Electrochemical Synthesis of *o*-anisidine - *o*-aminophenol Copolymers

Mutlaq Shediad Al-jahdali[1] Abdel Aziz Qasem Mohammad Jbarah[2*]
Amneh Al-Ameen Mohammad Ahmed[3]

1. Chemistry Department, Faculty of Science, King Abdulaziz University, PO box 80200, Jeddah 21589, Saudi Arabia
2. Chemistry Department, Faculty of Arts and Science, Northern Borders University, PO box 1321, ArAr 91431, Rafha campus, Saudi Arabia
3. Pharmacy Department, Faculty of Pharmacy, Northern Borders University, PO box 1321, ArAr 91431, Rafha campus, Saudi Arabia

* E-mail of the corresponding author: jbr.abed@yahoo.com

Abstract

Electroactive conducting copolymers of *o*-anisidine and *o*-aminophenol molecules were pre-pared in aqueous solution by electrode potential cycling. Copolymerization was carried out at constant feed concentration of *o*-anisidine (30 mM) and different feed concentration of o-aminophenol on a gold electrode. Three different feed concentrations of o-aminophenol were used to prepare three different copolymers. The obtained copolymers were characterized with cyclic voltammetry. Both homopolymers and copolymers were obtained by cycling the potential between -0.20 V and different upper potential limits. The copolymers formed with different feed concentrations of o-aminophenol exhibited different cyclic voltammetric behaviors. In some cases using different cycling range of polymerization leads to different cyclic voltammetric behaviors of the obtained copolymers.

Keywords: cyclic voltammetry, *o*-anisidine, *o*-aminophenol, copolymers

1. Introduction

Conducting polymers attract great interest as promising candidates for various applications such as electrocatalysts, electrochromic devices, solar cells and rechargeable batteries (Sherman et al. 1994, Holze 2001). One of the most interesting conducting polymers is polyaniline and it has occupied the prime position because of its high conductivity, good redox reversibility, swift change of color with potential and good environmental stability (Manisankar et al. 2005). Several researches are made to improve the properties of polyaniline. These improvements can be reached either via the polymerization of substituted monomers and post treatment or copolymerization of aniline with other monomers (Holze 2001).

Copolymerization of two different monomers that bear different functional groups leads to modified copolymers that possessing interesting properties. The advantage of copolymerization is the possible homogeneity of the resulting material, the properties of which can be regulated by adjusting the ratio of the concentrations of the monomers in the feed. A self-doped polyaniline was obtained by copolymerization of aniline with some carboxyl- and sulfonyl-substituted derivatives (Karyakin et al.1994 and Karyakin et al. 1996). An electrochemical copolymerization of aniline with o/m-toluidines was reported (Wei et al. 1990). Aniline copolymerization with *o*-aminobenzonitrile yielded copolymers bearing cyanogroups. The electrochromic properties of these copolymers are different from those of polyaniline (Sato et al. 1994). Accelerations of the rate of electropolymerization of aniline with *p*-phenylenediamine and retardation with *m*-phenylenediamine have been reported (Yang & Wen 1994, Tang et al. 1995, Si et al. 1995).

The aniline derivative, *o*-aminophenol (OAP), has attracted much attention in the last few years. OAP can be polymerized by suitable oxidation either chemically or electrochemically (Kunimura et al. 1988, Barbero et al. 1989, Zhang et al. 1994, Pace & Kim 1988). The electropolymerization of OAP has been investigated with electrochemical techniques (Tucceri 2004), ellipsometry (Barbero et al. 1987), in situ Raman spectroscopy (Salavagione et al. 2005) and impedance measurements (Barbero et al. 1995). Poly(*o*-aminophenol), (POAP), has been assigned the structure of a ladder polymer based on repeating phenoxazine units. A preliminary study of Mu (2004) on the copolymerization of aniline (0.2 M) and OAP demonstrated that a small addition of OAP (0.01 M) to the aniline solution could result in copolymer formation on a platinum electrode. However, OAP concentration greater than 0.01 M in the feed strongly inhibited the growth of the copolymer and the author suggested that for higher OAP concentration in the feed the copolymer could not grow. Moreover, the upper potential limit for the homopolymer synthesis was rather high which could result in the overoxidation of aniline and partial degradation of polyaniline and is also not suitable for POAP synthesis as is well known in the literature (Ortega 2000, Zhang et al. 1994).

As in polyaniline, the electro-oxidation of poly(*o*-anisidine) in aqueous acidic media yields a film with desirable properties (Mottoso & Bulhoes 1992). According to our knowledge copolymerization of OAN with OAP are not investigated till now. In the present work we report the results of electrochemical copolymerization of the *o*-anisidine (OAN) and OAP.

