

Full Paper

Pre-Treated Cadmium Oxide Modified Carbon Paste Electrode Sensor for Catechol and Hydroquinone in the Presence of Resorcinol

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Abstract- In this study, by utilizing the co-precipitation method we prepared a cadmium oxide nanoparticle and analyzed by XRD, SEM, and EDS techniques. By utilizing prepared nanoparticles carbon paste electrode was modified and pretreated with 0.1M NaOH and sensing environmental pollutants. The CdO/PMCPE brings enhanced sensitivity in an electrochemical investigation of Hydroquinone (HQ) and Catechol (CC) in the existence of Resorcinol (RS) by using cyclic voltammetry (CV) and differential pulse Voltammetric techniques (DPV); The experimental parameter such as scan rate and concentration study was carried out for the determination of CC and HQ, the CdO/PMCPE is utilized for individual and concurrent determination of CC, HQ, and RS, then the detection limit for CC, HQ, and RS found to be 0.506 μM , 0.442 μM and 0.651 μM respectively. The effect of conflict was analyzed by using the Differential Pulse Voltammetry. The electrode showed excellent stability and reproducibility, the prepared sensor can be used for the detection of CC, HQ, and RS in real sample analysis.

Keywords- Catechol; Hydroquinone; Resorcinol; Cadmium oxide nanoparticle; Pre- treated carbon paste electrode

1. INTRODUCTION

Catechol, Hydroquinone, and Resorcinol are highly utilized as intermediates in the area of pesticides, beauty product industries, pharmaceuticals, dyes, rubber productions, and various industries which are closely related to human life [1-8]. These compounds are known as environmental contaminants because of their unregulated discharging from a variety of sources, mighty toxicity, and low degradability in nature, also harmful to the environment and organisms [8-16]. Therefore, the detection and evaluation of di-hydroxy benzene isomers in biological systems execute a decisive role in industrial research and environmental monitoring. Many methods have been constructed to determine and evaluation of HQ, CC, and RS some of them are listed below fluorescence [17], synchronous fluorescence [18], chromatographic techniques [19], and spectrophotometer [20]. As compared to these techniques detection of CC, HQ, and RS by electrochemical technique has more importance because of very less time-consuming, low cost, high sensitivity, and quite easy to handle. However other methods have liabilities such as time-consuming, high cost, low sensitivity, and difficulty while handling the instruments [21-29].

Nanotechnology has extensive attention in all fields due to its individual physical and chemical properties. Metal oxide nanoparticles showed high chemical, thermal, and electric conductivity and large surface areas such as copper oxide (CuO), iron oxide (Fe₂O₃), magnesium oxide (MgO), and cadmium oxide (CdO) nanoparticles [30-33]. The CdO nanoparticle (CdO/NPs) is used in lots of areas such as pigments, paint, petrochemical products, medical curative, and electronic devices [34-36]. It is a transparent conductive oxide material, in the visible light electromagnetic spectrum (EMS) shows both high conductivity and high optical transparency with a direct energy band gap of 2.3eV [37-39]. The synthesized CdO/NPs utilized in the preparation of pretreated modified carbon paste electrode (PMCPE) for the application as a biosensor due to their excellent biocompatibility, tunable porosity, and large surface area; the modified electrode was used as electrochemical sensors and examined its electrochemical parameter [40-44].

In this work, the CdO/PMCPE was established and employed for the simultaneous determination of CC, HQ, and RS in the presence of 0.2 M PBS (pH 7.4) with a scan rate of 0.05 Vs⁻¹. The present electrode exhibits enhanced sensitivity and stability towards the determination of CC, HQ, and RS by both CV and DPV techniques.

2. EXPERIMENTAL SECTION

2.1. Reagents and chemicals

CC, HQ, and RS having 99% purity were procured from Hi-media and of analytical grade used without further purification. 25×10⁻⁴ M each stock solution and 0.1 M NaOH is prepared in DD water. sodium hydroxide (NaOH) and Cadmium nitrate tetrahydrate [Cd(NO₃)₂.4H₂O];

KCl, $K_4[Fe(CN)_6]$, Na_2HPO_4 , NaH_2PO_4 were procured from Merck. PBS (0.2 M pH 7.4) was used as a supporting electrolyte. Fine 50 mM particle size graphite powder and silicone oil purchased from Fluka were utilized for the preparation of carbon paste, and double distilled water was used for all parameters.

