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Synthesis, Spectral and Electrochemical Characterization of Adipic Acid Doped Polyaniline

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Abstract:

Polyaniline (PANI) doped with adipic acid was prepared by in-situ chemical polymerization method using various concentrations of adipic acid. The structural characterizations of PANI were done by Fourier transform infrared (FTIR) and Ultra violet visible spectroscopy (UV-Visible). XRD studies reveal crystalline nature of the doped PANI. Scanning electron microscopy (SEM) and Transmission electron microscopy (TEM) were used to characterize the morphology of the composites. Thermal studies indicate that highly doped PANI was more stable. Electrochemical characterization was made using cyclic voltammetry (CV) and capacitive behaviour was analysed by impedance measurements (EIS).

1. Introduction

Polyaniline (PANI), a conducting polymer though discovered 150 years ago, has attracted the scientific community only since the early 1980s for its numerous applications in practice. It can be synthesized easily by chemical oxidation [1] and electrochemical [2] processes. The polymer may occur in different redox states in which the half oxidized emeraldine base is regarded as the most useful form of polyaniline due to its high stability at room temperature [3]. The main disadvantage of polyaniline is its insolubility. Doping alters the properties of polyaniline. Emeraldine base, the insulating form is the only form of polyaniline which can be doped by acids [4]. Acid doping converts the insulating form to the conductive form, the emeraldine salt [5] and this is the most conducting form of polyaniline [6]. Solubility is also increased by doping [7] Doping with protonic acids is a non-redox doping in which the number of electrons in the polymer do not change [8].

A number of studies on doped polyanilines have been reported. The use of dodecyl benzene sulfonic acid and camphorsulfonic acid as doping agent increases the solubility in organic solvent and these can be used as a sensing agent for O₂ gas detection [9]. There is experimental evidence that polyaniline based coatings had offered ability of corrosion protection of metals [10]. It was reported that anthracene sulfonic acid surfactant dopant modulated PANI nanocomposites were used as biosensors [11]. Polyaniline-Langmuir-Blodgett films can be used as a biosensor for the detection of β -D-glucose [12]. Electroactive conductive polyaniline was also used in cell-culture experiments and biotechnological applications [13]. Polyaniline coated on wood sawdust has been used for removal of methylene blue dye from aqueous solutions by adsorption [14].

Capacitors are devices in which have energy storage capacity. Recent studies show that polyaniline in the form of emeraldine salt have capacitance and they can be used in supercapacitors. Capacitance of conducting polyaniline thin films [15], polyaniline doped with HCl [16], manganese [17], copolymer of Polyaniline and poly(*N*-methylaniline) [18], carbon/polyaniline composites [19] and polymer coated carbon nanotubes [20] have been reported.

In this work emeraldine salt of polyaniline doped with adipic acid was prepared by oxidative polymerization. The structure of the doped PANI was studied by FTIR and UV-Vis studies. The polymer was electrochemically characterized using CV. The surface morphology was studied by SEM and TEM and degree of crystallinity of polyaniline was studied by XRD. Thermal stability of the doped polymer was investigated by TGA. The capacitive behaviour was examined by Electrochemical Impedance Spectroscopy (EIS).

2. Experimental

2.1. Materials

Monomer of aniline (ANI) was obtained from e-Merck and used after distillation. Adipic acid (AA) used as dopant and potassium persulphate oxidizing agent were also obtained from e-Merck. Millipore and double distilled water were used for dilution of chemicals and washing purposes respectively.

2.2. Synthesis of Polyaniline Doped with Adipic Acid

Adipic acid doped polyaniline (PANI) was synthesized by *in-situ* chemical polymerization [21] of aniline in the presence of adipic acid as dopant and potassium persulphate as oxidant. For the synthesis, 500mL of adipic acid (different concentrations of dopant, 0.02, 0.04, 0.06, 0.08, 0.1M) and 5 ml of aniline (0.1M) were taken in a 1L flat bottomed flask equipped with a Teflon coated magnetic stirrer. Then 6.5 g of potassium persulphate was added to the above solution. The contents of the beaker were stirred for two hours, for completion of polymerization. The colour of the solution gradually changed from a pale green to a dark green colour. This was refrigerated for 24h for aging. Then the dark green precipitate obtained was filtered. The product was washed successively with double distilled water and acetone until the wash solution turned colorless. The product was dried to get powder form PANI.