2. Experimental

All chemicals were of analytical grade. OAN (Aldrich) was distilled under vacuum and stored under nitrogen in a refrigerator. OAP (Fluka purum) was used as received. 18 MW water (Seralpur pro 90C) was used for solution preparation. All solutions were prepared with 0.5 M sulfuric acid (Merck) supporting electrolyte.

Thin films of poly (*o*-anisidine), poly (*o*-aminophenol) and poly(*o*-anisidine co *o*-aminophenol) were synthesized electrochemically under potentiodynamic conditions at a scan rate of 50 mV/s. A three-electrode geometry was employed with gold disc as working electrode and platinum wire as counter electrode and a saturated calomel reference electrode. The surface area of the working electrode was approximately electrode0.1 cm2. All potentials quoted in this work are referred to the saturated calomel electrode. Electrochemical experiments were performed at room temperature (20 ˚C) with nitrogen purged solutions. Cyclic voltammetry was performed with a computerized SP50 potentiostat (from Bio Logic Science Instruments, France). All solutions were freshly prepared and purged with nitrogen (99.999%).

3. Results and Discussion

3.1 Electrochemical homopolymerization of OAP

Figure 1a shows representative cyclic voltammograms (CVs) recorded during the homopolymerization of OAP (1 mM) by cycling the potential between -0.2 and 1.0 V (100 cycles) at a scan rate of 50 mV/s. In the first positive sweep of Figure 1a two anodic peaks are well defined. The first anodic peak observed at 0.65 V is caused by the oxidation of -OH group of OAP and the other peak at 0.81 V is due to the oxidation of -NH_2 as has been reported earlier (Mu 2004). On the cathodic sweep, these peaks are not show any corresponding reduction peaks. On further potential cycling the oxidation current of these peaks decreased rapidly. In the second cycle of Figure 1a, a redox pair was observed at 0.33/0.29 V. During continuous cycling it was observed that this redox pair diminished slowly while anodic and cathodic currents increase in the potential region between -0.20 and 0.30 V. The increases of anodic and cathodic currents in this region can be attributed to the formation and growing of the POAP film at the gold surface. Thus, the disappearing of the a redox pair at 0.33/0.29 V is a result of the polymer which results in the diminishing of the redox system at 0.35/0.33 V. it should be mention that the redox pair at 0.33/0.29 V is concerning the formation of a cyclic dimer of OAP, the 3-aminophenoxazone , which is formed by a relatively slow cyclization reaction of the oxidized C–N dimer of the OAP radical cation (Zhang et al. 1994). The formation of the 3-aminophenoxazone plays the role in the formation of POAP. The gold electrode that coated with POAP film was then immersed into monomer free electrolyte solution and its cyclic voltammogram (CV) was recorded in the potential range between -0.30 and 0.45 V as shown in Figure 1b. The CV of POAP shows one anodic peak at 0.13 V and its corresponding cathodic one at -0.02 V.

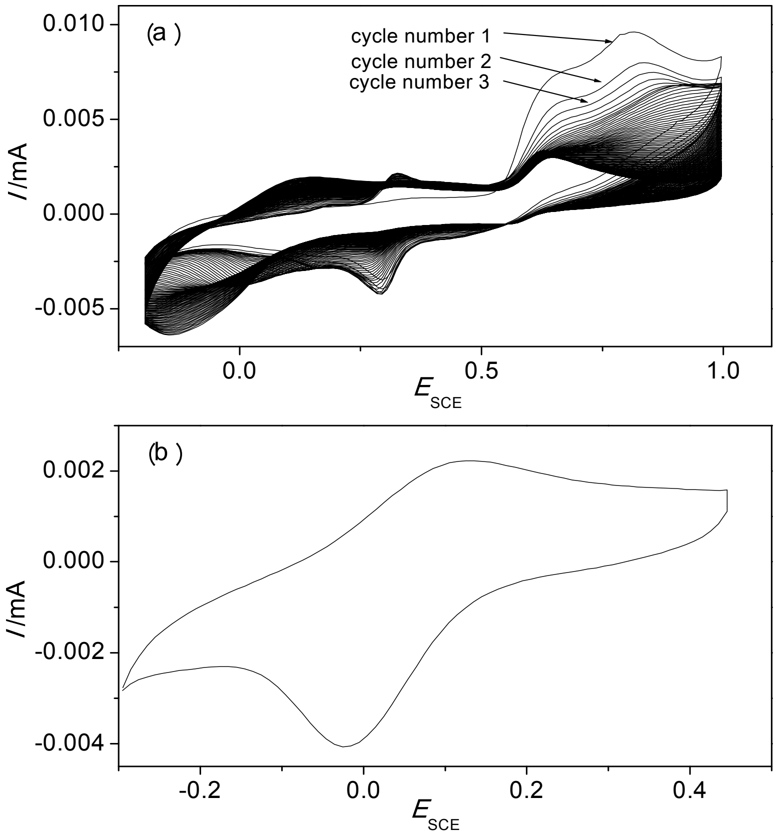

Figure 1. The cyclic voltammograms for (a) electrolysis of the solution containing 1 mM OAP in 0.5 M H_2SO_4 by cycling the potential between -0.20 and 0.10 V, (b) POAP in monomer free electrolyte solution at a scan rate of 50 mV/s

