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An electrochemical investigation of novel optically active poly(amide-imide)s based on natural amino acids using multi-wall carbon nanotubes paste electrode

Shadpour Mallakpour · Mehdi Hatami · Ali A. Ensafi · Hassan Karimi Maleh

Abstract The main aim of this research was to study the electrochemical behavior of novel optically active poly (amide-imide)s (PAIs). Polycondensation reactions of a 3,5-diamino-N-(4-hydroxyphenyl)benzamide with different synthetic diacid chlorides derivatives based on natural amino acids resulted in preparation of five different aromatic–aliphatic PAIs. These polymers were characterized by Fourier transform infrared, proton nuclear magnetic resonance, and elemental analyses. Also, we used electrochemical impedance spectroscopy for the evaluation of these novel optically active PAIs for the first time. Presence of p-substituted phenol groups in the structure of these polymers has been used for electrochemical investigation. Results showed that the oxidation currents in five diverse polymers were affected by their structures. In addition, the influence of carbon nanotubes on the oxidation of phenolic groups was studied using carbon paste matrix in an aqueous buffer solution (pH 7.0).

Keywords Optically active poly(amide-imide)s · Electrochemical investigation · Multi-wall carbon nanotubes paste electrode · Electrochemical impedance spectroscopy

Introduction

Wholly aromatic polyamides (aramides) are characterized by their outstanding balance of thermal and mechanical properties, which make them constructive as high-performance materials for advanced technologies [1]. However, the application of aramides is often difficult due to problem of processability. Modification of high-performance materials by increasing the solubility and lowering the transition temperatures while maintaining thermal stability is of meticulous interest [2].

It is known that the solubility of polymers is often increased when flexible bonds such as [–O–, –SO$_2$–, –CH$_2$–, –C(CF$_3$)$_2$-], bulky pendant groups (such as t-butyl and adamantyl), or polar constituents are incorporated into the polymer backbone due to the altering crystallinity and intermolecular interactions [3]. Chiral centers in materials are very important in biological systems, and as a result, the synthesis and application of optically active polymers have been the focus of attention recently. Several methods have been used for the preparation of optically active polymers. Among them, the uses of optically active monomers have been of great interest [4–6]. Amino acids are valuable building blocks in organic synthesis for introducing chiral unit in polymer structures. Polymers containing amino acid linkages are

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expected to show biocompatibility and biodegradability similarly to those of polypeptides.

In recent years, strenuous studies have been carried out to investigate electrochemical behavior of polymer structures [7–10]. Diverse strategies have been employed for the modification of the electrodes [11–20]. Chemically modified electrodes are very attractive for study of insoluble compounds in aqueous solution due to their fast, selective, reproducible, sensitive response and low cost for a chemical analysis. Among the many different approaches used for the alteration of an electrode, move toward of preparing bulk-modified electrode materials has been of particular interest. For this, a solid electroactive is mixed with a carbon composite matrix such as carbon paste, carbon powder-epoxy resin, or carbon nanotubes (CNTs) paste [12, 16, 20]. It has been shown that bulk modification is flexible and easy to execute. When the electrode surface has become inactive by, e. g., poisoning of the electroactive surface by impurities, a fresh surface layer containing the electroactive species is easily obtained by polishing.

Since the discovery of CNTs in 1991 [21], they have attracted many scientists in the fields of physics, chemistry, and material science. CNTs are of tremendous current interest. The structure of them is peculiar and different from graphite and traditional carbon fibers. CNTs are built from sp^2 carbon units and are seamless structure with hexagonal honeycomb lattices. They have closed topology and tubular structure and are typically several nanometers in diameter and many microns in length. The subtle electronic properties recommend that CNTs will have the ability to mediate electron transfer reactions with electroactive species in solution when used as the electrode material [22], so CNTs based electrodes could be used in electrochemical sensing and in investigation behavior of compounds that are insoluble in aqueous solution [15]. Electrode materials CNTs showed better behavior than traditional carbon electrodes, including good conducting ability and high chemical stability. Therefore, in the first step, we described the synthesis of novel optically active poly(amide-imide)s (PAIs) and then, in the next step, we use voltammetric methods such as cyclic voltammetry (CV) and electrochemical impedance spectroscopy for the study of electrochemical behavior of these polymers at the surface of carbon paste electrode (CPE) and especially CNTs paste electrode. To our knowledge, no paper has been reported about the using of electrochemical impedance spectroscopy with carbon paste and especially CNT paste electrodes for the oxidation behavior of optically active PAIs. In this paper, we used different electrochemical techniques for study of the oxidation behavior of the pendant *p*-substituted phenol units in novel optically active PAIs structure for the first time.

Experimental

Chemicals

All chemicals used were of analytical reagent grade purchased from Fluka Chemical Co. (Buchs, Switzerland), Aldrich Chemical Co. (Milwaukee, WI), Riedel-deHaen AG (Seelze, Germany), and Merck (Darmstadt, Germany) unless otherwise stated. Doubly distilled water was used throughout.

