

ELECTROCHEMICAL BEHAVIOUR OF DIHYDROXYBENZENE ISOMERS AT GLASSY CARBON ELECTRODE IN DIFFERENT ELECTROLYTIC MEDIA

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ABSTRACT

Electrochemical behaviour of catechol, hydroquinone and resorcinol was investigated in KCl, acetate buffer and phosphate buffer at glassy carbon electrode using cyclic voltammetry. The results imply that the glassy carbon electrode exhibited a synergistic and excellent electrocatalytic effect on the redox behaviors of the benzenediols. Catechol and hydroquinone show one pair of redox peaks in acetate and phosphate buffer but two pairs of redox peaks in KCl. In all cases the redox process is quasireversible. Resorcinol behaves almost irreversibly in all three electrolytic media. Good linear relationships were obtained between the peak currents and the concentrations of benzenediols. The electrochemical process in all the isomers was controlled by diffusion process.

Key words: Catechol, Hydroquinone, Resorcinol, Glassy carbon electrode

INTRODUCTION

Catechol (CT), hydroquinone (HQ) and resorcinol (RC) are three dihydroxybenzene isomers, which are widely used in medicines, pesticides, cosmetics, tanning, flavoring agents, antioxidant, dye and photography chemicals (Wang *et al.* 2003). Due to their high toxicity and low degradability in the ecological environment, they are considered as environmental pollutants (Xie *et al.* 2006). During the manufacturing and application process of these compounds, some of them are inadvertently released into the environment to contaminate rivers and ground waters. Thus, it is easy for them to enter into the environments, and exists in industrial effluents and sanitary wastewater. Moreover, these isomers are often coexisting in environmental samples (Cui *et al.* 1999, Ding *et al.* 2005). It is necessary to develop simple and rapid analytical method for the determination of different types of dihydroxybenzene isomers. In developing methods it is urgent to know the behavior of each isomer.

Nowadays, electrochemical methods have attracted more and more attentions due to the advantages of fast response, cheap instrument, low cost, simple operation, time saving, high sensitivity, and excellent selectivity. Among all voltammetric techniques cyclic voltammetry however, has attracted attention to scientists in electrochemical

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research, because, it can provide useful information about redox reaction in an easily interpretable form. In earlier communications the author reported electrochemical behavior of a few metal-ligand systems (Laizu *et al.* 2010, Akhter 2008, Islam 2009, Rahman 2011 and Hasan 2012). In this paper, the redox behaviour of CT, HQ and RC at glassy carbon electrode using cyclic voltametry in different electrolytic media are reported.

MATERIALS AND METHODS

The redox properties of dihydroxybenzene isomers, mechanism of reactions involving electro active species have been examined using cyclic voltametry (CV). Chemicals used in the experiments were: (i) catechol (BDH), (ii) hydroquinone (BDH), (iii) resorcinol (BDH), (iv) NaOH pellets (A.R. grade), (v) potassium chloride (MERCK, Germany), (vi) potassium phosphate (monobasic) (MERCK, Germany), (vii) sodium acetate (MERCK, Germany) and (viii) acetic acid (Sigma-Aldrich Laborchemikaline, GmbH). About 99.997% dry nitrogen (BOC, Bangladesh) was used for purging purpose. For cleaning and preparation of all solutions, deionized water was used.

This study was carried out using an Epsilon electroanalyser developed by Bioanalytical System, Inc. USA in a Pyrex glass micro cell with Teflon cap. Glassy carbon electrode was used as working electrode. Ag/AgCl electrode was used as reference and Pt wire was used as counter electrode. An AGE (Velp Scientifica) magnetic stirrer with a teflon coated magnetic bar, a pH meter (ORION 2 STAR made by Thermo Electron Corporation) was employed for stirring and maintaining the pH value of the solutions, respectively.

RESULTS AND DISCUSSION

The redox behavior of all the isomers of dihydroxybenzene, catechol, hydroquinone and resorcinol have been studied using cyclic voltametry. The study was carried out at glassy carbon electrode (GCE) in 0.1 M KCl, acetate buffer solution of pH 4.5 and phosphate buffer solution of pH 7.0 within the same potential window (from -600 mV to +1200 mV) at various scan rates and various concentrations.

