Conducting Ferromagnetic Copolymer of Aniline and 3,4-Ethylenedioxythiophene Containing Nanocrystalline Barium Ferrite Particles

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ABSTRACT: This article reports the synthesis of conducting ferromagnetic complex of 3,4-ethylenedioxythiophene (EDOT) and aniline (An) containing M-type hexagonal barium ferrite ($BaFe_{12}O_{19}$) particles using *in situ* emulsion polymerization and electrochemical oxidative polymerization. Magnetic and conductivity studies reveal that the conducting ferromagnetic complex possesses high-saturation magnetization (M_s) value of 29.2 emu/g and conductivity of the order of 0.256 S/cm determined through vibrating sample magnetometer and four-probe method. Microwave measurement has shown the reflection loss (R_L) of −12.1 dB in Ku-band that can be used as a microwave absorbing material. The polymer complex was further characterized by techniques like X-ray diffraction, Fourier transform infrared, UV–visible, cyclic voltammetry, and thermal analysis with thermogravimetric analysis. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 108: 2218–2225, 2008

Key words: conducting polymers; copolymerization; emulsion polymerization; synthesis and ferromagnetic conducting complex

INTRODUCTION

Electronically conducting polymers are special class of materials with potential applications in many devices like electrode material in energy storage systems,[1,2] OLEDs,[3–6] plastic solar cells,[7,8] antistatic coatings,[9,10] optoelectronic devices.[11,12] In recent years, conducting polymers have added new dimensions to the technology of protective sheath for electronic equipments against electromagnetic interference in microwave range[13–15] and RAM coatings.[16,17] The major forerunners like polyacetylene, polyaniline, polythiophene, and polypyrrole have shown interesting chemical and physical properties due to their conjugated delocalized π-electron network. Among these polymers much attention has been paid to polyaniline due to its unique doping mechanism, high environmental and thermal stability. The explosive growth in the application of electronic devices across a very broad spectrum of military, industry, commercial, and consumer electronic equipments have created a new form of pollution—electromagnetic radiation noise that causes interference or the malfunction of the equipment. Hence, the main objective of this research work is to develop ferromagnetic conducting polymer, which can be used as microwave absorbing material in X and Ku-band. Earlier studies have been made on the development of composites having both conducting and ferromagnetic properties by the incorporation of ferrite particles like Fe_3O_4, manganese-zinc ferrite particles.[18–29] But no work has been reported on the copolymer of aniline (An) and EDOT. This article reports the synthesis of copolymer poly(AnEDOT) of 3,4-ethylenedioxy thiophene (EDOT) and aniline monomers in the presence of dodecylbenzene sulfonic acid (DBSA) and its complex poly(AnEDOT)-BaF with nanocrystalline barium ferrite (BaF) particles through emulsion polymerization. To overcome the insoluble nature of EDOT in aqueous medium, homogenization of EDOT was carried out in DBSA, and subsequently copolymerization with barium ferrite was carried out. The resulting ferromagnetic conducting copolymer complex possesses good magnetization value with moderate conductivity, which was confirmed by vibrating sample magnetometer (VSM) and four-probe technique.

EXPERIMENTAL

Synthesis of barium ferrite

The synthesis of barium ferrite was carried out via precursor route[30] by dissolving 1 : 12 : 13 molar ratio of barium nitrate (Qualigens, India), ferric nitrate

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(Qualigens, India), and citric acid (Merck, India), respectively, in distilled water. Aqueous ammonium solution (Merck, India) was added to maintain the pH of the solution at 9.0. The resulting solution was heated at 90°C with continuous stirring to form a viscous gel. The gel so formed was ignited resulting in the formation of dendrite structure, which was crushed to powder and calcined in a programmable tubular furnace at 900°C for 2 h. The barium ferrite particles so obtained were further grinded using Retsch ''PM-400'' planetary-ball mill in tungsten carbide jars.

Synthesis of copolymer poly(AnEDOT) and poly(AnEDOT)-BaF complex

Copolymer poly(AnEDOT) doped with DBSA and its conducting ferromagnetic copolymer poly(AnEDOT)-BaF were prepared by emulsion polymerization. Poly(AnEDOT) was synthesized by adding 0.05*M* EDOT (Bayer AG, Germany) 0.05*M* An (Merck, India) in 0.3*M* aqueous DBSA solution. The mixture was homogenized using AR-MICCRA D8 homogenizer for 2 h to get homogenous emulsion. The resulting solution was polymerized by 0.1*M* ammonium persulfate (APS) (Merck, India) solution at 0°C with continuous stirring. A bluish-green polymer so obtained was washed with isopropanol and distilled water, and dried at 60°C in vacuum oven. The synthesis of poly(AnEDOT)-BaF complex was carried out by homogenizing barium ferrite particles in aqueous DBSA solution. Then monomers aniline (0.05*M*) and EDOT (0.05*M*) were added to the reaction mixture. Here, barium ferrite and monomer were taken in 2 : 1 weight ratio. The solution so formed was homogenized for 2 h, and polymerization was carried out with APS at 0°C. The blackish-green precipitates of copolymer embedded with barium ferrite (poly(AnEDOT)-BaF) were washed and dried in vacuum oven. Besides this, pure polyaniline and poly(3,4-ethylenedioxythiophene) (PEDOT) doped with DBSA were also synthesized by chemical oxidative polymerization using the APS at 0°C.