4-Aminophenol, 3,5-dinitrobenzoylchloride, *N,N*-dimethyl acetamide (DMAc), and propylene oxide from Merck were used for the synthesis of mediators. DMAc as a solvent (d=0.94 g cm^{-3} at 20 °C) was distilled over barium oxide under reduce pressure. Other reagents were used without further purification.

Phosphate buffer (sodium dihydrogen phosphate and disodium monohydrogen phosphate plus sodium hydroxide, 0.1 mol L^{-1}) solutions with different pH values were used.

High-viscosity paraffin (d=0.88 Kg L^{-1}) from Merck was used as the pasting liquid for the preparation of CPE. Spectrally pure graphite powder (particle size <50 μm) from Merck and CNT (>90% multi-wall CNT (MWCNTs) basis, $d×l$=(70–110 nm)×(5–9 μm) were used as the substrate for the preparation of the CPE as a working electrode (WE).

Apparatus

Proton nuclear magnetic resonance (^{1}H-NMR, 500 MHz) spectrum was recorded in dimethyl sulfoxide (DMSO)-d_6 solution using a Bruker (Germany) Avance-500 instrument. Proton resonances are designated as singlet (*s*), doublet (*d*), and multiplet (*m*). Fourier transform infrared (FT-IR) spectra were recorded on Jasco-680 spectrophotometer (Japan). The spectra of solids were obtained using KBr pellets. The vibrational transition frequencies are reported in wavenumbers (per centimeter). Inherent viscosities were measured by using a Cannon-Fenske Routine Viscometer (Germany) at concentration of 0.5 g dL^{-1} at 25 °C. Specific rotations of polymer samples were measured by a Jasco Polarimeter (Japan). Elemental analyses were recorded on Leco, CHNS-932 by Isfahan University, Isfahan, Iran. Cyclic voltammetry (CV) was performed in an analytical system, micro-Autolab, potentiostat/galvanostat connected to a three-electrode cell, Metrohm Model 663 VA stand, linked with a computer (Pentium IV, 1,200 MHz) and with micro-Autolab software. In addition, impedance spectroscopy was performed in an analytical system using an Autolab PGSTAT 12, potentiostat/galvanostat connected to a three-electrode cell, Metrohm Model 663 VA stand, linked with a computer (Pentium IV, 1,200 MHz) and with Autolab software. The system was run on a PC using GPES and FRA 4.9 software. For impedance measurements, a
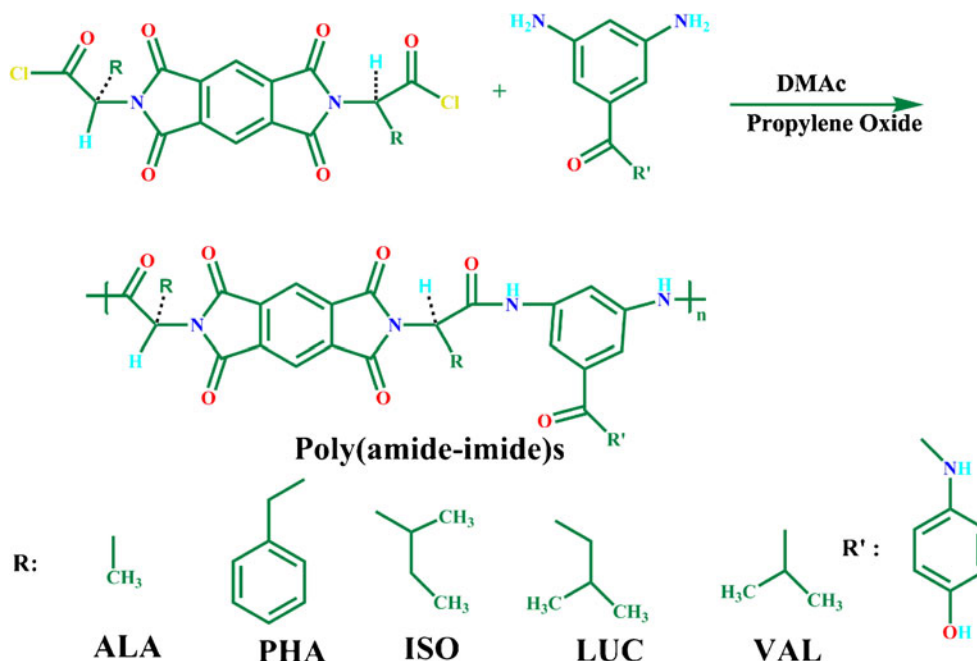
Scheme 1 Preparation of optically active PAIs from the reaction of diamine with optically active diacid chloride

frequency range of 100 kHz to 1.0 Hz was employed. The AC voltage amplitude of 5 mV was used, and the equilibrium time was 10 min. A conventional three-electrode cell assembly consisting of a platinum wire as an auxiliary electrode and an Ag/AgCl (KCl$_{sat}$) electrode as a reference electrode was used. The WE was either a CNTs paste electrode modified with polymers or CPE modified with polymers. The prepared electrodes with CNTs and polymers were characterized by scanning electron microscopy (SEM; XLC Philips).