The cyclic voltammogram of 1 mM catechol in 0.1 M KCl was taken within the potential window -600 mV to +1200 mV. Fig. 1 depicts voltammograms of catechol and KCl solutions. There are two anodic peaks at (+134 mV and + 618 mV) and two cathodic peaks at (+157.6 mV and -17.6 mV) respectively. The pair of peaks at + 134 mV and -17.6 mV is very weak.

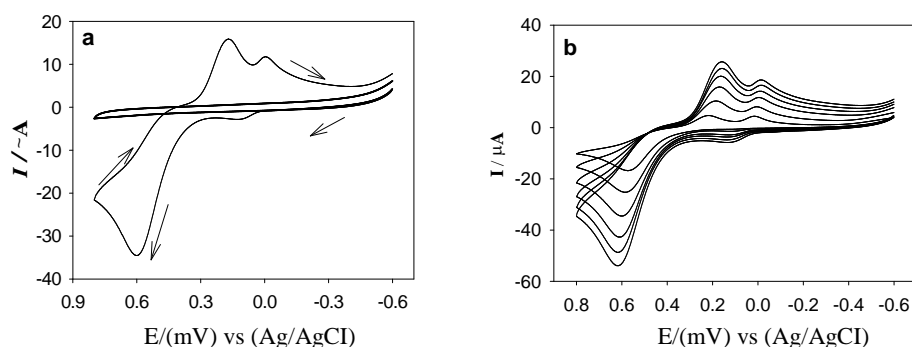


Fig. 1a. Cyclic voltammograms of 1 mM catechol solution in 0.1 M KCl and the supporting electrolyte 0.1 M KCl at 100 mVs⁻¹ scan. b. CV of catechol solution at 20, 50, 100, 150, 200, 250 mVs⁻¹ scan rate.

The redox reactions involve, can be explained in terms of electron transfer mechanism (Mascus *et al.* 1966) corresponding to the overall two one-electron transfer reaction for both anodic and cathodic process. The electrode reactions may be shown as,

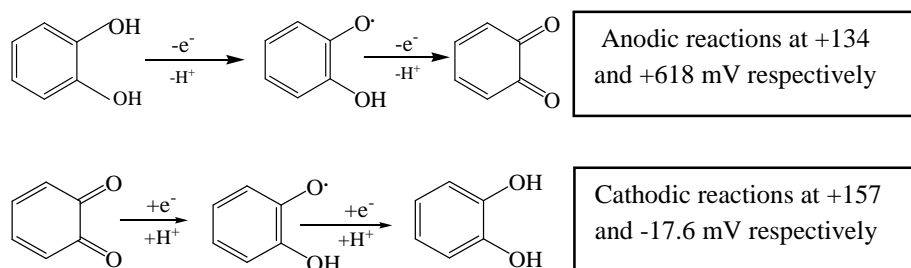


Table 1. Current-potential data, peak potential separation, peak current ratio of the voltammograms of 1 mM catechol in 0.1 M KCl at different scan rates.

V _s ⁻¹	1/2	E _{pa1} V(+)	E _{pc1} V(-)	i _{pa1} μA(-)	i _{pc1} μA(+)	E ₁ = E _{pc1} ~E _{pa1} (V)	i _{pa1} /i _{pc1}
0.020	0.141	0.155	0.009	2.12	5.65	0.164	0.375
0.050	0.223	0.157	0.012	4.96	8.52	0.169	0.582
0.100	0.316	0.162	0.015	6.97	10.72	0.177	0.650
0.150	0.387	0.165	0.032	8.45	12.01	0.197	0.704
0.200	0.447	0.170	0.035	11.33	14.14	0.205	0.801
0.250	0.500	0.174	0.039	12.88	16.03	0.213	0.803

V _s ⁻¹	1/2	E _{pa2} V(+)	E _{pc2} V(+)	i _{pa2} μA(-)	i _{pc2} μA(+)	E ₂ = E _{pc2} ~E _{pa2} (V)	i _{pa2} /i _{pc2}
0.020	0.141	0.570	0.210	16.10	3.89	0.360	4.138
0.050	0.223	0.580	0.195	28.60	10.83	0.385	2.641
0.100	0.316	0.598	0.178	37.89	18.45	0.420	2.054
0.150	0.387	0.608	0.165	42.51	20.93	0.443	2.031
0.200	0.447	0.615	0.160	49.23	24.19	0.455	2.035
0.250	0.500	0.621	0.152	57.84	30.53	0.469	1.895

= Scan rate; 1/2 = SQRT of scan rate; E_{pa} = Anodic peak potential; E_{pc} = Cathodic peak potential; i_{pa} = Anodic peak current; i_{pc} = Cathodic peak current; E = Peak potential separation.