Electrochemical polymerization

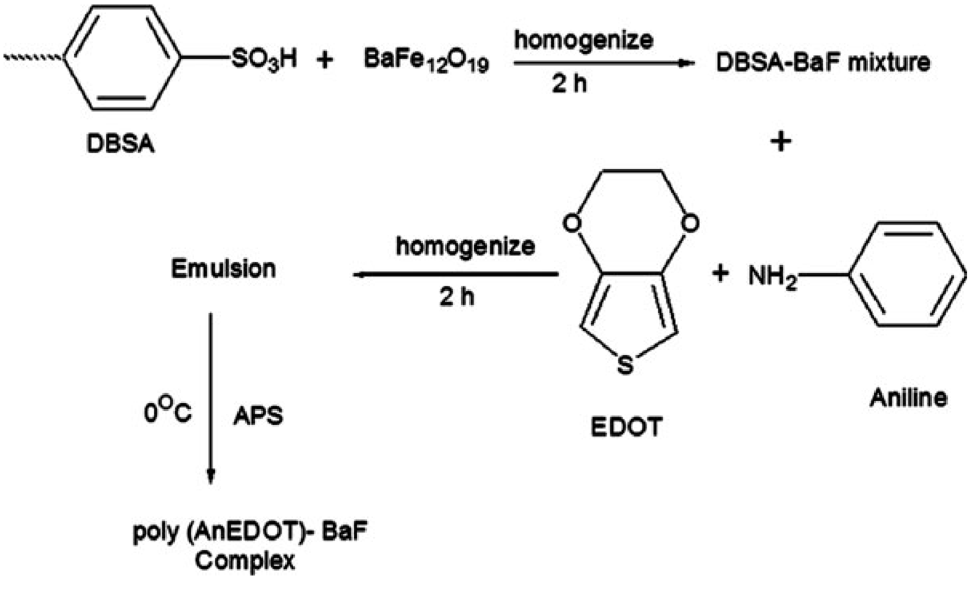
Polyaniline, PEDOT, poly(AnEDOT), and poly(AnEDOT)-BaF were prepared electrochemically in 0.3*M* DBSA medium on platinum electrodes by cycling the potential between −0.2 to 0.85 V and −0.2 to 0.95 V versus saturated calomel electrode (SCE) with Pt as counter electrode. Cyclic voltammograms of the corresponding polymers and copolymers were recorded on Electrochemical Workstation, PSG 30 (Autolab, The Netherlands).

CHARACTERIZATION

X-ray studies were carried out on D8 Advance X-ray diffractometer (Bruker) using Cu Kα radiation (λ = 1.540598 Å) in scattering range (2θ) of 10–70° with a scan rate of 0.025°/s and slit width of 0.1 mm.

The magnetic measurements of the samples were performed using the VSM model 7304 Lakeshore Cryotronics, with a maximum magnetization of 1.2T, and the sample is vibrated horizontally with the frequency of 76 Hz.

Fourier transform infrared (FTIR) spectra were recorded on Nicolet 5700 in transmission mode in



Scheme 1 Schematic representation of the copolymerization of aniline and EDOT in the presence of barium ferrite particles.

the range of 400–4000 cm^{-1}. The spectroscopic grade KBr disks were used for collecting the spectra with a resolution of 4 cm^{-1} performing 32 scans.

The band positions were determined using UV/visible spectrophotometer (Shimadzu 1601) in the range of 270–1100 nm. All measurements were recorded by dissolving the known amount of samples in chloroform, and using the filtrate of the above solution for UV/visible spectroscopy.

Room temperature conductivity was measured with four-probe technique using Keithley programmable current source (model 6221) and nanovoltmeter (model 2182A). For each sample, compressed pallets were formed at 5 ton in a rectangular die and four contacts were made on each end using silver paste.

Thermogravimetric analysis (TGA) was performed by Mettler Toledo TGA 851e in nitrogen atmosphere with a flow rate of 60 mL/min, heating rate at 10°C/min from 25 to 700°C.