2.2. Apparatus

CV and DPV investigations were performed by a model CHI660c with the standard three-electrode system, are the reference electrode (Ag/AgCl), the Platinum thin wire as a counter electrode, and the carbon paste electrode as the working electrode, 1) Bare carbon paste electrode (BCPE) also 2) CdO pre-treated modified carbon paste electrode (CdO/PMCPE) as working electrode.

2.3. CdO nanoparticles preparation

The synthesized CdO nanoparticles data already discussed in the previous paper, reference number [54].

2.4. Preparation of CdO/PMCPE and BCPE

70:30 graphite powders and silicon oil mixture were ground to prepare Bare CPE, The CdO-MCPE was prepared by grinding the mass ratio adding 2, 4, 6, and 8mg of prepared CdO/NPs with BCPE and the same procedure as fallow in BCPE. Through the electrochemical pre-treated method, CdO/PMCPE was prepared using 0.1 M NaOH as a supporting electrolyte; Finally, The CdO-PMCPE was chosen for further investigation.

3. RESULTS AND DISCUSSION

3.1. Characterization

The synthesized CdO nanoparticles data results were already discussed in the previous paper, reference number [54].

3.2. Comparison of the amount of CdO/MCPE

Different amount of prepared CdO NPs 2,4,6 and 8 mg in CPE was investigated in 1M KCl for 1 mM $K_4[Fe(CN)_6]$ at 0.05 Vs^{-1} of scan rate by CV as demonstrated in Figure 1. Redox peak current for $K_4[Fe(CN)_6]$ increases up to 4 mg, thereafter 6 and 8 mg it is going to decrease.

3.3. Electropolymerization of CdO/MCPE

CdO/MCPE was executed 0.1 M NaOH electrochemical pre-treatment method by using cyclic voltammetry and oxidized at the intervals -0.4 to 1.2 V sweeping potential with scan rate

0.05 Vs^{-1} as indicated in Figure 2A then the developed electrode was known as CdO/PMCPE. In the plot of the graph between the number of cycles versus anodic peak current, the maximum current at 8 cycles was observed then after it will decrease, it is indicated in Figure 2B.

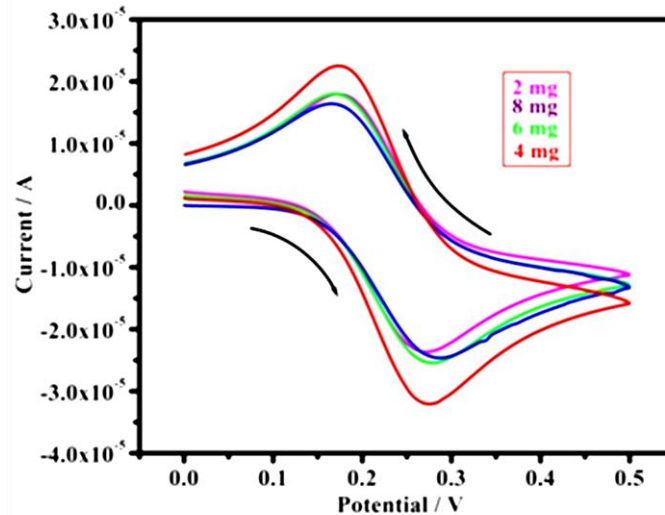


Figure 1. Cyclic voltammograms of 1 mM $\text{K}_4[\text{Fe}(\text{CN})_6]\cdot 3\text{H}_2\text{O}$ at a disparate weight of CdO in a CPE in 1 M KCl at a sweep rate of 0.05Vs^{-1}