A pH meter (Corning, Model 140) with a double junction glass electrode was used to check the pH of the solutions.

Preparation of monomers and PAIs

3,5-Diamino-*N*-(4-hydroxyphenyl)benzamide (DNHB) as a diamine monomer was prepared according to report procedure by In et al. [23].

N,N′-(pyromellitoyl)-bis-*L*-α-amino acids and *N,N′*-(pyromellitoyl)-bis-*L*-amino diacid chlorides were prepared according to our previous reports [24–28].

A typical synthesis of PAIs was carried out as follows: A 100 mL, two-necked, round-bottomed flask equipped with a magnetic stirrer, nitrogen gas inlet tube, and calcium chloride drying tube was charged with 5 mmol of the diamine and 10 mL of dry DMAc. The solution was stirred at 0 °C for 0.5 h. Then, 1 mL of propylene oxide was added, and after a few minutes, 5 mmol of diacid chloride was added and the mixture was stirred at 0 °C for 0.5 h. The solution was stirred for 6 h at room temperature, and then it was precipitated in water. The polymer was filtered, washed with warm water and methanol, and dried at vacuum oven at 100 °C.

Preparation of the electrode

MWCNTs were refluxed in a 2.0 M HNO$_3$ for 20 h, and then was washed with twice-distilled water and dried at

Table 1 Elemental analysis of DNHB-ISO polymer

Polymer	Formula	Elemental analysis (%)				Moisture intake (%)[a]
			C	H	N	
DNHB-ISO	C$_{35}$H$_{33}$N$_5$O$_8$	Calculated	64.51	5.10	10.75	4.4
		Found	62.00	5.51	10.23	
		Corrected[b]	64.72	5.26	10.68	

[a] Moisture intake$(\%) = (W - W_0)/W_0) \times 100$, where W is the weight of polymer sample after standing at room temperature and W_0 is the weight of polymer sample after dried in vacuum at 100 °C for 10 h

[b] Corrected value for C, N, and S: found value×(100+moisture intake)/100, and corrected value for H: found value×(100−moisture intake)/100

Table 2 Some physical properties of PAIs

Polymer	Yield (%)	η_{inh} (dL g^{-1})[a]	$[\alpha]^{25,b}_{Na,589}$	Color
DNHB-ALA	84.69	0.21	−22.7	Orange
DNHB-PHA	82.41	0.30	−105.0	Orange
DNHB-ISO	81.15	0.48	−21.3	Orange
DNHB-LUC	85.13	0.24	−23.6	Orange
DNHB-VAL	78.48	0.18	−63.0	Orange

[a] Measured at a concentration of 0.5 g dL^{-1} in DMF at 25 °C

[b] Measured under the conditions the same as inherent viscosity

Fig. 2 Cyclic voltammograms of different polymers in CNTs paste matrix *a* DNHB-ISO, *b* DNHB-PHA, *c* DNHB-LUC, *d* DNHB-ALA, and *e* DNHB-VAL in PBS (pH 7.0) at a scan rate of 100 mV s^{-1}

room temperature. Then, 6.0 mg of the polymers was hand-mixed with 84.0 mg of graphite powder and 10.0 mg of CNTs in a mortar and pestle. Using a syringe, 0.50 g of paraffin was added to the mixture and was mixed well for 40 min until a uniformly wetted paste was obtained. The paste was then packed into a glass tube. Electrical contact was made by pushing a copper wire down the glass tube into the back of the mixture. When necessary, a new surface was obtained by pushing an excess of the paste out of the tube and polishing it on a weighing paper.

Recommended procedure

The MWCNTs paste electrode was polished with a white and clean paper. To prepare a blank solution, 10.0 mL of phosphate buffer solution (PBS, pH 7.0), was transferred into an electrochemical cell. The initial and final potentials were adjusted to 0.00 and +0.80 V vs. Ag/AgCl, respectively. The cyclic voltammograms were recorded with scan rate 100 mV s^{-1} to obtain signals.

Stability and reproducibility

The repeatability and stability of the polymers in CNTs paste matrix were investigated by CV measurements. The relative standard deviation (RSD%) for five successive assays is 3.5%. When using four different electrodes, the RSD% for five measurements is 3.2%. When stored in a laboratory, the modified electrode retains 96% of its initial response after a week and 92% after 30 days.