Microwave measurements were carried out on Agilent E8362B Vector Network Analyzer in microwave range of 12.4–18.0 GHz (Ku-band) through 15.8 mm × 7.9 mm × 6 mm copper sample holder connected by wave-guide flanges back by perfect conductor. To avoid air gap, the above sample holder is modified with a grove of 1.5 mm on each side with a depth of 3 mm.

Polymer samples prepared by chemical oxidative polymerization were used for the characterization of UV–visible, TGA, XRD, FTIR, microwave measurement, and VSM. Electrochemical studies were carried out only to check the oxidation potential of polyaniline, PEDOT, and their copolymer. Cyclic voltammetric studies were carried out to look into the redox behavior of copolymer, and to see the effect of oxidation potential on the electrochemical behavior when barium ferrite particles were incorporated in the reaction cell.

RESULTS AND DISCUSSION

The polymerization of EDOT and aniline to copolymer poly(AnEDOT) proceeds through the formation of radical cations by redox reaction, which causes the reorganization of electronic structures, to give two semiquinone radical cations. These radical cations lead to the formation of dimer, which was further oxidized to form stable electrically conducting polymer. During the copolymerization of EDOT and aniline in the presence of DBSA, it has been observed that dopant gets attached to the EDOT and anilinium ring resulting in the formation of a flexible and soluble conducting polymer. Incorporation of barium ferrite particles during the process of copolymerization in presence of surfactant DBSA results in the formation of a conductive as well as ferromagnetic polymer. Here, DBSA also helps in the functionalization of barium ferrite and avoids the phase segregation. The schematic representation of copolymerization is shown in Scheme 1.

The electrochemical growth of polyaniline, PEDOT, poly(AnEDOT), and poly(AnEDOT)-BaF was investigated in DBSA medium. On carrying out electrochemical polymerization of aniline in DBSA medium by sweeping the potential between −0.2 and 0.85 V versus SCE, it has been observed that in the first cycle only one oxidation peak is observed which is related to the oxidation of aniline. In subsequent cycles, new oxidation peaks appear indicating that the radical cations are generated at the electrode surface and help to attain the constant peak potential. The increase in the intensity of peak (*I*) demonstrate the growth of polymer. The electrochemical growth of aniline is believed to proceed via the generation of radical cations, which react together to form dimer and their further oxidation leads to form poly-