Shah & Holze (2006) reported that the POAP could not be deposited at gold surface from a solution of 1 mM OAP in 0.5 M H_2SO_4 by using the upper value of cycling potential higher than 0.84 V. They concluded that the POAP degrades at high anodic potentials. According to our results this is not correct. Actually, the electrochemical behavior of the POAP in monomer free electrolyte varies according to the used deposition condition. To show that, we record a second CVs (100 cycles) for the homopolymerization of OAP (1 mM) by cycling the potential between -0.2 and 0.84 V at a scan rate of 50 mV/s which are displayed in Figure 2a. It should be noted that the only difference between the first experiment (Figure 1a) and the second one (Figure 2a) is the upper value of the cycling potential. It is clear that the CVs of Figure 2a different from that of Figure 1a. This difference appears in the potential range from -0.20 to 0.30 V of the CVs. The obtained POAP modified electrode from the second experiment was immersed into monomer free electrolyte solution and its CV was recorded in the potential range between -0.20 and 0.45 V as depicted in Figure 2b. The CV (Figure 2b) of POAP shows one anodic peak at 0.14 V and its corresponding cathodic one at 0.10 V. By comparing the CV of Figure 1b with that of Figure 2b, one can note that the potential gab difference between the oxidation potential and its corresponding reduction one of the POAP increases in the case of Figure 1b. Also the value of anodic and cathodic peaks currents of the CV of Figure 1b are higher than those of the CV of Figure 2b.

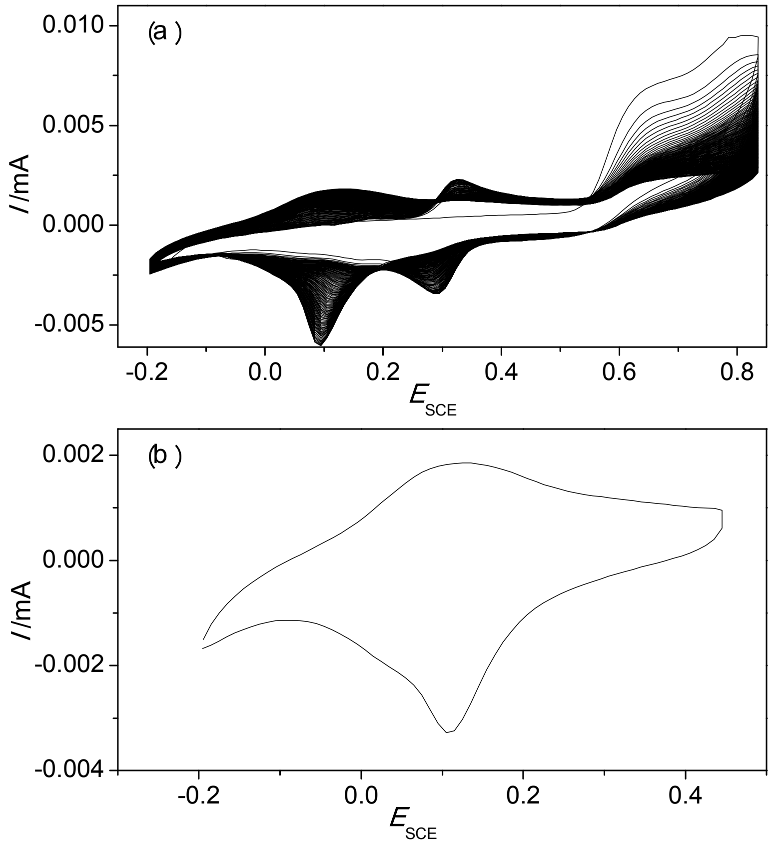

Figure 2. The cyclic voltammograms for (a) electrolysis of the solution containing 1 mM OAP in 0.5 M H_2SO_4 by cycling the potential between -0.20 and 0.84 V, (b) POAP in monomer free electrolyte solution at a scan rate of 50 mV/s