Results and discussion

Polymer synthesis

A series of new optically active PAIs were prepared from an equimolar mixture of diamine monomer and five
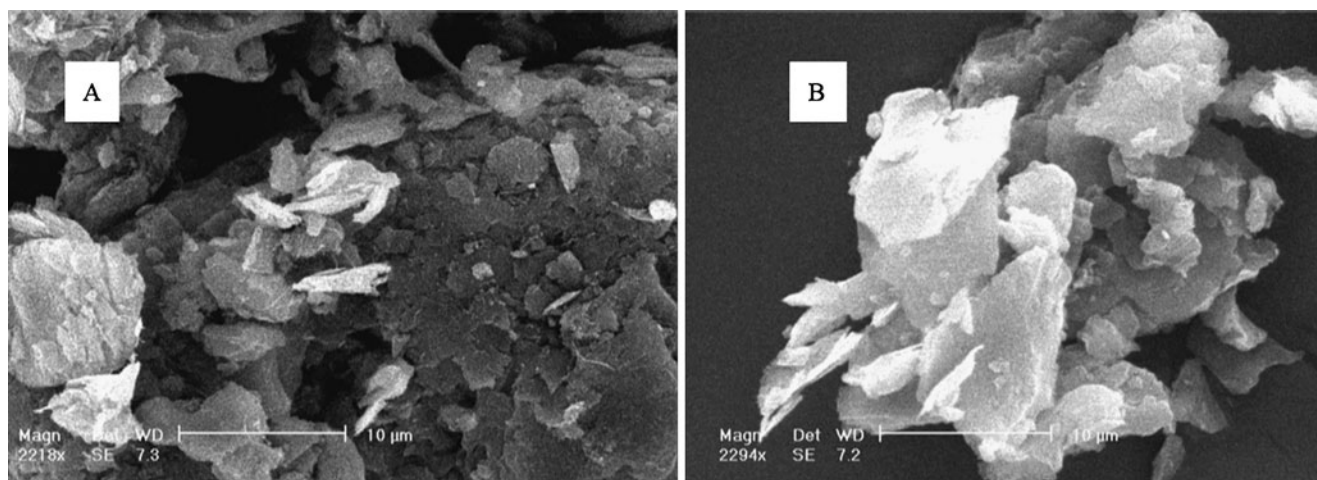

Fig. 1 SEM image of **a** DNHB-PHA-modified carbon nanotubes paste electrode. **b** DNHB-ALA-modified CPE

different diacid chlorides derived from *L*-amino acids including phenylalanine (PHA), leucine (LUC), isoleucine (ISO), valine (VAL), and alanine (ALA) under condensation reactions (Scheme 1). Synthetic diamine as a nucleophile reacts with diacid chloride as an electrophile in the presence of propylene oxide. HCl as a byproduct of polymerization process was trapped by propylene oxide.

Structures of these PAIs were characterized by FT-IR, ^{1}H-NMR, and elemental analyses. As a representative, FT-IR spectrum of DNHB-ISO polymer showed bands around 3,336–3,110 cm^{-1} (N–H), 3,025 cm^{-1} (Ar–CH), 2,965 cm^{-1} (aliphatic C–H), 1,775 (imide –C=O unsymmetrical stretching), 1,722 cm^{-1} (imide –C=O symmetri-

cal stretching), 1,664–1,660 cm^{-1} (N–H stretching of –NHCO), 1600 (N–H), 1,383 cm^{-1} (imide C–N stretching), and 756 cm^{-1} (–C=O out of plane deformation). ^{1}H-NMR spectrum of DNHB-ISO polymer showed peaks at ^{1}H-NMR (DMSO-d_6): δ10.29 (*s*, 2H, NH), 10.16 (*s*, 1H, NH), 9.19 (*s*, 1H, OH), 8.30(*s*, 2H, phenyl), 7.95(*d*, 1H, phenyl, *J*=8.5 Hz), 7.71 (*d*, 2H, phenyl, *J*=8.5 Hz), 7.44 (*d*, 2H, phenyl), 6.67 (*d*, 2H, phenyl, *J*=8.5 Hz), 4.73 (*d*, 2H, aliphatic, *J*=7.65 Hz), 2.67 (*s*, 2H, aliphatic), 1.52 (*s*, 4H, aliphatic), 1.02 (*m*, 6 H, aliphatic), and 0.92 (*s*, 6H, aliphatic).