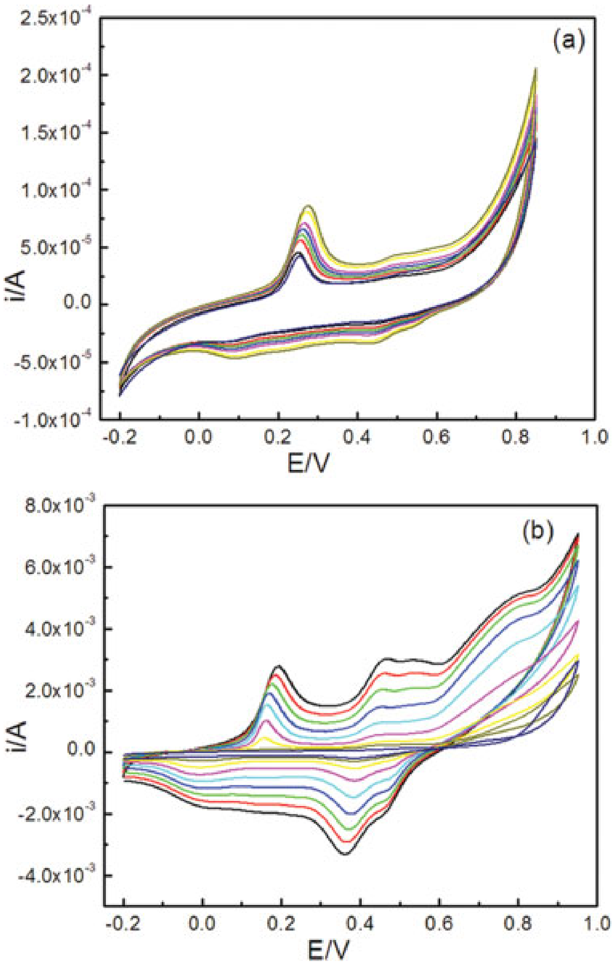
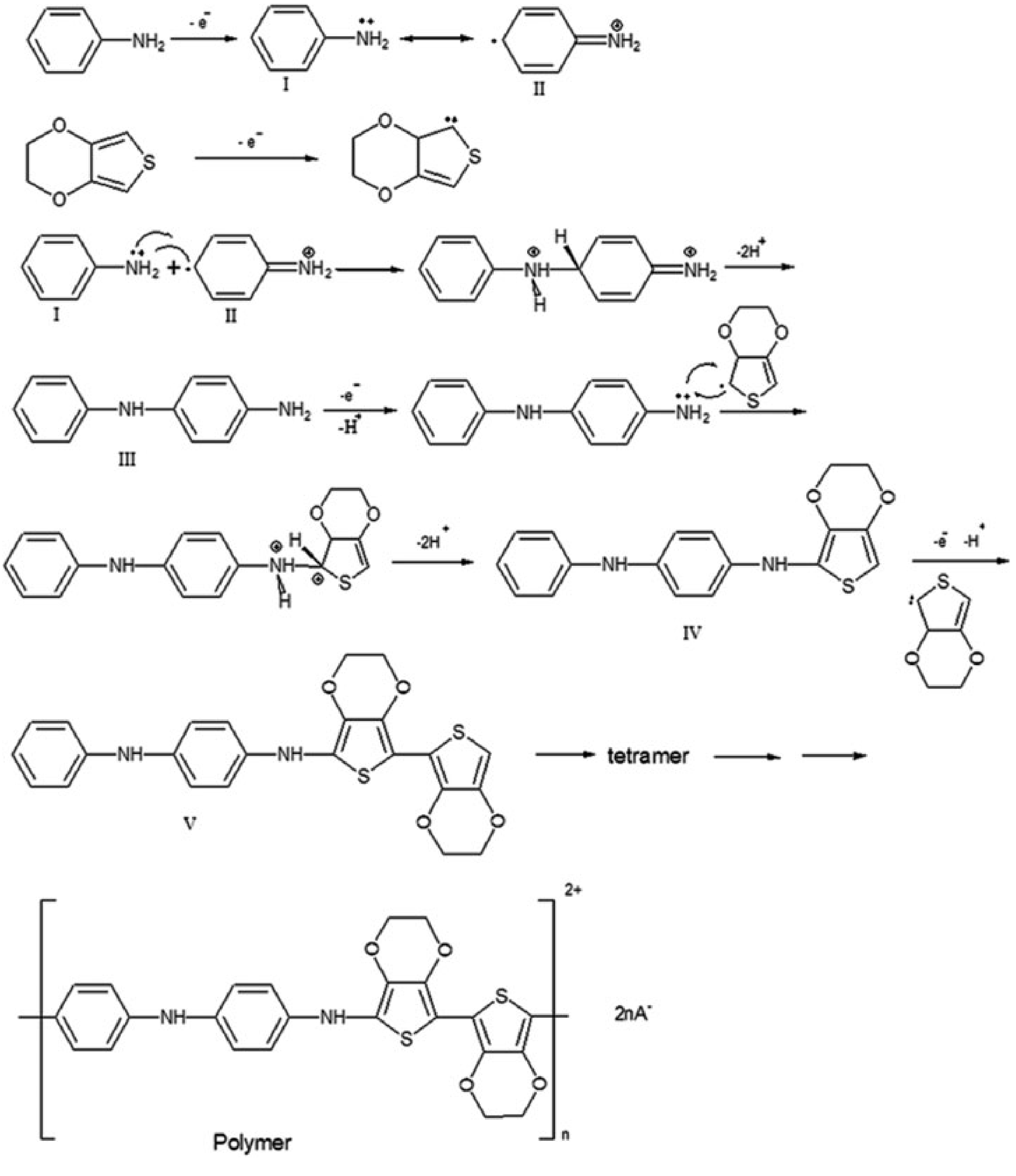


Figure 1 Electrochemical growth behavior of aniline (0.05*M*) and EDOT (0.05*M*) in 0.3*M* DBSA on cycling the potential between −0.2 and 0.85 V (curve a); 0.05*M* aniline, 0.05*M* EDOT, and barium ferrite in DBSA from −0.2 to 0.95 V (curve b) versus SCE on platinum electrode at a scan rate of 20 mV/s. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



Scheme 2 Proposed mechanism of copolymerization of aniline and EDOT.

mer chain. In case of polymerization of EDOT, it has been observed through galvanostatic technique that the growth of polymer starts at 0.8 V. Hence, electropolymerization of EDOT was also studied by cyclic voltammetry technique by cycling the potential between −0.2 and 0.85 V versus SCE on platinum electrode. For aniline and EDOT, it was observed that the current increases at same potential, therefore copolymerization was carried out electrochemically. The growth of poly(AnEDOT) and poly(AnEDOT)-BaF are shown in Figure 1 (curves a and b), while Scheme 2 demonstrates the possible interaction mechanism of aniline radical cations with ethylenedioxythiophene radical cations to form copolymer.