A series of cyclic voltammograms of catechol in KCl at different scan rates are shown in Fig. 1b. The potential and current of all the CV for both pair of peaks are listed in Table 1. From the figure it is seen that for the cathodic peaks, the peak potentials gradually decreased and for the anodic peaks, the peak potentials gradually increased as the scan rate increased. But the increasing and decreasing rate of potential is very small. This behavior can be described by slower charge propagation, probably due to difference in solvation and or permeability (Wopshall and Shain 1967).

It is observed that the peak current increases with scan rate. In a slow voltage scan the diffusion layer grows much further from the electrode in comparison to a fast scan. Consequently, the flux to the electrode surface is considerably smaller at slow scan rates than it is at faster rates. As the current is proportional to the flux towards the electrode the magnitude of the current becomes lower at slow scan rates and higher at high scan rates. The general conclusion is that the redox system is diffusion controlled (Mascus *et al.* 1966).

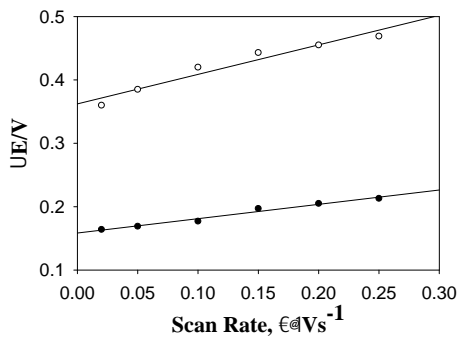


Fig. 2. Variation of peak potential separation against scan rate.

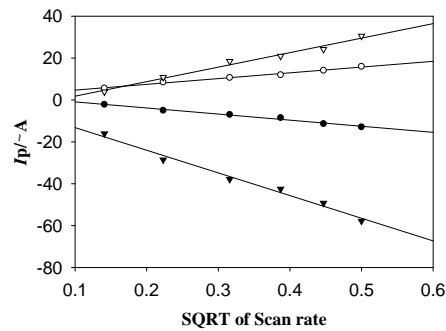


Fig. 3. Variation of peak current against SQRT of scan rate.

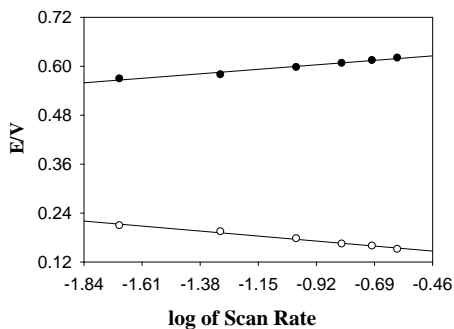


Fig. 4. Variation of peak potential against log of scan rate.

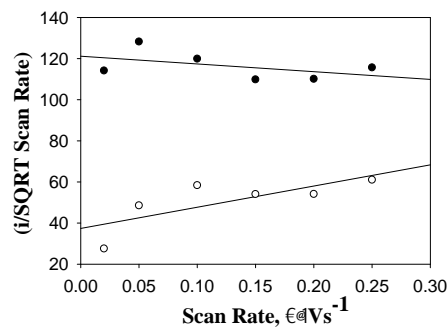


Fig. 5. Variation of peak current function against scan rate.