Also, due to the presence of polar groups in the structure of polymers, they showed moisture uptake characters.
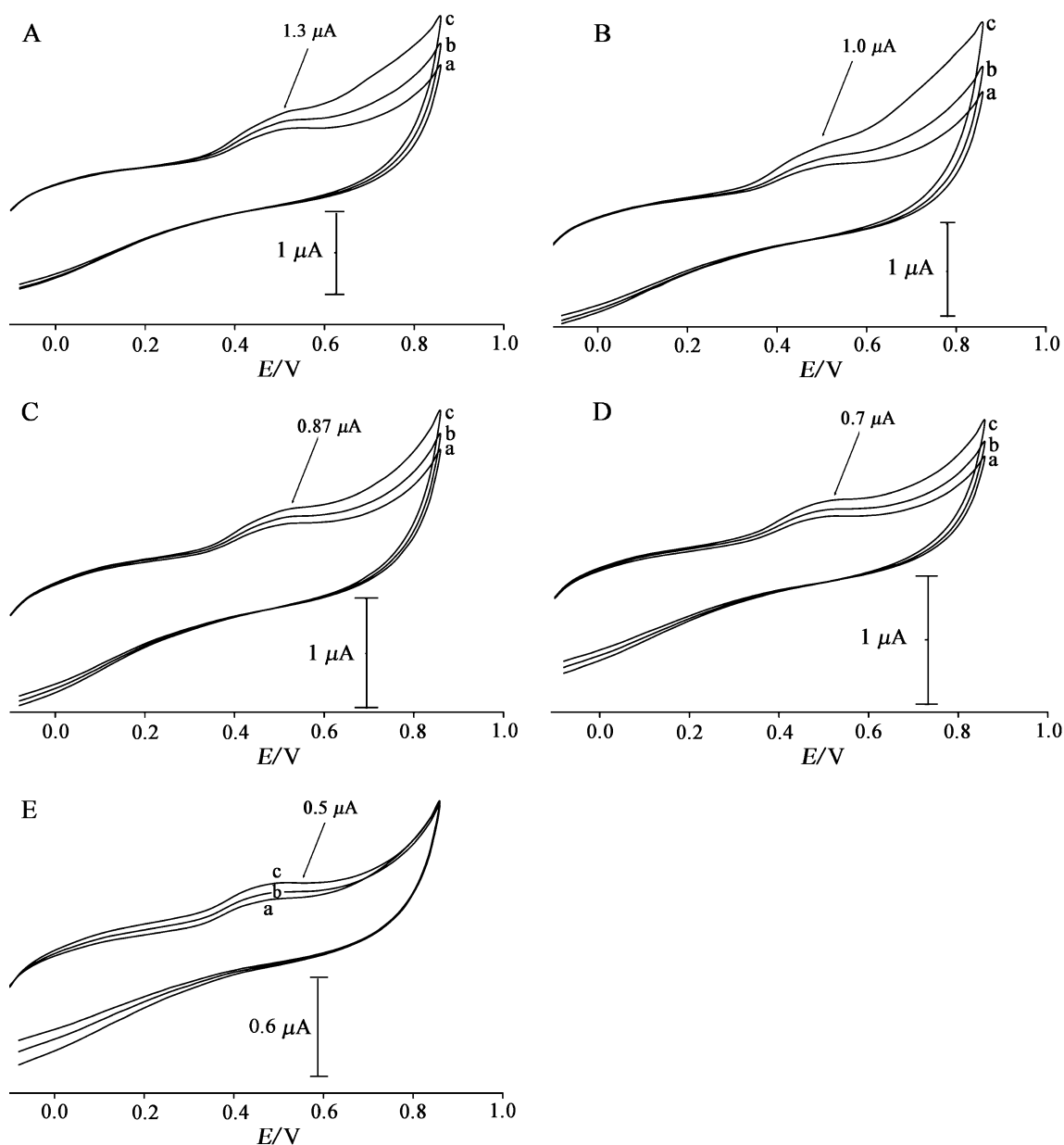
Fig. 3 Cyclic voltammograms of modified electrodes with different amounts of polymers in CNTs paste matrix for **a** DNHB-ISO, **b** DNHB-PHA, **c** DNHB-LUC, **d** DNHB-ALA, and **e** DNHB-VAL; *a* 5%, *b* 7%, and *c* 9% in PBS (pH 7.0) at a scan rate of 100 mV s^{-1}

Elemental analysis data of PAI based on isoleucine amino acid (DNHB-ISO) as a representative polymer is listed in Table 1.

The yield, inherent viscosity, optical rotation, and color of the obtained PAIs are collected in Table 2.

SEM characterization

Scanning electron microscope (SEM) technique as powerful and robust method was used to study the morphology of PAI-carbon paste and as well as MWCNTs paste.