3.2 Electrochemical homopolymerization of OAN

Figure 3 a shows representative CVs (50 cycles) recorded during the growth of a POAN film deposited from an aqueous solution of 30 mM OAN in 0.5 M H_2SO_4 by cycling the potential from -0.20 to 1.0 V. On the first cycle of Figure 3a an anodic currents starts at 0.72 V and reach its maximum value at 0.91 V. This anodic currents is due to electro oxidation of OAN. During continuous cycling of the CVs of Figure 3a it was observed that this anodic currents disappeared while anodic and cathodic currents increase in the potential region between -0.20 and 0.65 V. The increases of anodic and cathodic currents in this region can be attributed to the formation and growing of the POAN film at the gold surface. Then the gold electrode that coated with POAN films were then immersed into monomer free electrolyte solution and its CV of were recorded in the potential range between -0.20 and 0.7 V as shown in Figure 3b. The CV of Figure 3b shows three redox pair at 0.21/0.15, 0.42/0.36 and 0.57/0.49 V. The third anodic peak of these pairs at 0.57 V appears like shoulder. The first redox pair at 0.21/0.15 arises due to the formation of radical cations via oxidation of amine nitrogen, while the pair at 0.42/0.36 V can be attributed to the formation of benzidine derivatives (Wei et al. 1989, Patil et al. 1997). The benzoquinone produced as a result of hydrolysis represents the third redox couple at 0.57/0.49 V (Patil et al. 1997, Stilwell & Park 1988, Cui et al. 1993). It should be mentioned that a forth cathodic peak appears as a shoulder at 0.08 V. This cathodic peak might be related the first redox pairs.

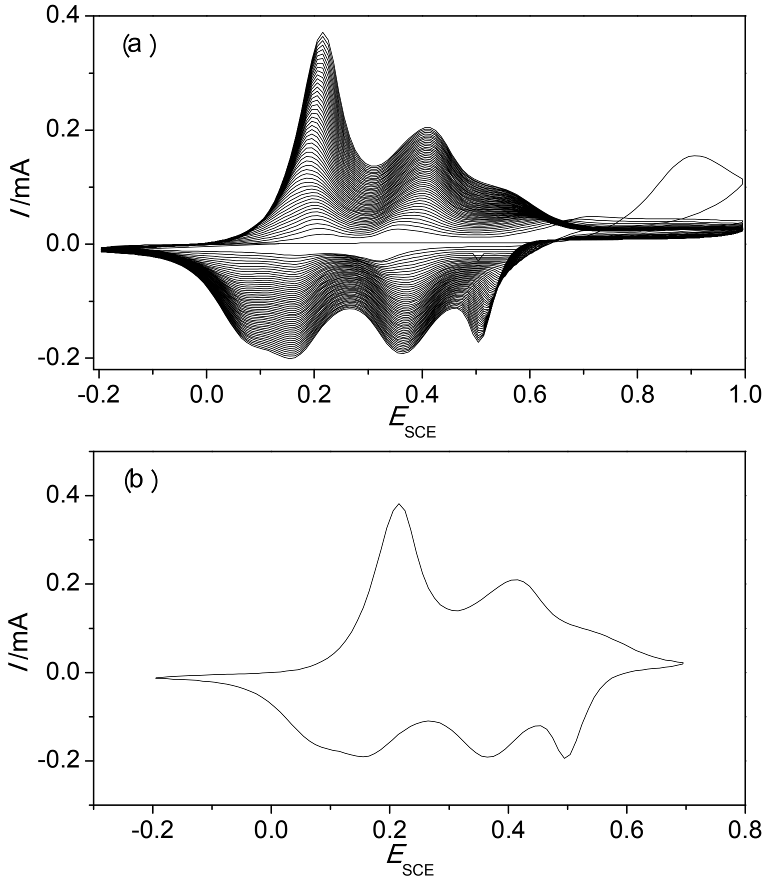

Figure 3. The cyclic voltammograms for electrolysis of solution containing 30 mM OAN in 0.5 M H_2SO_4 by cycling the potential between (a) -0.20 and 1.0 V and (b) POAN in monomer free electrolyte solution at a scan rate of 50 mV/s

OAN was also electrochemically polymerized by cycling the potential between -0.20 V and different upper potential limits. Figure 4a shows representative CVs (50 cycles) recorded during the growth of a POAN film deposited from an aqueous solution of 30 mM OAN in 0.5 M H_2SO_4 by cycling the potential from -0.20 to 0.84 V. The CV of the obtained POAN film was displayed in Figure 4b. The electrochemical features of the CVs of Figure 4 are very similar to that of Figure 3. The only difference between the CVs of these figures is that the cathodic peak of Figure 3b at 0.08 V becomes well defined with an increasing in its current value in the case of Figure 4b.