The cyclic voltammogram of these polymers were studied by cycling the potential between −0.2 and 0.85 V versus SCE reference electrode at a scan rate of 20 mV/s. Cyclic voltammetry studies has indicated that the peak potential (E_{pa}) of polyaniline in DBSA lies at 0.115, 0.540, and 0.774 V versus SCE, while CV of PEDOT showed only one peak at 0.354 V. However, the peak potential values of copolymer of aniline and EDOT were observed at 0.22 and 0.501 V (Fig. 2, curve a). This implies that redox

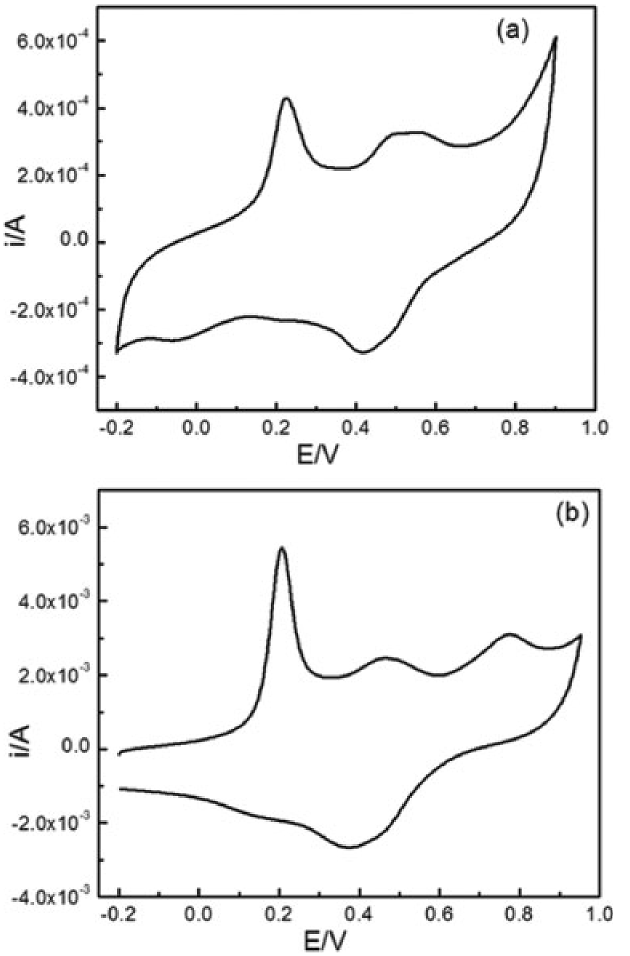


Figure 2 Cyclic voltammetric studies of poly(AnEDOT) (curve a) and poly(AnEDOT)-BaF (curve b) in DBSA medium at a scan rate of 20 mV/s on Pt electrode versus SCE.

peaks of copolymer poly(AnEDOT) have shifted showing a possible interaction of two monomeric moieties. A cyclic voltammetric study of poly(AnEDOT)-BaF complex was also carried out to see how the incorporation of barium ferrites particles affects the cyclic voltammetric behavior of copolymer (Fig. 2, curve b). The cyclic voltammogram indicates that the first redox peak was observed at 0.205 V, and consequently other peaks are observed at 0.464 and 0.76 V. This shows that incorporation of barium ferrite has resulted in the shifting of peak potential value from 0.22 to 0.205 V, which indicates the interaction of ferrite particles in the copolymer matrix.

The UV–visible spectra of polyaniline, PEDOT, poly(AnEDOT), and poly(AnEDOT)-BaF were carried out in chloroform medium (See supplementary material). Polyaniline exhibit characteristic absorption bands at 352, 426, and 735 nm, which are due to π–π^* transition of the benzenoid ring, polaron and bipolaron transitions, while PEDOT shows the band position at 358 and 622 nm. However, the absorption bands of poly(AnEDOT) were observed at 292, 380, and 662 nm, which indicate a structural rearrangement between the aniline and thiophene ring of EDOT to form the copolymer chain. The shifting of absorption peak from 735 to 662 nm indicates that polarons are less delocalized than polyaniline due to structural conjunction in the polymer chain. Poly(AnEDOT)-BaF shows absorption bands at 293, 381, and 632 nm. Further hypsochromic shifting in polaron band from 662 to 632 nm may be due to the interaction of ferrite particles to the —NH of anilinium unit, which is attributed to the change in conductivity of the copolymer poly(AnEDOT)-BaF complex.