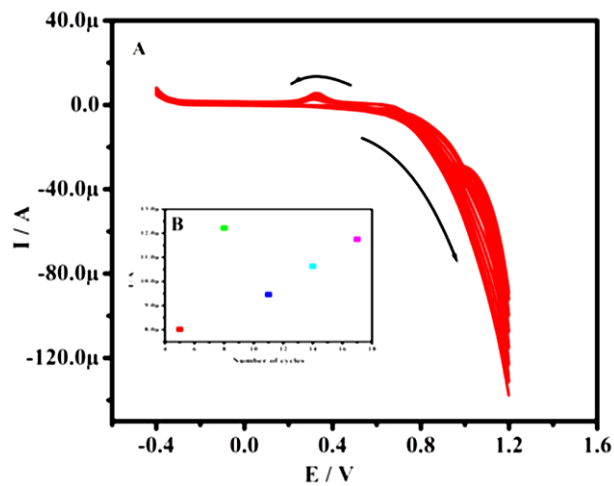


Figure 2. A) Cyclic voltammograms for sodium hydroxide (0.1 M NaOH) at a sweep rate of 0.05Vs^{-1} for 8 multiple cycles at CdO/MCPE; B) Graph of the number of cycles versus I_{pa}

3.4. Electrochemical activity of CC at CdO-PMCPE

Figure 3 indicates the cyclic voltammograms recorded for $10 \mu\text{M}$ CC in pH 7.4 (0.2 M PBS) at both Bare CPE (red-colored line) and CdO-PMCPE (blue-colored line) at the sweep rate of 0.05Vs^{-1} . At BCPE, the oxidation peak was observed with an anodic peak potential

(E_{pa}) at 0.15 V. Here we noticed that poor I_{pa} response because of the transfer of slow electrons. Although, the CdO-PMCPE demonstrated an increase in I_{pa} with an oxidation peak at 7.99 μA under congruent conditions. This shows that the CdO-PMCPE enhanced towards CC electrocatalytic activity.

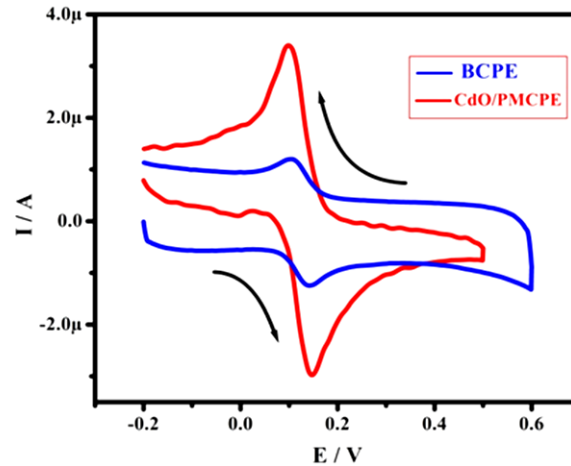


Figure 3. Voltammograms for 10 μM CC at BCPE (blue color line) and CdO/PMCPE (Red color line) at pH7.4 (0.2M PBS) with Sweep rate 0.05 Vs^{-1}

3.5. Influence of sweep rate of CC at CdO-PMCPE

A series of CVs collected for different sweep rates from 0.05 to 0.5 Vs^{-1} is noticed in Figure 4A. The impact of sweep rates on the electrocatalytic response of CC is investigated at CdO-PMCPE. Figure 4B illustrates the sweep rate increases the E_{pa} of CC also enhanced and a linear relationship was noticed and it can be demonstrated as I_{pa} (μA) = $9.19236\text{E}^{-4}(\text{Vs}^{-1}) + 5.208\text{E}^{-5} \mu\text{A}$ ($r^2 = 0.99365$) [55,56]. The process of electrode for CC at the CdO-PMCPE is adsorption controlled.