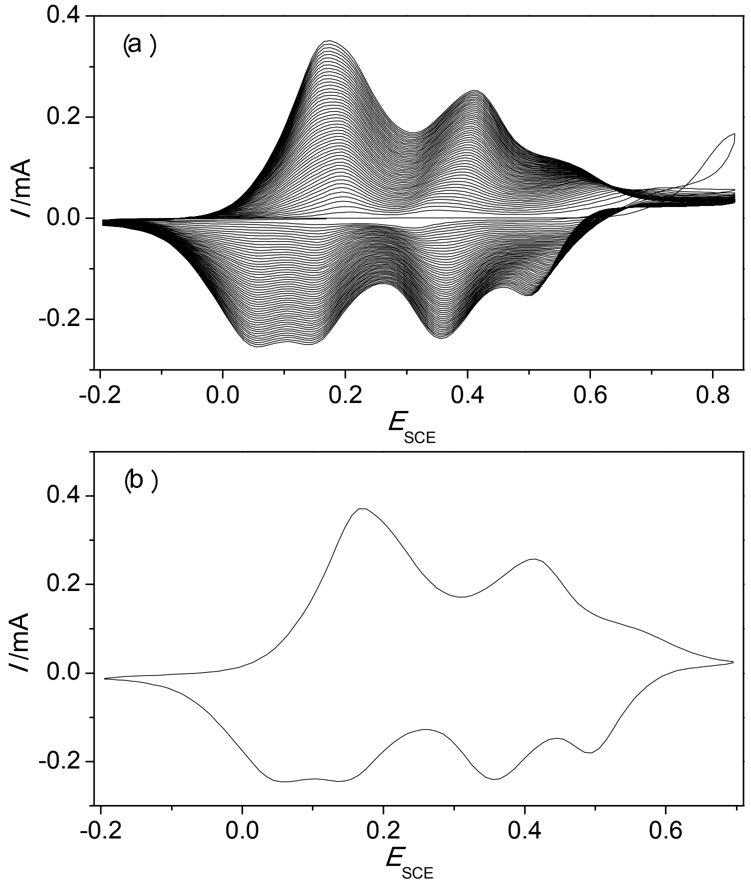

Figure 4. The cyclic voltammograms for electrolysis of solution containing 30 mM OAN in 0.5 M H_2SO_4 by cycling the potential between (a) -0.20 and 0.84 V and (b) POAN in monomer free electrolyte solution at a scan rate of 50 mV/s

3.3 Electrochemical copolymerization of OAN with OAP

In order to determine the suitable feed ratio concentrations of the monomers solutions for the electrochemical coplymerization we chose first a high concentrations from each monomer. We used for this experiment a concentration of 30 mM of both OAN and OAP in the polymerization solutions. The CVs (50 cycles) recorded during the copolymerization of OANI with OAP for this feed ratio concentrations in 0.5 M H_2SO_4 and by cycling the potential from -0.20 to 1.00 V are shown in Figure 5. The first cycle of this CVs shows huge anodic peak at 0.74 V. During continuous cycling this peak disappeared without observation of any new peaks. This result suggesting that the copolymer could not be formed by using this feed ratio of the monomer concentrations. A similar result was reported in a previous study (Mu 2004) on the copolymerization of aniline and OAP. The author suggested that the using of OAP concentration greater than 10 mM in the feed strongly inhibited the growth of the copolymer . Also he demonstrated that a small addition of OAP (10 mM) to the aniline solution could result in copolymer formation on a platinum electrode. Thus in our study, copolymerization was carried out with constant concentration of OAN and a different feed concentrations of OAP which are less than 10 mM.

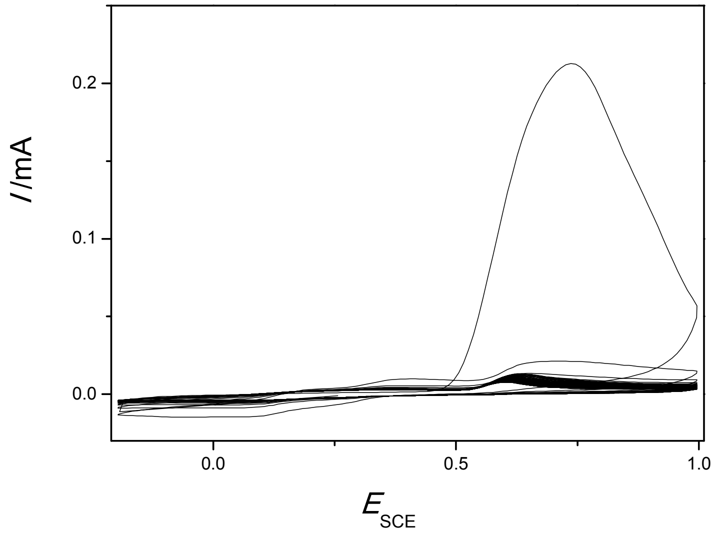

Figure 5. The cyclic voltammograms (50 cycle) for the solution of 30 mM OAN and 30 mM OAP in 0.5 M H_2SO_4 by cycling the potential between -0.20 and 1.00 V at 50 mV/s

The copolymerization condition that used in our study are listed in Table 1 together with the copolymers labels. Like the homopolymerization of OAP and OAN, electrolysis of mixed solutions containing both OAP and OAN was carried out by cycling the potential between -0.20 V and different upper potential limits.

Table 1. Monomers concentration, cycling range and number of cycles that used during electrochemical preparation of each case of the copolymers.