2.3. Characterization

UV-VIS spectral studies were carried out using JASCO-V-530 UV-VIS spectrophotometer. FTIR was recorded on a Thermo Scientific Nicolet iS5 spectrometer equipped with ATR. X-ray diffraction patterns of the polymers were obtained by employing XPERT-PRO diffractometer using CuK_α ($k_\alpha = 1.54060$) radiation. The diffractometer was operated at 40Kv and 30mA. Powder X-ray diffraction pattern was recorded. The morphological study of the polymers was carried out using Scanning electron microscope (SEM Model: JEOL JSM 6360) operating at 25kV. TEM images were recorded using Philips CM 200 model with the operating voltage range of 20-200 and with a resolution of 2.4 Å. The cyclic voltammetric and impedance studies were done using CHI650C electrochemical workstation by three electrode configurations comprising of doped PANI coated ITO glass plate as a working electrode, a platinum plate as a counter electrode and Ag/AgCl as a reference electrode.

3. Results and Discussion

3.1. Solubility and UV-VIS Spectral Studies

The solubility of the prepared PANI was tested in a number of solvents. The results are presented in Table 1. PANI is soluble in a number of polar organic solvents, but insoluble in nonpolar organic solvents. The processibility of polyaniline has been limited due to its insolubility. This has been rectified by doping with adipic acid. The presence of large anions has increased the solubility by masking the hydrophobic part, i.e. aniline from coming into contact with the solvent.

Organic solvents	Solubility
DMF	Soluble
DMSO	Soluble
Chloroform	Soluble
Hexane	Insoluble
Benzene	Insoluble
Xylene	Insoluble
Toluene	Insoluble
Acetone	Soluble
carbon tetra chloride	Insoluble
Acetonitrile	Soluble
Ethanol	Soluble
Water	Partially soluble
NMP	Soluble

Table 1: Solubility of doped PANI

The UV-Vis spectra of PANI were recorded between the wavelengths 200 nm and 1100 nm. Fig 1. shows UV-VIS absorption spectra of PANI doped with different concentrations of adipic acid in ethanol. The spectra show the presence of two absorption bands as those of reported polyaniline, one in the UV region and the other in the visible region. It shows the characteristic bands due to π - π^* transition at 375nm and the band due to the inter-ring charge transfer associated with excitation from benzenoid to quinoid moieties at 550nm [22]. It is seen that as the concentration of dopant increases the absorption increases slightly. Low aniline to adipic acid ratio favors reduced state formation in the backbone of the polymer [23]. The doped polymer shows absorbance at 250nm due to the π - π^* transition of the doped carboxylic acids.

3.2. Band Gap Energy

The band gap energy calculation provides a method of evaluating the optical absorption of a material. The energy can be deduced from the formula $E = hc/\lambda$, where h is Planck's constant, c is velocity of light and λ is the wavelength. The band gap energy, calculated for the absorption at 375nm and 250nm is found to be 3.48eV and 4.97eV respectively, which shows the doped PANI could be used for optoelectronic, high frequency and high power devices.

3.3. FTIR studies

The results of FT-IR (Fig 2) show that polyaniline backbone is in the form of emeraldine salt. The C-N stretch of the benzene -NH group appears around 1290 cm^{-1} . Vibrations of protonated imine groups are shown around 1148 and 1128 cm^{-1} and they are

responsible for conductivity of polyaniline. The symmetric N-H stretching vibration is seen around 3200cm^{-1} . The aliphatic C-H stretch of the doped adipic acid is seen at 2850cm^{-1} . All characteristic bands coincide well with the literature [24-28]. The characteristic -C=O stretching vibration of acid shifts to a lower frequency, after doping due to delocalization that lowers the atomic force constant of the polymer chain [29]. The presence of carboxylate as anion is seen by the two characteristic coupled carbonyl absorption bands at 1650cm^{-1} and 1450cm^{-1} . The skeletal vibrations of para coupled rings are seen at 800cm^{-1} .