From Fig. 2 it is clear that with increasing scan rate, the peak potential separation increases because the cathodic peak shifts towards negative potential and that of anodic towards positive potential. Here the cause is the effect of IR drop (Zhang and Anson 1992). Fig. 3 illustrates that with increasing SQRT of scan rate ($v^{1/2}$), the peak currents increases, concluding that the process is adsorptive controlled (Bard and Faulkner 1980). The peak current ratio is found to be far from unity, which implies the system to be quasireversible. The slope of the potential vs log scan rate plot (Fig. 4) is not zero so, the process is not reversible. Peak current function vs scan rate plot (Fig. 5) shows the linear increment with scan rate, which implies that the electrochemical process under the investigation do not followed by any chemical process, i.e. it does not follow EC mechanism (Nicholson and Shain 1964, Golabi and Nimatollahi 2000).

The cyclic voltammograms of catechol of various concentrations (1, 2 and 3 mM) in 0.1 M KCl at glassy carbon electrode at 50 mV/sec scan rate and the variation of peak current with concentration are shown in Fig 6. With the increase in concentration there is a gradual linear increase in peak current, which may be due to the presence of a large amount of electroactive species at higher concentration. The peak current increases with concentration also give the idea that the system may be diffusion controlled.

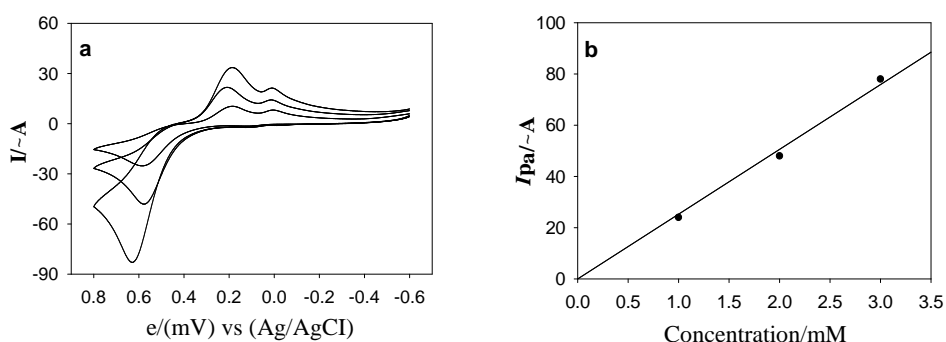
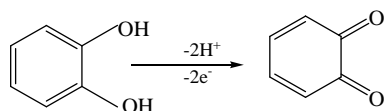


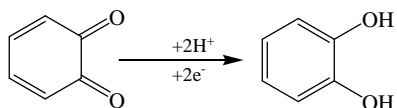
Fig. 6a. Cyclic voltammograms of 1, 2, 3 mM catechol in 0.1 M KCl at GCE at 50 mVs⁻¹ scan rate.
b. Variation of anodic peak current with concentration of catechol.

The cyclic voltammograms of 1 mM catechol in acetate buffer and acetate buffer were taken within the same potential window and are shown in Fig. 7. Here the redox behavior of catechol has been modified. There is only one pair of anodic (at +614 mV) and cathodic peaks (at +80.0 mV). So, the redox process of catechol in acetate buffer involve one two-electron transfer reaction for both cathodic and anodic processes.

The electrode reactions may be shown as,



Anodic reaction at +614 mV.



Cathodic reaction at +80 mV.

The CVs at different scan rates are shown in Fig. 7b. The current-potential data, peak potential separation, peak current ratio of the voltammograms at different scan rates have been studied carefully. The change of peak separation with scan rate plot clearly indicate that with increasing scan rate, the peak potential separation increases because of the effect of IR drop. The increase of peak currents with increasing SQRT of scan rate ($v^{1/2}$), indicates that the process is adsorptive controlled. The peak current ratio was found to be very near to unity, which implies the system to be reversible.