FTIR spectra of the polymer samples were recorded in KBr. The major peaks of polyaniline are observed at 1460 and 1570 cm^{-1} due to C=C bond stretching of benzenoid and quinoid rings, respectively. The peak at 1124 cm^{-1} may be due to plane bending vibration of C—H bond of the N=Q=N, Q=NH—B, and B—NH—B. In case of PEDOT the characteristic vibrational bands are observed at 1522 cm^{-1} due to C=C bond stretching of thiophene ring, and at 1194, 1136, and 1089 cm^{-1} due to C—O—C bond stretching, while band at 681, 833, and 975 cm^{-1} are due to C—S bond of thiophene ring. Peak at 1330 cm^{-1} has been reported to be the main characteristic peak of PEDOT.[31] FTIR spectra of poly(AnEDOT) shows the major peaks at 1463, 1299, 1218, 1131, 826, and 668 cm^{-1}. On comparing the spectra of poly(AnEDOT) with polyaniline and PEDOT, shifting in position of the major peaks were found which arises due to the formation of copolymer of aniline and EDOT. FTIR spectra of barium ferrite show characteristic peaks at 590, 545, and 443 cm^{-1} which are due to Fe—O bond stretching (Fig. 3, curve a). In case of poly(AnEDOT)-BaF complex, the peaks at 545 and 432 cm^{-1} confirm the presence of the ferrite particles in the polymer matrix (Fig. 3, curve b). The disappearance of peak at 590 cm^{-1} and the shift of peak from 577 cm^{-1} of poly(AnEDOT) (Fig. 3, curve c) copolymer to 581 cm^{-1} in poly(AnEDOT)-BaF can be assigned to

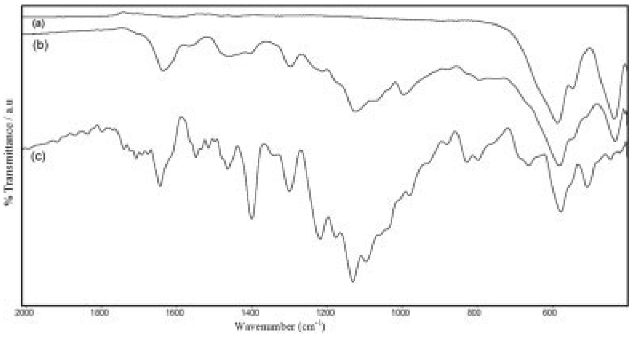


Figure 3 FTIR spectra of barium ferrite powder (a); poly(AnEDOT)-BaF (b), and poly(AnEDOT) (c) in KBr.

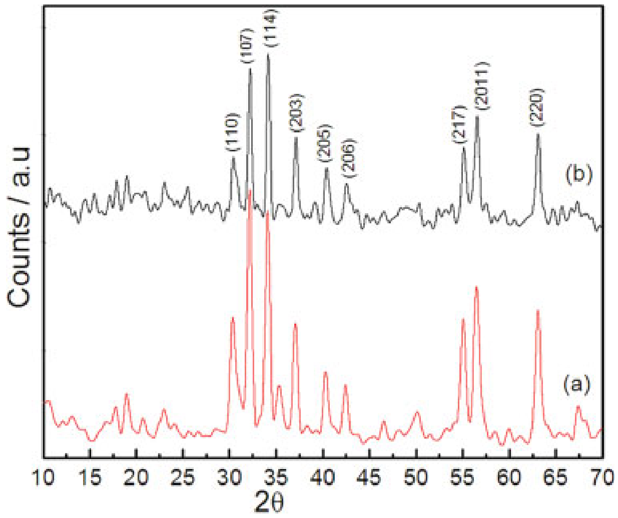


Figure 4 XRD data of barium ferrite (curve a) and poly(AnEDOT)-BaF (curve b). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the interaction of ferrite particles with the copolymer chain.

X-ray diffraction pattern of the $BaFe_{12}O_{19}$ and poly(AnEDOT)-BaF are shown in Figure 4, which matches with all peaks of standard pattern of barium ferrite (Powder Diffraction File, JCPDS No. 39-1433). The presence of all the peaks of barium ferrite in poly(AnEDOT)-BaF complex confirms the incorporation of ferrite particles in the polymer matrix. The broadness of the peaks in both patterns indicates about the small dimensions of the ferrite particles. The average crystallite size of barium ferrite particle was calculated by Scherrer's formula,

$$D = k\lambda/\beta\cos\theta$$

where D is the crystallite size for the individual peak, λ is the X-ray wavelength, k the shape factor, θ the Bragg angle in degrees, and β is the line broadening measured by half-height in radians. The value of k is often assigned a value of 0.89, which depends on several factors, including the Miller index of the reflecting plane and the shape of the crystal. From the above formula, the crystallite size of the barium ferrite particles is estimated as 24.84 nm for barium ferrite sample and 23.46 nm for poly(AnEDOT)-BaF complex. XRD pattern shows the hexagonal structure of barium ferrite with a = 5.894 Å and c = 23.25 Å in ferrite sample, while poly(AnEDOT)-BaF has lattice value a = 5.887 Å and c = 23.196 Å. It was observed that lattice spacing decreases as barium ferrite is incorporated in the polymer matrix, which may be responsible for the enhancement of the conductivity in poly(AnEDOT)-BaF complex.