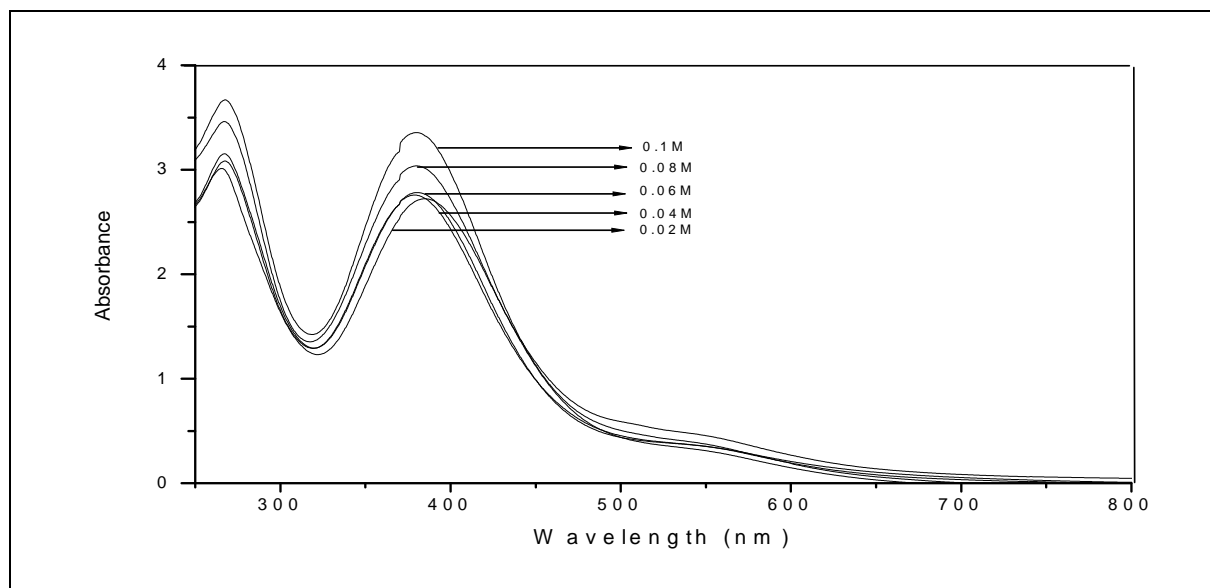


Figure 1: UV-VIS spectra of adipic acid doped PANI

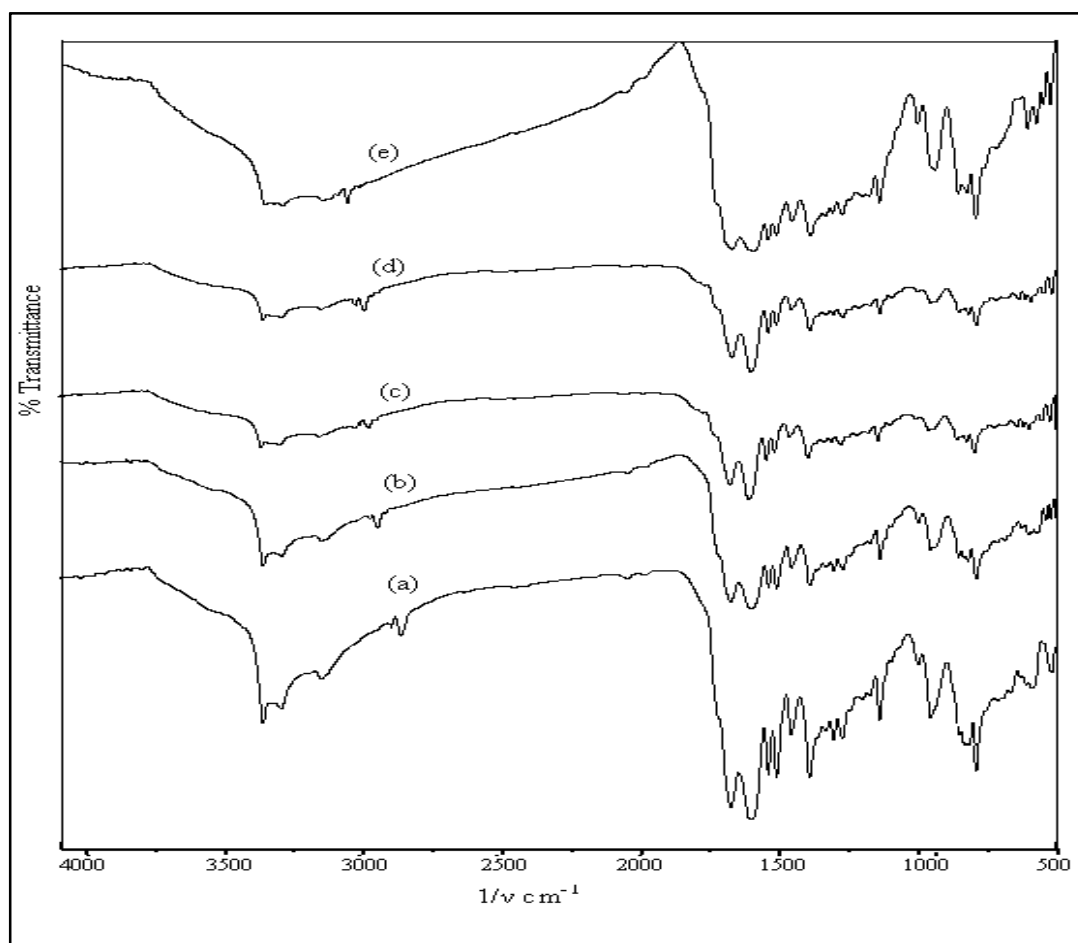


Figure 2: FT-IR behavior of PANI doped with adipic acid of different concentrations a) 0.1 b) 0.08 c) 0.06 d) 0.04 e) 0.02M

3.4. XRD Studies

The structural analyses were carried out by X-ray diffractometer and given in figure 3. From the value of 2θ , d values were calculated using Bragg's equation, $2d \sin \theta = n \lambda$. The values are presented in Table 2. The XRD of PANI prepared by different methods have been reported to have amorphous to crystalline nature with different percentage of dopant [30], but here the synthesized polymers have crystalline structure due to planar nature of benzenoid and quinoid functional groups. The d spacing values are found to be around 13.5, 4.8, 4.5, 3.82 and 3.5 for the sharp bands, which is similar to the values predicted for polymers of PANI, where there is distance between adjacent planes having benzene rings [31]. The XRD patterns show peaks at $2\theta = 6.5, 18$ and 25 . The 2θ values of 18 and 25 are common for polymers of polyaniline having periodicity parallel to the polymer chain and perpendicular to the polymer chain respectively. A sharp peak at $2\theta = 6.50^\circ$, attributed to the separating aliphatic chains[32] and showing the orientation of the dicarboxylic acids in the polymeric chain, is observed in the XRD spectrum of nanostructured PANI. The peak at $2\theta = 6.5^\circ$ may be due to the distance between two stacks in a 2D arrangement of polymer chains with the dopant ions in between the stacks [33]. The intensity of this peak is seen to increase with increase in the [adipic acid] / [aniline] ratio. The average grain size is calculated using Debye Scherrer equation $D = [0.94 \lambda] / [\beta \cos \theta]$ where β is full width of half maxima (FWHM) and λ is 0.154nm. The average grain size is found to be 3.5, 3.9, 3.4, 3.04, 4.1 nm for PANI doped with adipic acid of concentrations 0.1M, 0.08M, 0.06M, 0.04M and 0.02M respectively. Moreover, as the dopant concentration increases the peaks are found to be sharper which shows that dopant affects the morphology of the polymer. Thus, the high crystallinity might result from the presence of the large anion with two $-\text{COO}^-$ groups of the dicarboxylic acid.