Figure 1a shows the presence of DNHB-PHA in the CNTs paste matrixes. As can be seen in Fig. 1a, the layers of irregularly flakes of graphite powder are present and are isolated from each other. However, MWCNTs and DNHB-PHA were distributed homogeneously on the surface of electrode with special three-dimensional structure. Figure 1b is similar to Fig. 1a without the presence of MWCNTs. As a result, the presence of CNTs in modified electrode structure can successfully fill the empty space of modified electrode and increase oxidation current of the phenolic groups.
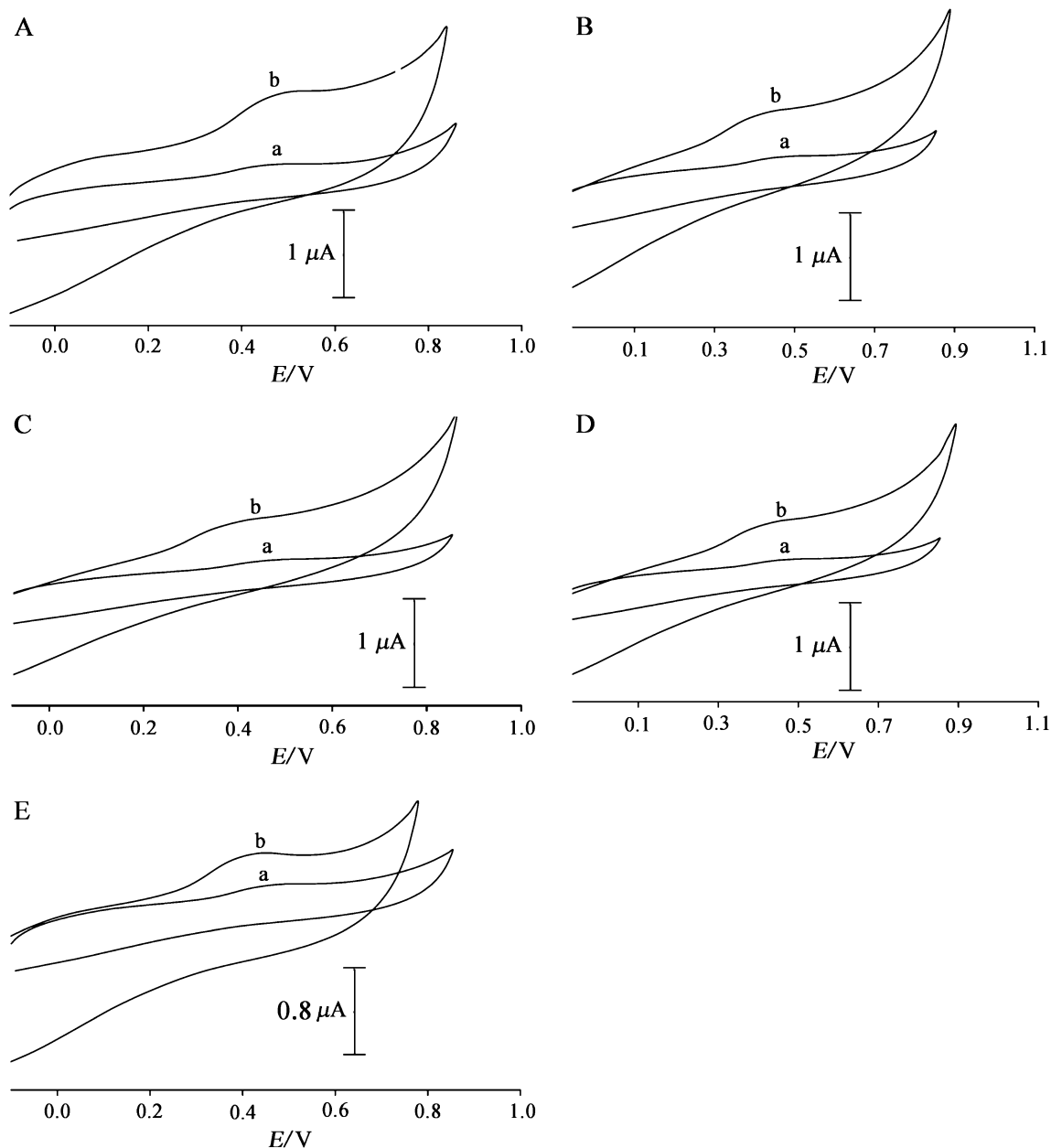
Fig. 4 Cyclic voltammograms of **a** DNHB-ISO, **b** DNHB-PHA, **c** DNHB-LUC, **d** DNHB-ALA, and **e** DNHB-VAL at a surface of *a* CPE and *b* at a carbon nanotubes paste electrode in PBS (pH 7.0) at a scan rate of 100 mV s^{-1}

Cyclic voltammetry investigation

Hydroxyl group in phenol structure can be oxidized at a suitable potential in electrochemical process [29]. Therefore, based on the structure of the designed polymers, we studied influences of hydroxyl units in the side chain of polymers as an electroactive species. On the other hand, we know that the physical and chemical properties of these polymers have close relationship with electrochemical behavior. One of the more important parameters that were measured in the structure of these polymers and have close relationship with electrochemical properties was inherent viscosity. According to inherent viscosity data, these polymers have different lengths. Therefore, with increasing

the chain lengths of the obtained polymers, the numbers of –OH phenolic groups are increased. This point shows that for these polymers, phenolic current are different and distinguishable. Figure 2 shows cyclic voltammograms of different polymers at CNTs paste matrix. As can be seen, for DNHB-ISO, the oxidation peak current is bigger than the others (3.19 µA) (curve e).

In addition, the currents for DNHB-PHA, DNHB-LUC, DNHB-ALA, and DNHB-VAL are 2.29, 1.92, 1.70, and 1.44 µA, respectively. This is very interesting, since these data have very good accordance with viscosity information.