TGA was carried out to see the thermal stability of poly(AnEDOT)-BaF complex with respect to copolymer poly(AnEDOT), polyaniline, and PEDOT. Thermogram of polyaniline (Fig. 5, curve a) has shown initial weight loss of 4.9% up to 100°C due to the loss of moisture content in the polymer matrix. From 100 to 230°C the polymer shows only 1% weight loss indicating that polymer is thermally stable up to 230°C, while 69% weight loss from 230 to 700°C shows the degradation of polymer backbone. Thermogram of PEDOT indicates that the polymer is thermally stable up to 180°C (Fig. 5, curve b) with initial weight loss of 3% up to 100°C. However, the copolymer poly(AnEDOT) was found thermally stable up to 210°C (Fig. 5, curve c) with initial weight loss of 5% which lies in between the polyaniline and PEDOT. The incorporation of ferrite particles in the copolymer matrix lead to increase in thermal stability up to 240°C (Fig. 5, curve d), which may be due to the interaction of barium ferrite with polymer matrix.

The DC conductivity measurement has been carried out using the compressed pellet of dimensions 13 mm × 7 mm with thickness of 2 mm by four-probe method. The room temperature conductivity of the samples is shown in Table I. The conductivity of 0.003 S/cm was observed for poly(AnEDOT), which is lower than polyaniline and PEDOT because of structural rearrangement of two monomer units. The less delocalization of polaron and bipolaron can arise through some structural conjunction in the polymer chain possibly caused by oxygen atom of ethylenedioxy unit present in EDOT, which interacts with nitrogen atom of aniline ring due to electrone-

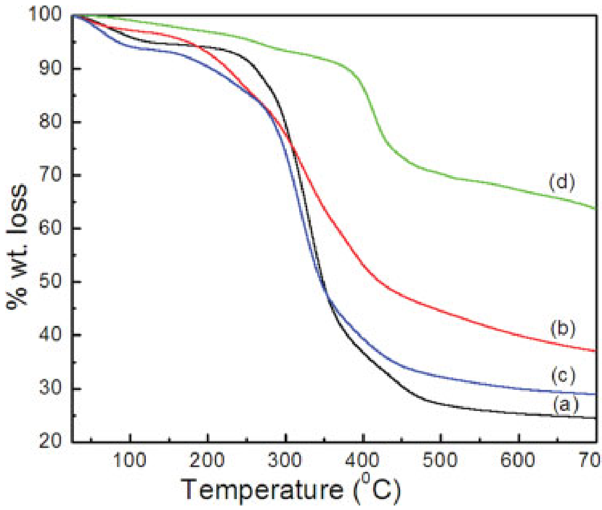


Figure 5 TGA data of polyaniline (curve a); PEDOT (curve b); poly(AnEDOT) (curve c), and poly(AnEDOT)-BaF (curve d) at a scan rate of 10°C/min under N_2. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

TABLE I
Anodic Peak Potential Values, Conductivity, and Magnetic Characteristics of Polyaniline, PEDOT, Poly(AnEDOT), Poly(AnEDOT)-BaF, and Barium Ferrite

	Peak potential values				Magnetic properties		
Sample name	E_{pa} (I) (V)	E_{pa} (II) (V)	E_{pa}(III) (V)	σ (S/cm)	M_s (emu/g)	H_c (G)	M_r (emu/g)
Polyaniline	0.115	0.540	0.774	2.27	0.25	7.3	5.3×10^{-2}
PEDOT	0.35	–	–	0.185	8.5×10^{-3}	0.0	9.2×10^{-3}
Poly(AnEDOT)	0.22	0.56	–	0.003	5.3×10^{-2}	0.0	3.2×10^{-2}
Poly(AnEDOT)-BaF	0.205	0.46	0.76	0.256	29.2	3,903	16.9
Barium ferrite	–	–	–	$\sim 10^{-8}$	51.5	3,229	29.2

gativity difference. In case of poly(AnEDOT)-BaF complex, the conductivity substantially increases up to 0.256 S/cm. The probable reason for this may be the interaction of ferrite constituent with nitrogen atom of the copolymer chain that leads to form some complex-like structure, resulting in the enhancement of conductivity.

VSM studies have revealed that the saturation magnetization value (M_s) of barium ferrite particles is found to be 51.5 emu/g (Fig. 6, curve a), whereas incorporation of ferrite particles in the polymer matrix leads to saturation magnetization (M_s) value of 29.2 emu/g (Fig. 6, curve b). From the hysteresis loop of barium ferrite and poly(AnEDOT)-BaF complex, it has been observed that the material possesses high retentivity (M_r) and coercive force (H_c) (Table I). The observed hysteresis loop shows little variation from the regular magnetization curve of barium ferrite that may be attributed due to annealing conditions, and also due to the increase in the iron concentration.[32] This type of behavior is observed in ferrites having high Curie temperature and due to the slow cooling of the sample which causes slight oxidation of the sample. It is also found that by changing the ratio of barium ferrite in the copolymer matrix, the coercive force remains same but the saturation magnetization (M_s) value increases with increase of ferrite content in the polymer matrix, which is due to the different alignment of magnetic dipoles in the polymer chain.