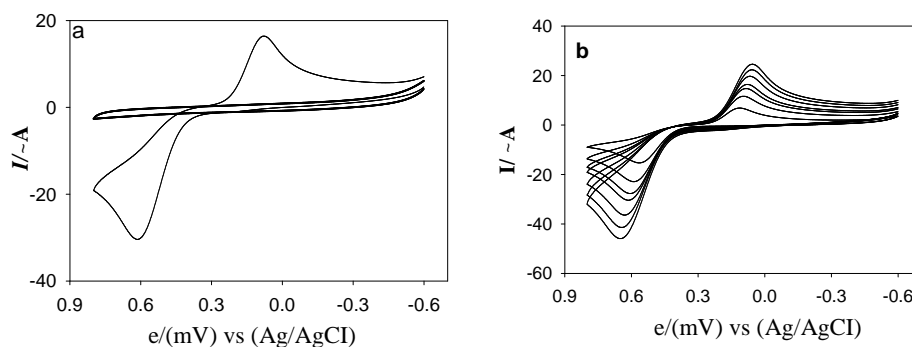


Fig. 7a. Cyclic voltammograms of 1 mM catechol solution in acetate buffer and the supporting electrolyte acetate buffer at 100 mVs^{-1} scan. b. CV of catechol solution at 20, 50, 100, 150, 200, 250 mVs^{-1} scan rate.

With the increase in concentration there is a gradual linear increase in peak current, which may be due to the presence of a large amount of electroactive species at higher concentration. The peak current increases with concentration also give the idea that the system may be diffusion controlled.

The redox behavior of catechol in the phosphate buffer is almost similar to that in the acetate buffer. There is only one pair of anodic and cathodic peaks but the peak positions are very much different. One pair of peaks implies that the redox process of catechol in phosphate buffer involve one two-electron transfer reactions for both cathodic and anodic processes and follows same mechanism. The probable reactions are as like that in the

acetate buffer. The scan rate effect and concentration effect on the redox behavior of catechol in phosphate buffer follows similar trend and pattern.

The cyclic voltammogram of 1 mM hydroquinone in 0.1 M KCl was taken within the potential window -600 to $+1200$ mV. Fig. 8a shows voltammograms of hydroquinone and KCl. There are two anodic peaks at $(-59$ and 459 mV) and two cathodic peaks at $(+119$ and -128 mV). In this case the two pairs of cathodic and anodic peaks are very well defined.

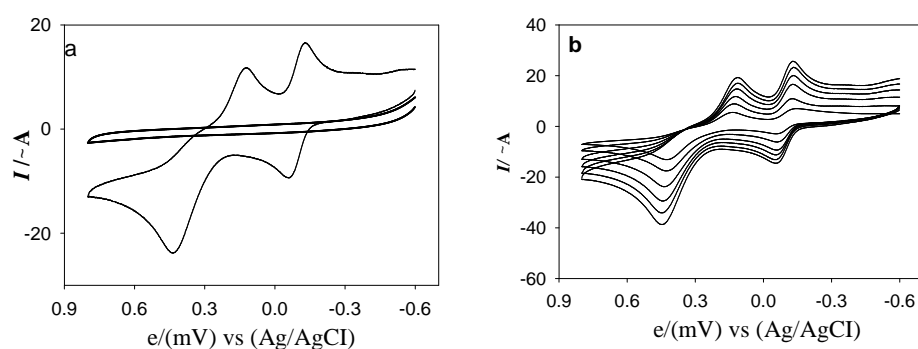
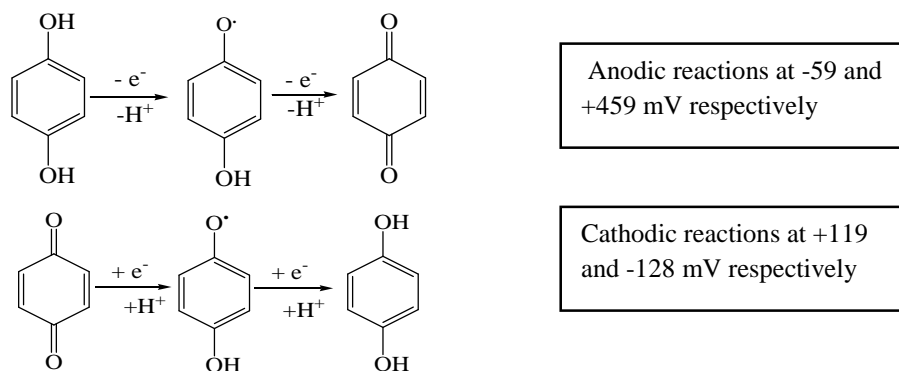


Fig. 8a. Cyclic voltammograms of 1 mM hydroquinone solution in 0.1 M KCl and the supporting electrolyte 0.1 M KCl at 100 mVs^{-1} scan rate. b. CV of hydroquinone at 20, 50, 100, 150, 200, 250 mVs^{-1} scan rate.