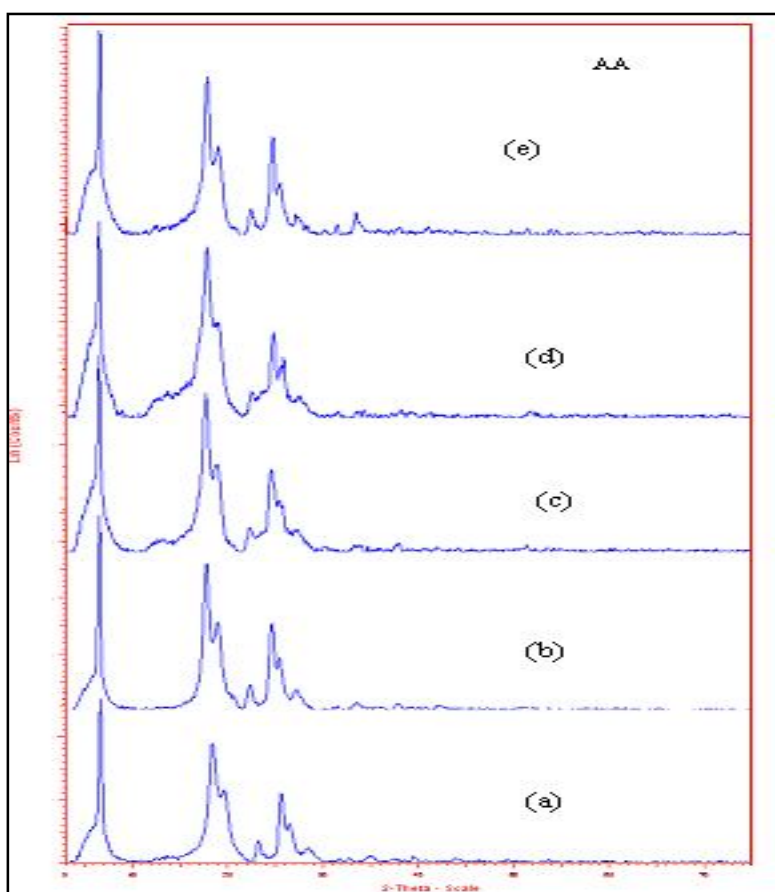


Figure 3: XRD pattern of PANI doped with adipic acid of different concentrations a) 0.02 b) 0.04 c) 0.06 d) 0.08 e) 0.1M

[Adipic acid] M	2θ	d spacing \AA	Average particle size (nm)
0.02	6.56, 18.337, 19.5, 25.56	13.46, 4.83, 4.3, 3.5	3.60
0.04	6.57, 28.4, 19.6, 23.2, 25.6	13.43, 4.8, 4.5, 3.82, 3.46	4.00
0.06	6.55, 18.35, 19.5, 23.3, 25.6	13.48, 4.83, 4.52, 3.8, 3.47	3.50
0.08	6.5, 18.32, 23.2, 25.63	13.6, 4.84, 3.84, 3.47	3.00
0.1	6.45, 18.35, 23.19, 25.6	13.67, 4.82, 3.8, 3.47	4.10

Table 2: XRD data of adipic acid doped PANI.

3.5. SEM, TEM and EDX

SEM and TEM analysis was carried out to study the morphology of the doped polymer. The results are given in figure 4. The SEM micrographs reveal that the polymer is aggregated due to normal filtration. TEM of PANI doped with 0.06M adipic acid clearly shows nanobud like structure with a size of 100nm. The presence of adipic acid in the reaction medium facilitates the formation of micelles due to the presence of hydrophilic $-\text{COOH}$ groups. Aniline enters these micelles and polymerization takes place in these micelles in the presence of oxidant. When polymerization is complete the doped polymer comes into the water due to the presence of hydrophilic groups[21]. The bud like structure may be due to the presence of polymer chains in the core surrounded by the large carboxylate anions. The result of EDX shows the presence of C, O and N in the polymer. The presence of oxygen confirms the presence of acid as dopant in the polymer.