Figure 7 shows the variation of reflection loss (R_L) with frequency for poly(AnEDOT) and poly(AnEDOT)-BaF. It is calculated by using S parameters in the frequency range of 12.4–18.0 GHz using following equation:[33]

$$R = |S_{11}|^2 \tag{1}$$

$$R_L = 10 \log R \tag{2}$$

The S parameters are the scattering parameters by which reflection and transmission coefficients of the material can be defined. Here, S_{11} or S_{22} and S_{12} or S_{21} of two-port network system representing the reflection coefficient and transmission coefficient respectively. R_L calculated from eqs. (1) and (2) was found to vary from −6.3 to −2.6 dB for the

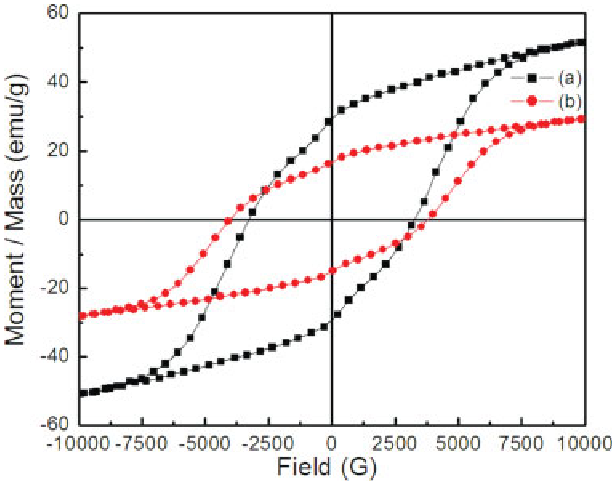


Figure 6 VSM plots of barium ferrite (curve a) and poly(AnEDOT)-BaF (curve b). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

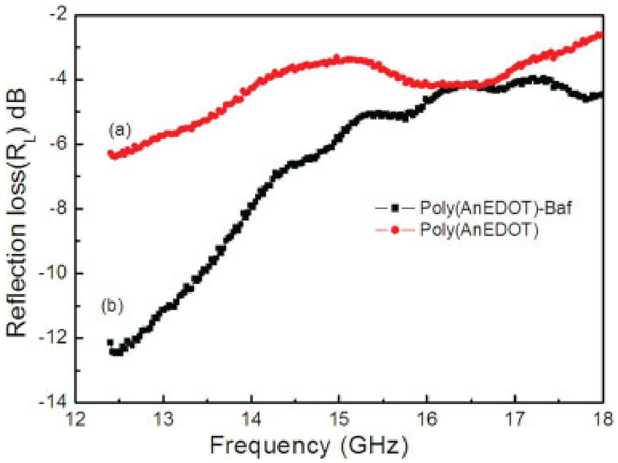


Figure 7 Reflection loss (R_L) of poly(AnEDOT) (a) and poly(AnEDOT)-BaF (b) in Ku-band (12.4–18.0 GHz). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

poly(AnEDOT). However, for poly(AnEDOT)-BaF, it varies from −12.1 to −4.5 dB. This shows that incorporation of the ferrite particles in the copolymer matrix leads to the improvement in the microwave absorption properties.

CONCLUSIONS

This study has shown that poly(AnEDOT) and its ferromagnetic complex poly(AnEDOT)-BaF synthesized by chemically oxidative polymerization possesses better processability and thermal stability. The cyclic voltammetric studies of the copolymer show the shifting of the peak potential values with respect to peak potential values obtained for the corresponding conducting polymers polyaniline and PEDOT, which indicates the formation of the copolymer. Furthermore, shifting of E_{pa} values of the copolymer in the presence of barium ferrite confirms the incorporation of ferrite particles in the copolymer matrix, which is also confirmed by FTIR studies. VSM and conductivity measurements of poly(AnEDOT)-BaF complex shows a M_s value of 29.2 emu/g and conductivity of the order of 0.256 S/cm respectively, which can be used for its applications in microwave absorption. Microwave measurements have shown a R_L of −12.1 dB for poly(AnEDOT)-BaF, while poly(AnEDOT) gives a R_L of −6.3 dB. This indicates that incorporation of barium ferrite in the copolymer matrix leads to enhancement in microwave absorbing properties.

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