The redox reactions involve in this system may be represented as two one-electron transfer reaction for both anodic and cathodic processes. Anode and cathode corresponds to the following reactions:



Cyclic voltammograms of hydroquinone in KCl at different scan rates are shown in Fig. 8b. The potential and current of all the CVs for both pair of peaks are listed in Table 2. Like that of catechol, the peak current increases with scan rate, the cathodic peak potentials are gradually decreased and the anodic peak potentials are gradually increased as the scan rate increased. This behavior indicates that the redox system is diffusion controlled.

Table 2. Current-potential data, peak potential separation, peak current ratio of the voltammograms of 1 mM hydroquinone in 0.1 M KCl at different scan rates.

ν Vs^{-1}	$\nu^{1/2}$	E_{pa1} V(-)	E_{pc1} V(-)	ip_{a1} $\mu A(-)$	ip_{c1} $\mu A(+)$	E_1 $E_{pc1} \sim E_{pa1}(V)$	ip_{a1}/ip_{c1}
0.020	0.141	0.054	0.121	5.65	6.43	0.067	0.878
0.050	0.223	0.057	0.125	8.25	10.80	0.068	0.764
0.100	0.316	0.060	0.130	11.29	16.73	0.070	0.675
0.150	0.387	0.064	0.136	13.30	20.65	0.072	0.644
0.200	0.447	0.070	0.140	15.10	22.9	0.070	0.659
0.250	0.500	0.075	0.148	17.26	26.94	0.073	0.640

ν Vs^{-1}	$\nu^{1/2}$	E_{pa2} V(+)	E_{pc2} V(-)	ip_{a2} $\mu A(-)$	ip_{c2} $\mu A(+)$	E_2 $E_{pc2} \sim E_{pa2}(V)$	ip_{a2}/ip_{c2}
0.020	0.141	0.422	0.132	13.10	6.89	0.554	1.901
0.050	0.223	0.426	0.125	17.60	10.83	0.551	1.625
0.100	0.316	0.434	0.128	23.89	16.45	0.562	1.452
0.150	0.387	0.443	0.132	29.51	19.93	0.575	1.480
0.200	0.447	0.447	0.134	34.23	23.19	0.581	1.476
0.250	0.500	0.449	0.130	38.84	27.53	0.579	1.410

ν = Scan rate; $\nu^{1/2}$ = SQRT of scan rate; E_{pa} = Anodic peak potential; E_{pc} = Cathodic peak potential; ip_a = Anodic peak current; ip_c = Cathodic peak current; E = Peak potential separation;

With the increasing SQRT of scan rate ($\nu^{1/2}$), the peak currents increases, giving the conclusion that the process is diffusion controlled and to some extent adsorptive controlled. Peak current function increases linearly with scan rate. Which implies that the electrochemical process under the investigation do not followed by any chemical process (Golabi and Nimatollahi 2000). The change of potential with log scan rate shows that for 1st pair of peaks the slope is not zero but for the 2nd pair of peaks it is almost zero. The peak current ratio for the 1st pairs is less than unity and for 2nd pair it is greater than unity. So, for the 1st pair the process is nearly reversible and for the 2nd pair the process is not reversible.

The effect of concentrations of hydroquinone on cyclic voltammograms found to be similar as that of catechol.

The cyclic voltammograms of 1 mM hydroquinone and acetate buffer and the buffer solution are shown in Fig. 9. Here the redox behavior of hydroquinone has been modified. There is only one pair of anodic (at +502.3 mV) and cathodic peaks (at -18.3 mV). Which implies that the redox process of hydroquinone in acetate buffer involve one two-electron transfer reactions for both cathodic and anodic processes.