For more study, the effect of polymer concentration in CNTs paste matrix was investigated. The results showed that with increasing the percent of these compounds in
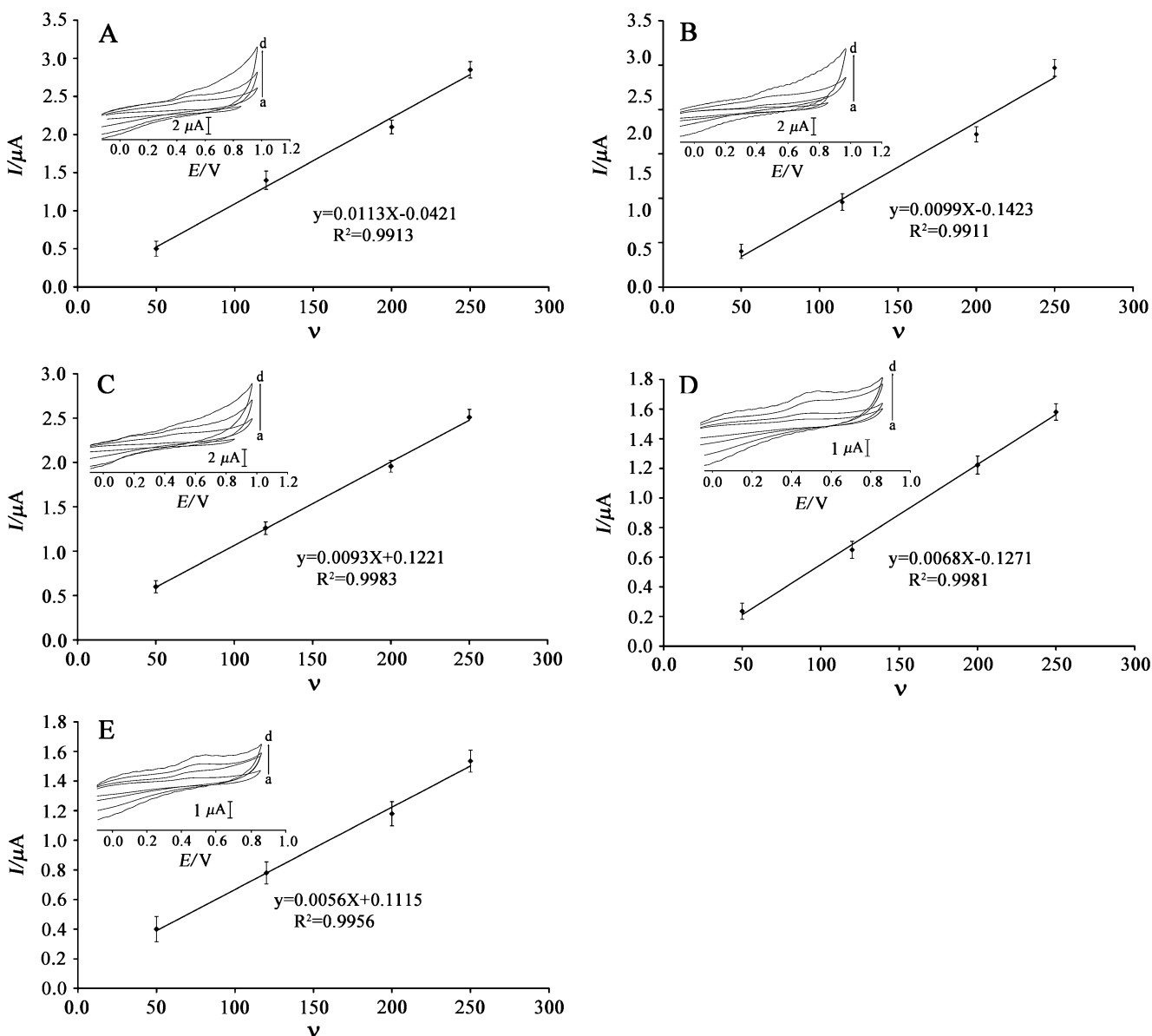
Fig. 5 Cyclic voltammograms of a DNHB-ISO, b DNHB-PHA, c DNHB-LUC, d DNHB-ALA, and e DNHB-VAL recorded at different scan rates (50, 120, 200, and 250 mV s^{-1}, respectively) at a natural pH condition

modified electrodes, the oxidation peak currents were increased (Fig. 3a–e; curves a–c). Consequently, with rising of polymer contents, the coverage of them in the CNTs matrix was increased, and this phenomenon caused to change in oxidation currents.

As a result, it can be proposed that these polymers may find applications in different industries such as coating, membrane, and composites due to the interactions with their active groups present in the structure. The influence of CNTs into the carbon paste matrix on the oxidation of these polymers was studied in aqueous solution at pH 7.0. Figure 4a–e (curves a) shows the oxidation currents of polymers at the surface of CPE. Similarly, Figure 4 (curves b) shows phenolic oxidation in the MWCNTs paste matrix.