Copolymer lable	**Cycling range (mV)**	**Concentration of OAN (mM)**	**Concentration of OAM (mM)**	**Number of cycles**
A1	-200 - 840	30	1	50
B1	-200 - 840	30	3	50
C1	-200 - 840	30	5	50
A2	-200 - 1000	30	1	50
B2	-200 - 1000	30	3	50
C2	-200 - 1000	30	5	50

CVs recorded during the potentiodynamic copolymerization of OAN with OAP for system A1 are shown in Figure 6a. In the first cycle of Figure 6a there are one anodic peak at 0.69 V and increasing in current between 0.69 V and 0.84 V. The anodic peak at 0.69 V can be assigned to the oxidation of the hydroxyl group of OAP. The observed current between 0.69 V and 0.84 V is caused by the oxidation of amino groups from both monomers. Actually the the oxidation of amino groups from both monomers occurs at electrode potential more than 0.84 V as indicated from Figure 6d. In the cathodic scan of first cycle of Figure 6a there are two cathodic peaks. From cycle number 10 to cycle number 50 the CVs of Figure 6a show well defined three anodic peaks and four cathodic ones. These anodic and cathodic peaks are clearly indicated the deposition of the copolymer. The behaviour of CVs of system A2 (Figure 6d) are similar to that of system A1 with limited differences. The first difference is the observation of the anodic peak in the first cycle of system A2 at 0.88 V. This anodic peak is not observed for system A1 because of using upper potential lower than 0.88 V. The second difference is that the overall anodic and cathidc current in the CVs of system A2 are higher than that of system A1. This difference also can be attributed to the difference in the upper potential limits that used in each case.

The first cycle behaviour of the CVs of the systems B1 (Figure 6b) and B2 (Figure 6e) are very similar to that of systems A1 (Figure 6a) and A2 (Figure 6d), respectively. The behaviour of the subsequent cycles (from cycle number 2 to cycle number 50) of the CVs of the systems B1 and B2 are different from that of systems A1 and A2. In the CVs of systems B1 and B2 the anodic peaks position shifts to higher potential upon cycling. This behaviour is not observed in the CVs of systems A1 and A2. The overall anodic and cathodic peak currents of the systems B1 and B2 are lower than that of systems A1 and A2. These differences suggests the copolymers formed in the case of systems A1 and A2 are different from that for systems B1and B2.

The CVs of the systems C1 and C2 are displayed in Figure 6c and 6f. The first cycle of these systems are similar to that of other systems that discussed previously. The second cycle of the CVs of system C1 shows four anodic peaks while it shows only three anodic peaks in the case of system C2. This results suggest that the oligomers forms in the case of system C1 are different from that of system C2. This conclusion is based on that the second cycle is concerns the formation of oligomers. The subsequent cycles of systems C1 and C2 show a very different behaviour than that of systems A1, A2, B1 and B2 (Figure 6). This is very clear from the cycle number 50. This cycle shows three anodic peaks and four cathodic ones in the case of systems A1 and A2. In the case of systems B1 and B2 the cycle number 50 show three anodic peaks and three cathodic ones. In the case of systems C1 and C2 the cycle number 50 show only one broad anodic peaks and two cathodic ones. It should be mentioned that the over all anodic and cathodic currents of the peaks in the cycle number 50 of the CVs of system C1 and C2 are very low as compared to those of systems A1, A2, B1 and B2 (Figure 6). Based on the results presented, it can be concluded that the overall rate of electrochemical copolymerization decreases with increase in the molar fraction of OAM in combination with OAN. The differences in electrochmical behaviour of these systems will be discussed later in this text.