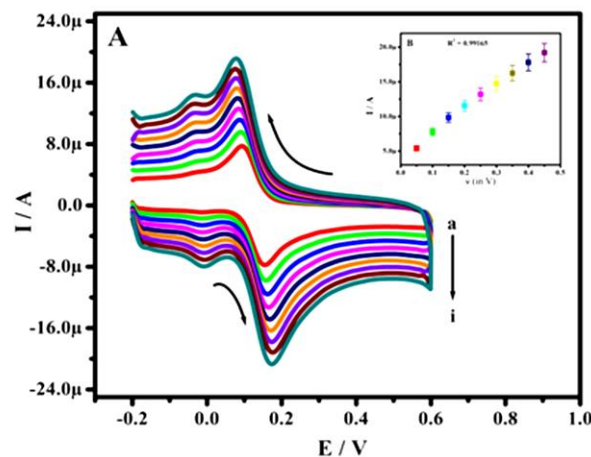


Figure 4. A) Cyclic voltammograms at CdO/PMCPE of 10 μM CC in pH7.4 (0.2 M PBS) with different scan rates 0.05-0.5 Vs^{-1} (a-j); B) Graph between I_{pa} of CC versus SR

3.6. Impact of Concentration of CC at CdO-PMCPE

Figure 5A depicted the DP Voltammograms taken for different concentrations of CC and Figure 5B indicated that enhanced in anodic peak current due to a gradual increase in concentration of CC from 10 to 90 μM . The correlation coefficient was established to be (r^2) = 0.99668 and by using equations E_1 and E_2 the LOD and LOQ for CC were determined [45,46]:

$$\text{LOD} = 3S/M \text{ -----}(E_1)$$

$$\text{LOQ} = 10S/M \text{ -----}(E_2)$$

where 'S' is the standard deviation and 'M' is the slope obtained and LOD is found to be 0.506 μM and LOQ is 1.687 μM . The contrast of CdO-PMCPE for CC with already published modified electrode data is pointed out in Table 1 [47-53].

Table LOD for CC, HQ and RS with already reported data

Sl. No.	Electrodes	Reaction	Method	LOD=3s/m (μM)			Ref
				CC	HQ	RS	
1	GNPs/CNF/Au	Oxidation	DPV	9	9	-	[37]
2	Influence of micelles/GCE	Oxidation	DPV	8.0	3.0	-	[38]
3	GCE/Pt-MnO ₂	Oxidation	DPV	15	3	-	[39]
4	MWCNTPMG/GCE	Oxidation	DPV	5.8	1.6	-	[40]
5	PNR/MCPE	Oxidation	CV	6.4	4.9	-	[41]
6	AuPdNF/rGO/GCE	Oxidation	LSV	0.5	0.8	0.7	[42]
7	MCNTs/GCE	Oxidation	DPV	0.6	0.6	1	[43]
8	CdO/MCPE	Oxidation	DPV	0.5	0.41	0.6	This work

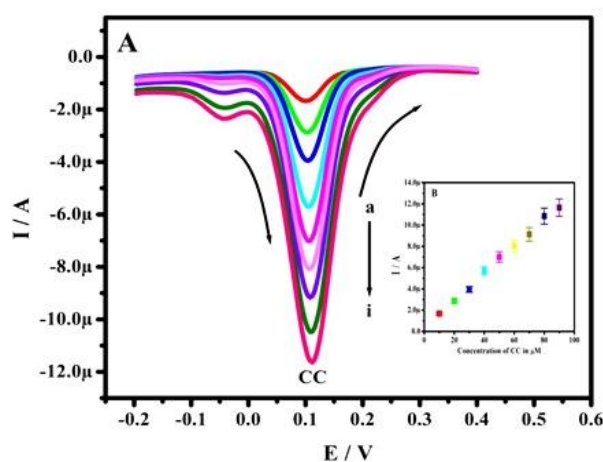


Figure 5. A) The concentration of CC DPV recordings [a-j: 10 μM -90 μM] of CC CdO/PMCPE in the existence of pH7.4 (0.2 M PBS); B) Graph between CC peak current versus concentration

3.7. Response of HQ at BCPE and CdO-PMCPE

Figure 6 displays the CV recordings for 10 μM HQ at Bare CPE and CdO-PMCPE, at Bare CPE (red color line) HQ responding the lowest I_{pa} but at CdO-PMCPE (blue color line) exhibited a sensitive voltammogram with enhanced peak current with a sharp peak.