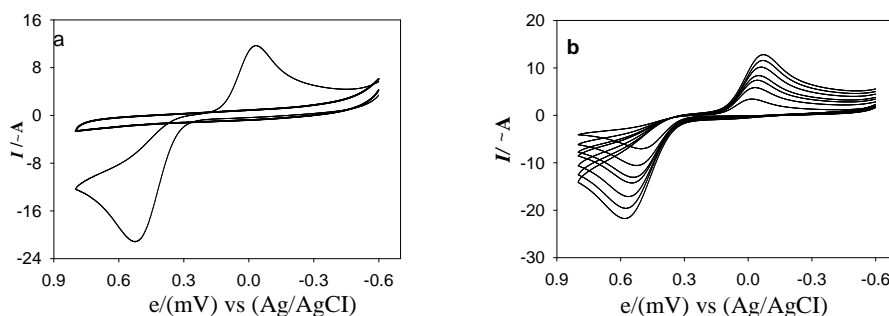
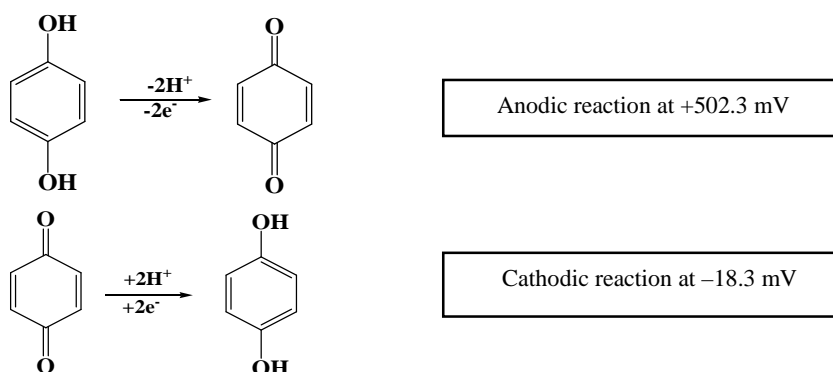


Fig. 9a. Cyclic voltammograms of 1 mM hydroquinone solution in acetate buffer and supporting electrolyte acetate buffer at 100 mVs^{-1} scan rate. b. CV of catechol solution at 20, 50, 100, 150, 200, 250 mVs^{-1} scan rate.

Two-electron transfer reactions at anode and cathode may be represented as follows:



A series of cyclic voltammograms of 1 mM hydroquinone in acetate buffer at different scan rates are shown in Fig. 9b. The pattern of change of peak currents and peak positions with scan rate are similar to that in KCl solution. It is observed that the peak current increases with scan rate, indicating that the redox system is diffusion controlled. The peak current ratio is found to be greater than unity, which implies the system may be quasi-reversible.

The cyclic voltammograms of hydroquinone various concentrations in acetate buffer solution of were studied at 50 mV/sec scan rate. With the increase in concentration there is a gradual linear increase in peak current suggesting the diffusion controlled process.

The redox behavior of hydroquinone in the phosphate buffer is almost similar to that in the acetate buffer. There is only one pair of anodic and cathodic peaks but the peak positions are very much different. One pair of peaks implies that the redox process of hydroquinone in phosphate buffer involve one two-electron transfer reaction for both cathodic and anodic processes and follows same mechanism. The probable reaction would be as like in the acetate buffer.

The cyclic voltammograms of 1 mM resorcinol in 0.1 M KCl and KCl are shown in Fig. 10a. There is one anodic peak at (+908 mV) and one very weak cathodic peak at about (-200 mV). The process is almost irreversible.

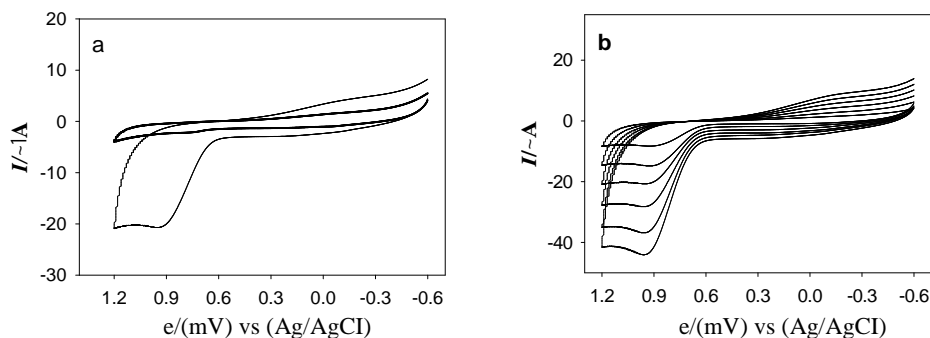
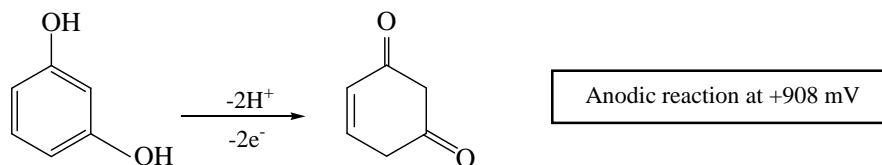