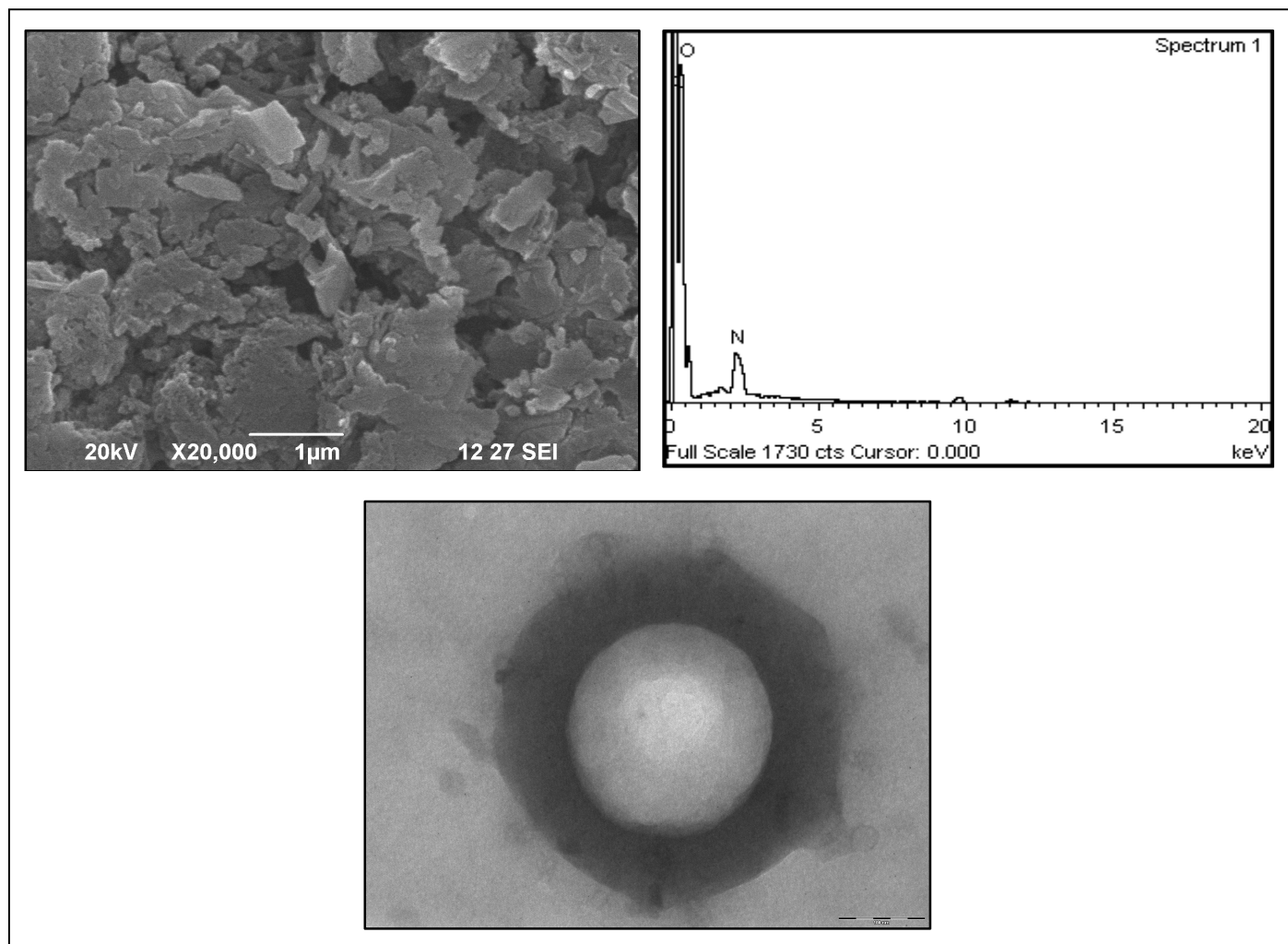


Figure 4: Micrographs of polyaniline doped with 0.06M adipic acid (A) SEM and EDX (B) TEM

3.6. Thermogravimetric Analysis

The TGA of adipic acid doped polyaniline at 0.02M and 0.1M concentration is shown in figure 5. Thermogram clearly shows that high concentration of dopant (0.1M) has more thermal stability and it exhibits single step degradation, but at low concentration (0.02M) of doped adipic acid it is two step degradation and it has a lower thermal stability. Literature studies show that polyaniline emeraldine base shows more thermal stability than doped polyaniline and composites [34, 35]. The thermogram of polyaniline emeraldine base form shows a weight loss of 7-8% upto 100°C, a weight loss of 22% from 100-450°C and a weight loss of 40% from 450-800°C [36].