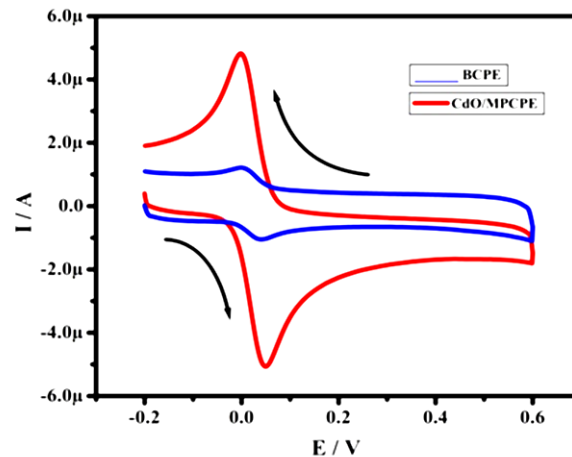


Figure 6. Voltammograms for 10 μM HQ in pH 7.4 (0.2 M PBS) with scan rate 0.05 Vs^{-1} on BCPE (blue color line) and CdO/PMCPE (red color line)

3.8. Impact of sweep rate on the oxidation of HQ at CdO-PMCPE

Here, the 10 μM HQ was examined with various scan rates from 0.05-0.5 Vs^{-1} , as the scan rate raises the I_{pa} also raises and the E_{pa} of HQ slightly drifted towards the higher potential which is noticed in Figure 7A. The graph between sweep rates versus I_{pa} of HQ was plotted and the result we got very good linearity with the correlation coefficient $I_{\text{pa}} (\mu\text{A}) = 3.09847E^{-5}(\text{V/s}) + 3.83709E-6\mu\text{A}$ ($r^2 = 0.98912$) which is shown in Figure 7B [55,56]. This brings us to the process of the electrode was adsorption controlled.

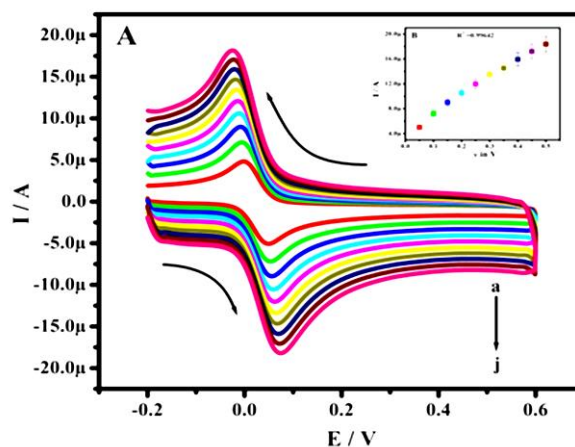


Figure 7. A) Cyclic voltammograms of 10 μM HQ at CdO/PMCPE in pH7.4 (0.2 M PBS) at Scan rates (a-j: 0.05-0.5 Vs^{-1}); B) Graph between sweep rates versus I_{pa}

3.9. Concentration study on HQ oxidation at CdO-PMCPE

Figure 8A brings that as the rise in HQ from 20 to 80 μM , the I_{pa} also rises. In Figure 8B the graph of I_{pa} versus HQ concentration was drawn, and the proper correlation coefficient was constructed to be $(r^2) = 0.9969$. The LOD is 0.442 μM (E_1) and LOQ is 14.734 μM (E_2). The LOD of CdO-PMCPE for CC with already published modified electrode data is pointed out in table [47-53].

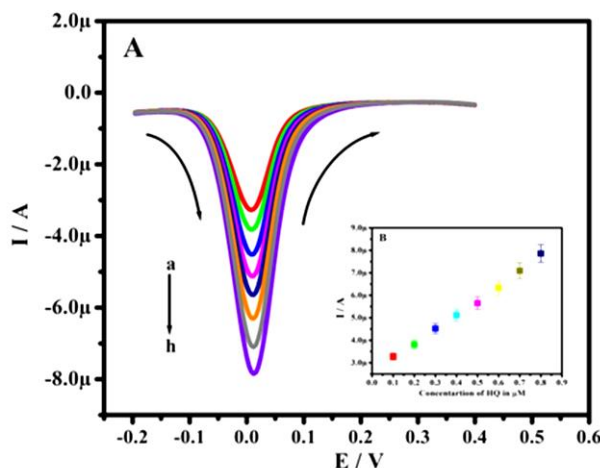


Figure 8. A) The concentrations of HQ DPVs (a-h: 20 μM -80 μM) at CdO/PMCPE in the existence of pH 7.4 (0.2 M PBS); B) Linearity graph between HQ concentration versus I_{pa}