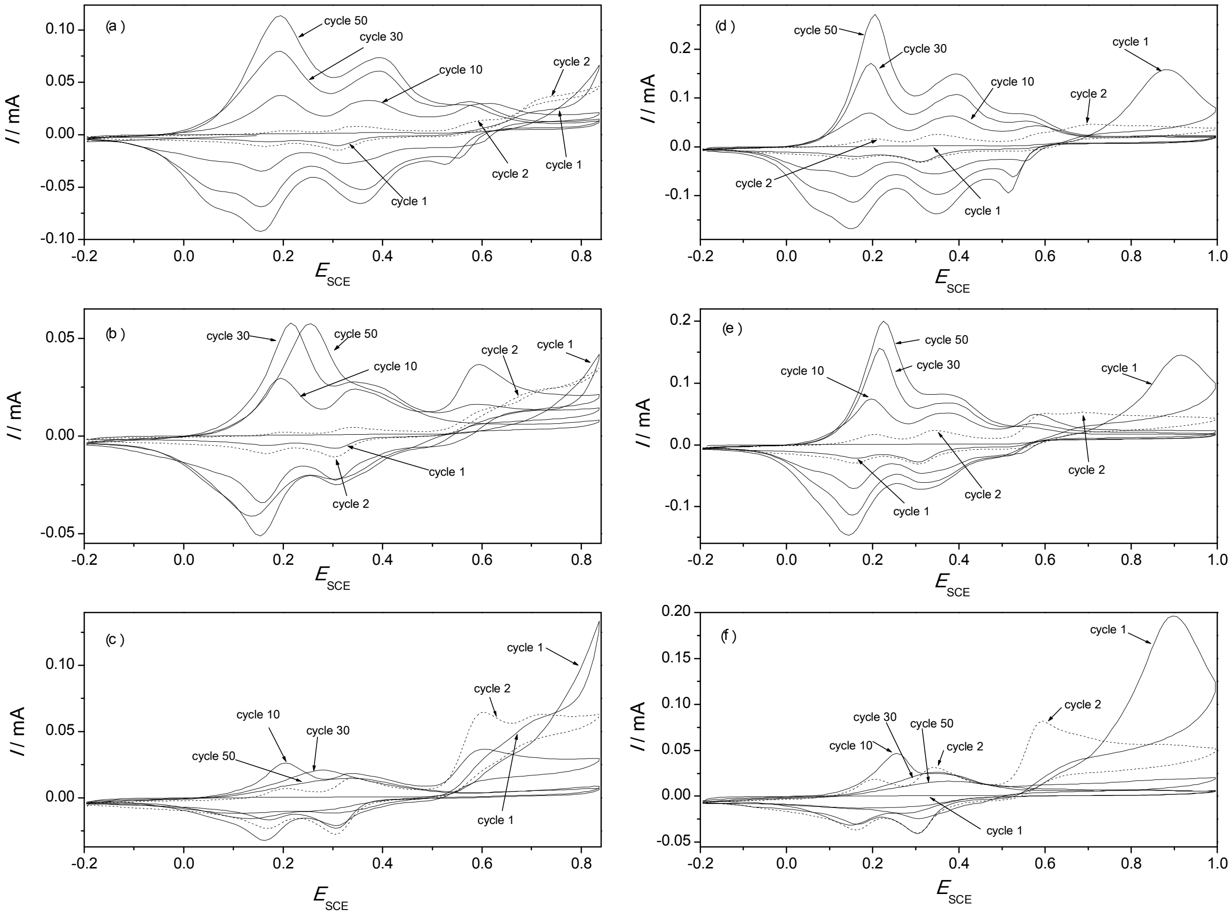

Figure 6. The cyclic voltammograms for the growth of (a) copolymer A1 (50 cycles), (b) copolymer B1 (50 cycles), (c) copolymer C1 (50 cycles), (d) copolymer A2 (50 cycles), (e) copolymer B2 (50 cycles) and (f) copolymer C2 (50 cycles) at scan rate of 50 mV/s

Figure 7 shows the CVs of copolymer A1, A2, B1, B2, C1 and C2. The CVs were recorded after several potential cycles in the supporting electrolyte solution between 0.20 and 0.65 V until a stable CV was established. The peak potential positions of the observed anodic and cathodic peak of the CVs of copolymers (Figure 7) are summarized in Table 2 along with that of the CVs of homopolymers (Figures 1b, 2b, 3b and 4b).