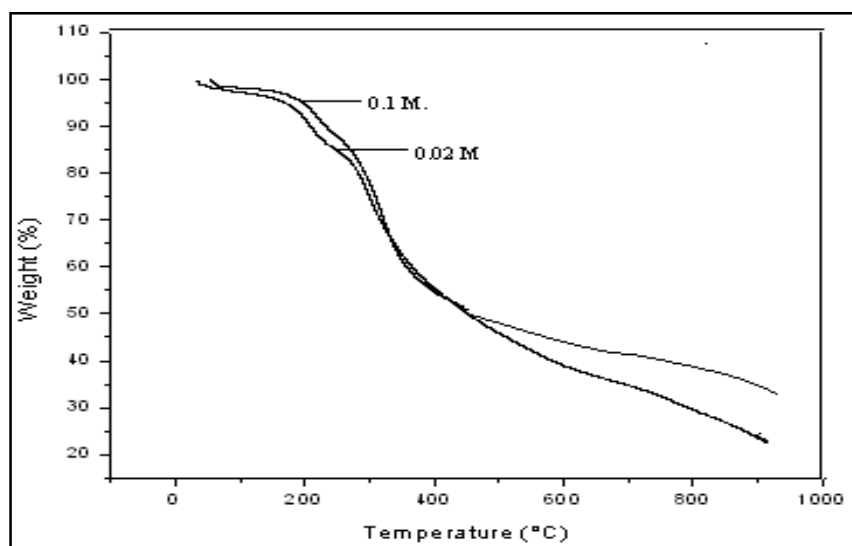


Figure 5: Thermogram of PANI doped with adipic acid of concentration 0.1M and 0.02M

For PANI doped with adipic acid there is no significant loss in weight upto 100°C. There is a degradation and loss in weight between 100 - 200°C. For both concentrations of dopant between 200-400°C, there is 30% decrease in weight. Above 400°C for 0.1M dopant the loss in weight is very slow while for 0.02M it is very rapid. This shows that as dopant concentration increases polyaniline stability also increases. So polyaniline in the emeraldine salt form is stabilized by doping with the large anion.

3.7. Cyclic Voltammetry

Cyclic voltammograms of polyaniline doped with different concentrations were recorded between -0.6V and 1.6V at a scan rate of 0.025V/s and presented figure 6. CV of polyaniline generally shows two reduction and two oxidation peaks due to the exchange between the leucoemeraldine/emeraldine and emeraldine/parnigraniline states of the PANI [31]. The leucoemeraldine to emeraldine salt oxidation is observed at about 0.22V versus SCE for undoped Pani [14]. All doped polyanilines show reduction and oxidation peaks in the corresponding region with slight difference in current. With increase in concentration of adipic acid the oxidation peak is shifted to higher potential and the area under the curve decreases. An oxidation peak is seen at 0.9V and reduction peaks are seen at 0.25V and -0.15V.