3.10. Electrochemical response of RS at CdO-PMCPE

Figure 9 indicates the CVs collected for 10 μM RS in pH 7.4 at both BCPE (red colored line) also at CdO-PMCPE (blue colored line) with a sweep rate of 0.05 Vs^{-1} . At BCPE, the RS oxidation peak was observed with an anodic peak potential of 0.62 V.

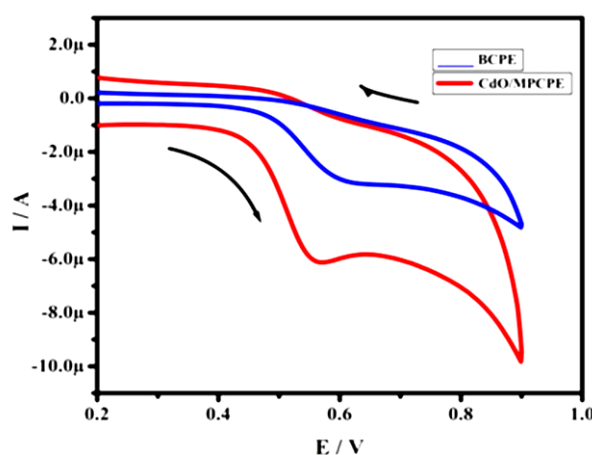


Figure 9. Cyclic voltammograms of 10 μM RS in pH 7.4 (0.2 M PBS) and scan rate of 0.05 Vs^{-1} at CdO/PMCPE (red color line) and BCPE (blue color line)

Here we noticed poor I_{pa} response because of the transfer of slow electrons. Although, the CdO-PMCPE demonstrated an increase in I_{pa} with an oxidation peak at 6.2 μA under proper conditions. This shows that the CdO-PMCPE an enhanced towards RS electro-catalytic activity.

3.11. Influence of scan rate on RS oxidation at CdO/PMCPE

From Figure 10A, different CVs were collected for a variety of sweep rates from 0.05 to 0.5 Vs^{-1} . The influence of sweep rates on the oxidation of RS is examined at CdO-PMCPE. Figure 10B demonstrates that as the sweep rate raises, the RS I_{pa} raises and noticed a linear relationship. Equation of linear regression indicated as $I_{pa}(\mu\text{A})=9.19236E^{-4}(\text{Vs}^{-1})+5.208E-5\mu\text{A}$ ($r^2=0.9988$) [55,56]. The electrode process of RS at the CdO-PMCPE is adsorption controlled.

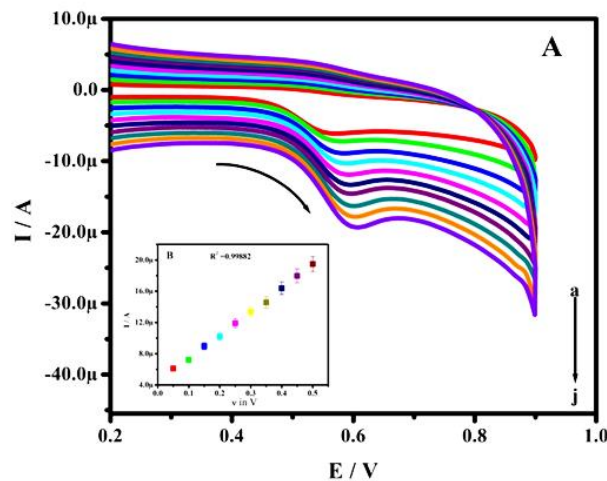


Figure 10. A) Cyclic voltammograms of 10 μM RS at CdO/PMCPE in pH 7.4 (0.2 M PBS) with scan rates: (a-j: 0.05-0.5 Vs^{-1}); B) Graph between I_{pa} versus sweep rate of RS