As can be seen, the oxidation current for the polymer chain at the surface of MWCNTs is bigger than the CPE. In other words, the data obtained clearly showed that the combination of CNTs and polymers definitely improved the characteristics of the oxidation of phenolic groups into prepared composite structures. As a result, the presence of CNTs in all of these composites caused to increase in electrical currents. Also, due to the different structures of polymers, the amounts of electrical currents for each structure were dissimilar.

Cyclic voltammograms of all polymers were recorded at different scan rates, υ (from 50 to 250 mV s^{-1}), and the results are presented in Fig. 5a–e (insert). The experimental results showed anodic peaks for phenolic oxidation. These cyclic voltammograms were used to examine the variation of the peak currents vs. potential scan rates. The plots of the anodic peak currents were linearly dependant on υ with a correlation coefficient of 0.9913, 0.9911, 0.9983, 0.9981, and 0.9956 for DNHB-ISO, DNHB-PHA, DNHB-LUC, DNHB-ALA, and DNHB-VAL at all scan rates, respectively (Fig. 5).

Therefore, the peak current must be related to the surface concentration of electroactive species, Γ, using

$$I_{\mathrm{p}} = n^2F^2A\Gamma v/4RT, \tag{1}$$

where n represents the number of electrons involved in the reaction ($n=1$), A is the surface area of the electrode, I_{P} is the peak current, Γ represents the surface coverage concentration (moles per square centimeter), and ν is the scan rate. From the slope of the anodic peak currents vs. scan rate, the calculated surface concentration of DNHB-ISO, DNHB-PHA, DNHB-LUC, DNHB-ALA, and DNHB-VAL were 1.32×10^{-7}, 1.15×10^{-7}, 1×10^{-7}, 8×10^{-8}, and 6.55×10^{-7} mol cm^{-2}, respectively.

Electrochemical impedance spectroscopy study

Electrochemical impedance spectroscopy (EIS) has been used as a way to investigate the adsorption kinetics of molecules at solid electrodes, since it is a powerful technique for determining conveniently kinetic as well as mass transport parameters and charge transfer coefficient using minor electrochemical perturbation, as compared with other transient electrochemical techniques [15].

Figure 6 compares the Nyquist diagram of the electrodes recorded in PBS (pH 7.0). Under these conditions, charge transfer resistance (R_{ct}) is the only element that has a simple physical meaning describing how fast the rate of charge transfer is during the oxidation of phenolic groups in polymers. It is known that the amount of R_{ct} has reverse relationship with electrochemical activity of the species. In fact, with increasing of electrochemical oxidation current, the value of R_{ct} is decreased. The R_{ct} value of the DNHB-VAL was achieved as 135 KΩ (curve a). The R_{ct} value of DNHB-ALA, DNHB-LUC, DNHB-PHA, and DNHB-ISO were decreased to 131, 128, 124, and 120 KΩ (curves b–e), indicating that the number of phenolic group in these polymers were increased, respectively (These values obtained using a simple electric circuits Rs (Rc C)). According to the obtained results, the amount of R_{ct} for these series of polymers was decreased from DNHB-ISO to DNHB-VAL, respectively. Data had correlation with the results obtained from CV technique.

Conclusions

In this study, we described synthesis and electrochemical behavior of a new series of PAIs base on natural amino acids. Electrochemical techniques such as CV and EIS were used for the investigation of oxidation of the phenolic groups in the polymers side chains. The influence of nano-compound substance such as CNTs on the oxidation of phenolic groups was also studied. Finally, the electrochem-
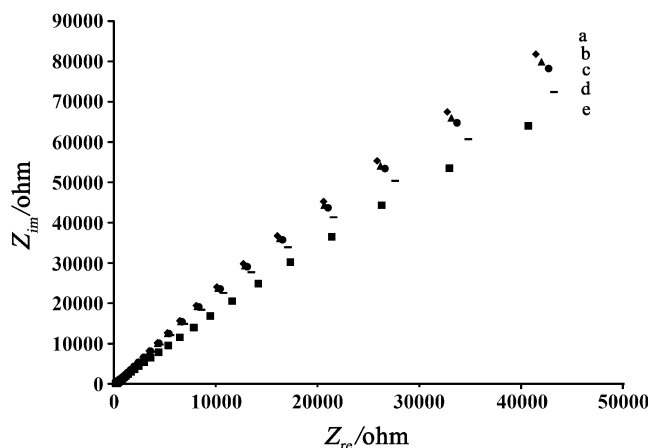
Fig. 6 Nyquist diagrams of *a* DNHB-VAL, *b* DNHB-ALA, *c* DNHB-LUC, *d* DNHB-PHA, and *e* DNHB-ISO at a surface of MWCNTs at pH 7.0. Bias is 0.45 V with $E_{\mathrm{ac}}=5$ mV with a frequency range of 10 kHz to 1 Hz

ical results have very good correlation with the viscosity data.

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