan I., Niculescu A. –M., Sandu I., Ilie A., Stamatin I., Iordache A., Vasile E., Prodan G., "Synthesis and Characterization of polyaniline-Fe@C magnetic nanocomposite powder", Applied Surface Science , Vol. 374, 2016, pp. 213-221.
16. Pawar S. G., Patil S. L., Chougule M. A., Mane A. T., Jundale D. M., Patil V. B., "Synthesis and Characterization of Polyaniline:TiO_2 Nanocomposites", International Journal of Polymeric Materials and Polymeric Biomaterials ,2010, 59, 777-785.
17. Poyraz S., Cerkez I., Huang T. S., Liu Z., Kang L., Zhang X., "One-step synthesis and characterization of polyaniline nanofiber/silver nanoparticles composite networks as antibacterial agents", ACS Appl Mater Interfaces , 2014, 26, 20025-20034.
18. Goswami M., Ghosh R., Meikap A. K., "Synthesis and characterization of new Polyaniline-Ni nanocomposite", Advanced Science Letters, 2016, 22, 229-231.
19. Singh N., Kulkarni M. V., Lonkar S. P., Viswanath A. K., Khanna P. K., "CdS/Polyaniline nanocomposities: synthesis and characterization", Synthesis and Reactivity in Inorganic, Metal-Organic and Nano-Metal Chemistry, 2007, 37, 153-159.
20. Meng F., Yan X., Zhu Y., Si P., "Controllable synthesis of MnO_2 /polyaniline nanocomposite and its electrochemical capacitive property", Nanoscale Research Letters , 2013, 8, 179
21. Basavaiah K., Pavan Kumar Y., Prasada Rao A. V., "A facile one-pot synthesis of polyaniline/magnetite nanocomposites by micelles-assisted method", Appl Nanosci , 2013, 3, 409-415.
22. Sedaghat S., "Synthesis of Iron-polyaniline nanocomposites and the effect of emulsifier", World Applied Sciences Journal, 2013, 21 , 544-547.
23. Bogdanović U., Pašti I., Ćirić-Marjanović G., Mitrić M., Ahrenkiel S. P., Vodnik V., " Interfacial synthesis of Gold-Polyaniline nanocomposite and its electrocatalytic application", ACS Appl Mater Interfaces, 2015, 7, 28393-28403.
24. Dutt S., Kumar R., Siril P. F., "Green synthesis of a palladium-polyaniline nanocomposite for green Suzuki-Miyaura coupling reactions", RSC Adv ,2015, 5, 33786-33791.

25. Jing S., Xing S., Dong F., Zhao C., "Synthesis and characterization of PbS/polyaniline core-shell nanocomposities based on octahedral PbS nanocrystals colloid", Polymer composites, 2008, 29, 1165-1168.
26. He Y., "Synthesis of polyaniline/nano-CeO_2 composite microspheres via a solid-stabilized emulsion route", Materials Chemistry and Physics, 2005, 92, 134-137.
27. Shambharkar B. H., Umare S. S., Rathod R. C., "Synthesis and Characterization of Polyaniline-Mn_3O_4 Nanocomposite: Electrical Conductivity and Magnetic Studies" Transactions of the Indian Institute of Metals , 2014, 67, 827-834.
28. Khalili R., Eisazadeh H., "Preparation and Characterization of Polyaniline/Sb_2O_3 Nanocomposite and its application for removal of Pb(II) from aqueous media" IJE Transactions B: Applications, 2014, 27, 239-246.
29. Akelah A., El-Deen N. S., Hiltner A., Baer E., Moet A., "Organophilic rubber-montmorillonite nanocomposites", Materials Letters, 1995, 22, 97-102.
30. Jang S., Han M., Im S., "Preparation and Characterization of conductive Polyaniline/Silica hybrid composites prepared by Sol-Gel process", Synthetic Metals, 2000, 110, 17-23.
31. Ray S. S., Biswas M., "Water dispersible conducting nanocomposites of polyaniline and poly(n-vinylcarbazole) with nanodimensional Zirconium dioxide," Synthetic Metals, 2000, 108, 231-236.
32. He Y., "Synthesis of Polyaniline-CeO_2 composite microspheres via a solid-stabilized emulsion route," Materials Chemistry and Physics, 2005, 92, 134-137.
33. Zhang K., Park B-J., Fang F-F., Choi H. J., " Sonochemical preparationof Polymer nanocomposites" Molecules, 2009, 14, 2095-2110.
34. Ali M. A., Saion E., Yahya N., Kassim A., Dahlan K. M., Hashim S., "Synthesis of conducting polyaniline nanocomposites by radiation doping" Journal of Engineering Science and Technology , 2007, 2, 111-118

*****