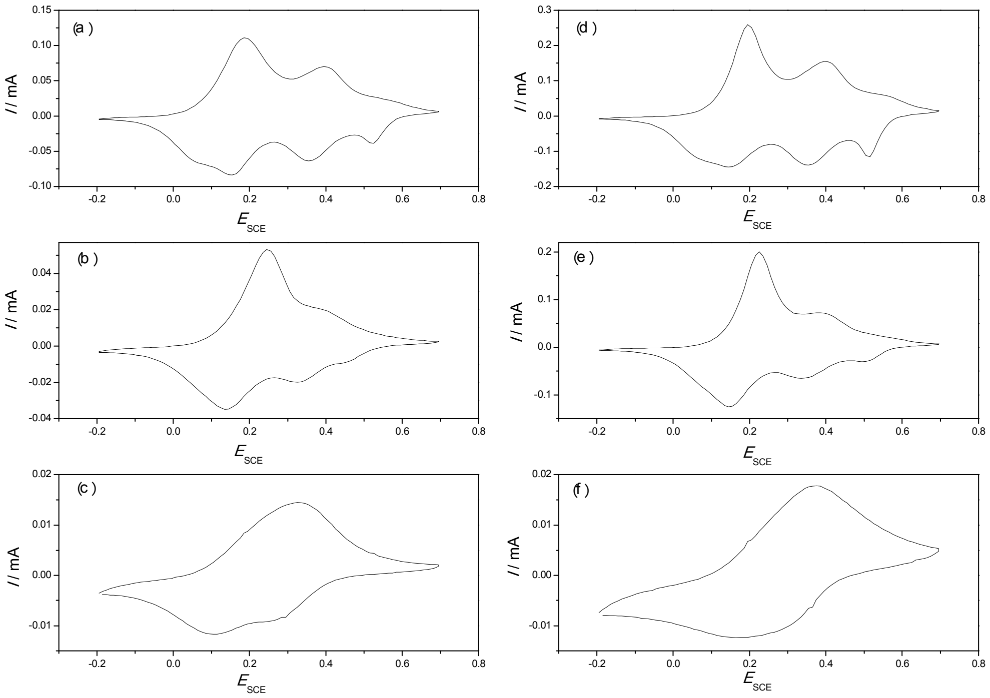

Figure 7. The cyclic voltammograms of (a) copolymer A1, (b) copolymer B1, (c) copolymer C1, (d) copolymer A2, (e) copolymer B2 and (f) copolymer C2 in monomer free electrolyte solution between -0.20 and 0.65 V at 50 mV/s.

The CVs of copolymers A1 (Figure 7a) and A2 (Figure 7d) show three anodic peaks and four cathodic ones. This behaviour are very similar to that of POAN. Also the values of the anodic and cathodic peaks of the copolymers A1 and A2 are very close to that of POAN (see Table 2). A small difference only between the peak potential positions in the CVs of the copolymers and that of POAN are observed. These results are expected and related to the fact that the polymerization solution that used to obtain these copolymers contains very small molar fraction of OAM as compared to that OAN. Upon increasing the molar fraction of OAM in the polymerization solution as in the case of copolymers B1 and B2, the CVs behaviour becomes very different from that of A1, A2 and POAN. In the CVs of the copolymers B1 and B2 (Figure 7b and 7e, Table 2) the first anodic peak shifts to higher oxidation potential as compared to those of A1, A2 and POAN. Also the third anodic peak and the first cathodic peak disappeared in the CVs of B1 and B2 copolymers. More over the over all peak currents of the CVs of B1 and B2 copolymers are lower than those of A1, A2 and POAN. Further increasing the molar fraction of OAM in the polymerization solution as in the case of copolymers C1 and C2, the CVs behaviour becomes totally different from that of A1, A2, B1, B2 and POAN. The CV of copolymer C1 (Figure 7c) shows only one broad anodic peak at 0.34 V and two corresponding cathodic peaks at 0.1 V and 0.27 V (Table 2). These features are not observed in the CVs of A1, A2, B1, B2 , POAN and POAP. In the case of the of copolymer C2 the CV shows only one broad anodic peak at 0.37 V and one broad cathodic one at 0.18 V. These electrochemical behaviours could not be observed in all CVs of the other copolymers and homopolymers. It should be mentioned that the peaks current of the CVs of copolymers C1 and C2 are very small as compared with those of A1, A2, B1, B2 and POAN.

Table 2. Peak potential postions of the observed anodic and cathodic peak of the CVs of the copolymers A1, A2, B1, B2, C1 and C2 and hmomopolymers POAN and POAP.

Polymer	E_{anodic1} (V)	E_{anodic2} (V)	E_{anodic3} (V)	$E_{\text{cathodic1}}$ (V)	$E_{\text{cathodic2}}$ (V)	$E_{\text{cathodic3}}$ (V)	$E_{\text{cathodic4}}$ (V)
POAP[a] (Figure 1b)	0.13	-	-	-0.02	-	-	-
POAP[b] (Figure 2b)	0.14	-	-	0.1	-	-	-
POAN[c] (Figure 3b)	0.21	0.42	0.57	0.08	0.15	0.35	0.49
POAN[d] (Figure 4b)	0.17	0.41	0.55[s]	0.05	0.15	0.35	0.49
A1 (Figure 7a)	0.19	0.4	0.55[s]	0.06[s]	0.14	0.35	0.51
A2 (Figure 7d)	0.20	0.40	0.55[s]	0.06[s]	0.14	0.35	0.51
B1 (Figure7b)	0.25	0.40	-	-	0.13	0.33	0.45
B2 (Figure 7e)	0.23	0.4	-	-	0.14	0.34	0.49
C1 (Figure 7c)	-	0.34[vb]	-	-	0.10	0.27	-
C2 (Figure 7f)	-	0.37[vb]	-	-	0.18[vb]	-	-

[a, c]: The polymer obtaned by cycling the potential between -0.2 and 1.0 V; [b, d]: The polymer obtaned by cycling the potential between -0.2 and 0.84 V; [S]: shoulder shape, [vb]: very broad

5. Conclusion

Copolymers of OAN and OAP have been prepared potentiodynamically in a solution containing 0.5 M H_2SO_4, 30 mM OAN and different concentrations of OAP. The copolymers formed with different feed concentrations of OAP exhibited different cyclic voltammetric behaviours. In some cases using different cycling range of polymerization leads to different cyclic voltammetric behaviours as in the case of POAP, C1 and C2. Increasing the molar concentration of the OAP monomers in the feed greater than 10 mM strongly inhibited the growth of the co-polymer.

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