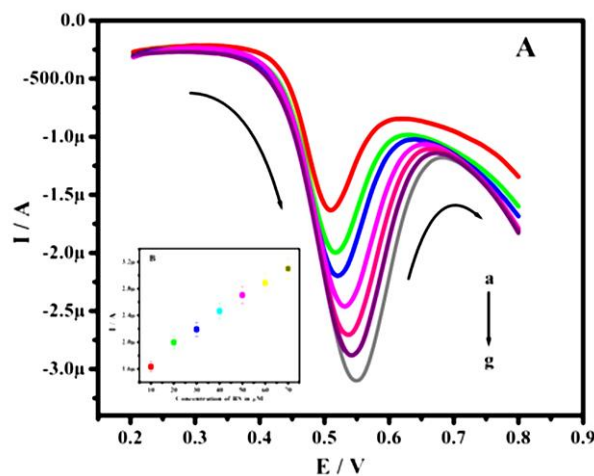


Figure 11. A) The Concentrations of RS DPVs: (a-h: 10 μM -70 μM) on CdO/PMCPE in the presence of pH 7.4 (0.2 M PBS); B) Graph between concentrations of RS versus I_{pa}

3.12. Concentration study on RS oxidation at CdO-PMCPE

Figure 11A exhibits the DP voltammograms at various concentrations of RS, in Figure 11B the Voltammograms indicate the gradual increase in I_{pa} because of the increase in the concentration of RS from 10 to 70 μM which is evolved that RS concentration influenced the oxidation of RS. The proper correlation coefficient was established to be $(r^2) = 0.99165$. The Detection limit was found to be 0.651 μM (E_1) and LOQ is 2.17 μM (E_2). The LOD of CdO-PMCPE for RS with already published modified electrode data is pointed out in Table [47-53].

3.13. CC, HQ, and RS concurrent investigation at CdO-PMCPE

CdO-PMCPE is manipulated to the investigation of CC, and HQ in the presence of RS. Figure 12 displays the cyclic voltammograms collected for 10 μM CC, HQ, and RS in pH-7.4 (0.2 M PBS), the blue colored line for BCPE and the red colored line is for CdO-PMCPE, at BCPE current response is less in simultaneous determination of CC, HQ and RS but the current response is greater in CdO-PMCPE and well-defined oxidation peak potentials at 0.15 V, 0.045 V and 0.57 V, respectively. Electro-oxidation of CC, HQ, and RS are shown in Scheme 1. It recommends that the electrochemical temperament of CdO-PMCPE is excellent with respect to the BCPE in the simultaneous determination of CC, HQ, and RS.

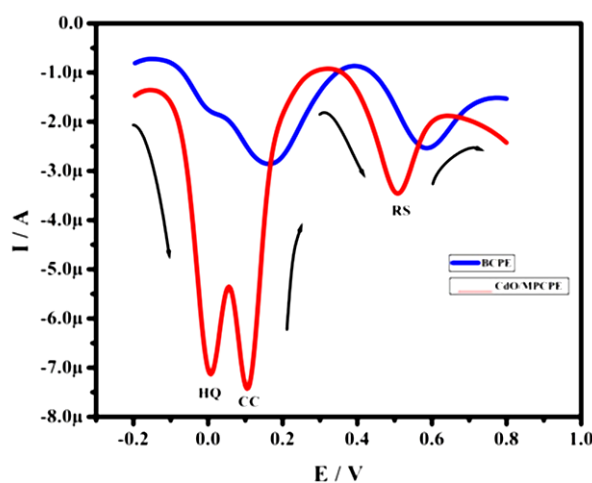


Figure 12. DPV for resolve of 10 μM CC, HQ, and RS with pH 7.4 (0.2 M PBS) at BCPE (blue line) and CdO/PMCPE (Red line) at sweep rate 0.05 Vs^{-1}