Fig. 10a. Cyclic voltammograms of 1 mM resorcinol solution in 0.1 M KCl and the supporting electrolyte 0.1 M KCl at 100 mVs^{-1} scan rate. b. CV of resorcinol solution at 20, 50, 100, 150, 200, 250 mVs^{-1} scan rate.

The reactions involve in the redox process can be explained in terms of electron transfer mechanism, corresponding to the overall one two-electron transfer reaction such as:



A series of cyclic voltammograms of 1 mM resorcinol in KCl at different scan rates were taken and are shown in Fig. 10b. The potential and current of all the CV are listed in Table 3. From the figure it is seen that, the peak potentials are almost same as the scan rate increased. This behavior can be described by extremely slower charge propagation, probably due to difference in solvation and or permeability.

Table 3. Current-potential data, peak potential separation, peak current ratio of the voltammograms of 1 mM resorcinol in 0.1 M KCl at different scan rates.

ν Vs^{-1}	$\nu^{1/2}$	E_{pa} V(+)	E_{pc} V(+)	i_{pa} $\mu\text{A}(-)$	i_{pc} $\mu\text{A}(+)$	$E = E_{pc} - E_{pa}$ (V)	i_{pa}/i_{pc}
0.020	0.141	0.882	0.255	9.10	0.51	0.627	17.843
0.050	0.223	0.890	0.249	16.60	1.03	0.641	16.116
0.100	0.316	0.910	0.242	21.89	1.45	0.668	15.096
0.150	0.387	0.915	0.240	30.51	2.53	0.675	12.059
0.200	0.447	0.922	0.238	38.23	3.59	0.684	10.649
0.250	0.500	0.932	0.235	48.84	5.63	0.697	8.675

ν = Scan rate; $\nu^{1/2}$ = SQRT of scan rate; E_{pa} = Anodic peak potential; E_{pc} = Cathodic peak potential; i_{pa} = Anodic peak current; i_{pc} = Cathodic peak current; E = Peak potential separation.

It was also observed that the peak current increased with scan rate. The flux to the electrode surface was considerably smaller at slow scan rates than it is at faster rates. As the current is proportional to the flux towards the electrode the magnitude of the current becomes lower at slow scan rates and higher at high scan rates. The general conclusion is that the redox system is diffusion-controlled. The peak current ratio was found to be greater than unity, which implies the system may be irreversible.

The cyclic voltammograms of resorcinol of various concentrations (1, 2 and 3 mM) in 0.1M KCl solution at glassy carbon electrode at 50 mV/sec scan rate. The peak current increases with the concentration also give the idea that the system may be diffusion controlled.

The cyclic voltammograms of 1 mM resorcinol in acetate buffer and phosphate buffer were studied in the similar way. The redox process in resorcinol in both the buffer systems were almost identical to that in KCl solution.

CONCLUSION

Both catechol and hydroquinone gave two anodic and two cathodic peaks in KCl solution. This indicates that the redox process involves two one-electron transfer reactions at glassy carbon electrode. On the other hand in acetate buffer and in phosphate buffer catechol and hydroquinone gave one anodic and one cathodic peak indicating that in these supporting electrolytes the redox process involves one two-electron transfer reaction in both anode and cathod. Both catechol and hydroquinone showed quasi-reversible behavior at glassy carbon electrode. Resorcinol gave only one sharp anodic peak and a very weak cathodic peak in all media except slight difference in peak potential. This indicates that in these supporting electrolytes each of the redox reaction involves two electron transfers. The peak current ratios were found to be greater than unity.

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