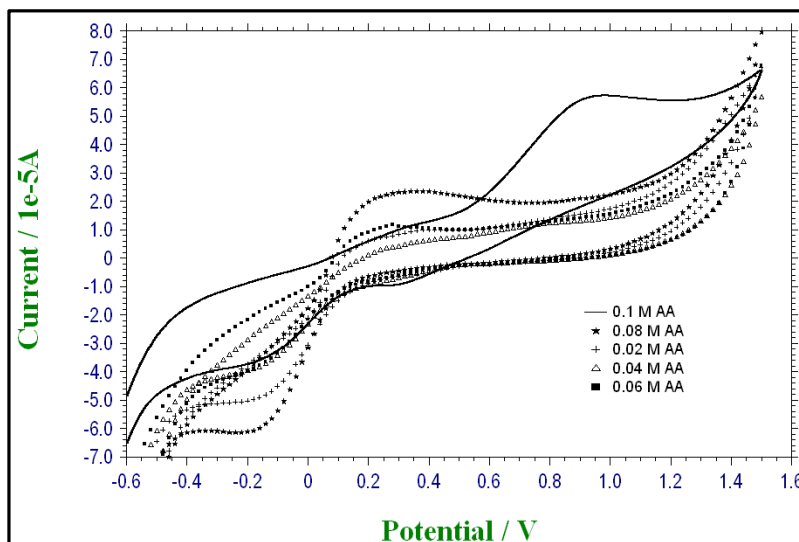


Figure 6: Cyclic voltammogram of adipic acid(AA) doped PANI coated on ITO plate

3.8. Electrochemical Impedance Spectroscopy (EIS) Studies

EIS is a powerful technique complementary to the galvanostatic cycling measurement which gives more information on the electrochemical frequency behavior of the system. The Nyquist plot for a Randles cell is a semicircle. The solution resistance can be found by reading the real axis value at the high frequency intercept near the origin of the plot (Fig 7). The real axis value of the other intercept (low frequency) is the sum of the polarization resistance and the solution resistance. The diameter of the semicircle is equal to the polarization or the charge transfer resistance. The charge transfer resistance R_{ct} and double layer capacitance C_{dl} only describe the resistance (Table 3) and capacitance provided by the electrochemical double layer at the interface [34]. In the Nyquist impedance

plot the imaginary part of impedance is plotted against its real component in the frequency range from 1MHz to 0.1Hz. These two resistances are plotted against the concentration. The decrease in the radius of the semicircle indicates the decrease in the resistance. It is seen that as the ratio of adipic acid increases the radius of the semicircle decreases, which shows that the resistance decreases and as a consequence, the double layer capacitance increases. Polyaniline doped with 0.06M and 0.04M adipic acid shows a peculiar increase in the radius of the semicircle which means it has higher charge transfer resistance.

Concentration of adipic acid (M)	Bulk Resistance R_s (k Ω)	Charge transfer Resistance R_{CT} (k Ω)	Double layer Capacitance C_{dl} (μ F)
0.10	53.63	9.156	28.77
0.08	57.79	7.007	20.11
0.06	310.4	12.11	17.79
0.04	71.84	12.92	12.69
0.02	57.18	8.085	16.48

Table 3: Data of electrochemical impedance spectroscopy

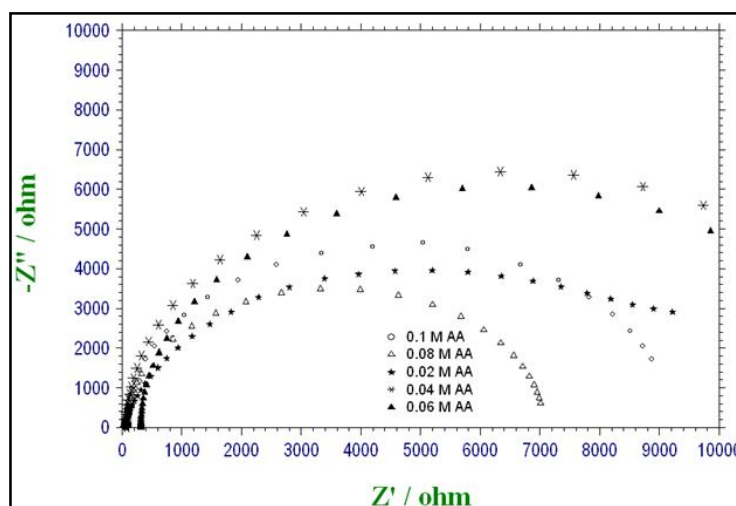


Figure 7: Nyquist plot of adipic acid(AA) doped PANI coated on ITO plate

4. Conclusion

Polyanilines doped with different concentrations of adipic acid were synthesized and characterized. Adipic acid behaves as an acid and helps in formation of emeraldine salt of polyaniline. The results of various characterization prove that PANI is in the emeraldine salt form. Thus doping increases the solubility and the processibility of PANI. It is also observed that with more aniline to adipic acid ratio the particles are fine, nanosized and crystalline. They show good capacitive behaviour and can be used as good supercapacitor material.

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