3.14. Conflict analysis of CC and HQ at CdO-PMCPE

Figure 13A and Figure 13B are the voltammograms for CC, HQ, and RS composite in 24 mL 0.2 M pH 7.4 at a sweep rate 0.05 Vs^{-1} , Figure 13A shows that the peak current of CC gradually rises due to the increase in CC concentration only, the peak current of HQ and RS remains constant and Figure 13B examines that the I_{pa} of HQ enhances gradually as a function of raise in a concentration of HQ only, the peak current of CC and RS kept sustained. These

results recommended that the CC and HQ arise separately and the issue of conflict was completely fixed at CdO-PMCPE.

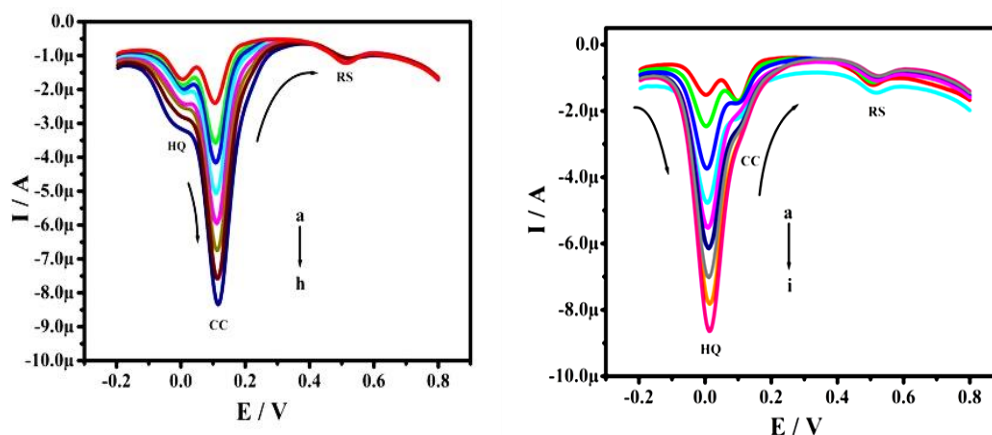
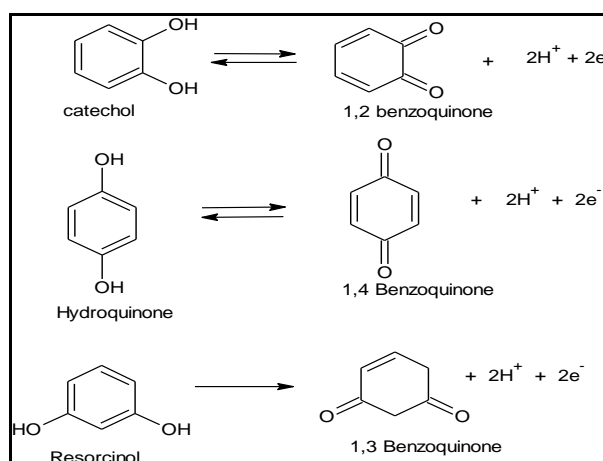


Figure 13. A) DP Voltammetry of CC (a-h: 10 μM –80 μM) in the existence of constant 10 μM HQ, 10 μM RS at pH 7.4 (0.2 M PBS); B) DP voltammetry of HQ (a-i: 10 μM -90 μM) in the existence of constant 10 μM CC, 10 μM RS at pH 7.4 (0.2 M PBS)



Scheme 1. Oxidation Scheme of CC, HQ, and RS

4. CONCLUSION

CdO-PMCPE shows fine selectivity and sensitivity having magnificent electro-catalytic nature on the oxidation of CC, HQ, and RS. It shows an adsorption-controlled electrode process. HQ and RS were taken as probable interference for interference study. CdO-PMCPE has excellent performance to investigate CC, and HQ separately and conflict is sufficient to recognize, also detects RS having LOD 0.506 μM , 0.442 μM , and 0.651 μM respectively. Thus, the CdO-PMCPE is a promising kit for the determination of environmental pollutants.

Declarations of interest

The authors declare no conflict of interest in this reported work.

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