



Sensitive voltammetry method for analysis of the antioxidant pyrogallol using a carbon paste electrode with CdS nanoparticle

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ABSTRACT

A voltammetry method for the determination of pyrogallol (PY) was developed employing a carbon paste electrode (CPE) modified with CdS nanoparticle that was synthesized by microwave. The effect of different parameters i.e. time and irradiation power on the morphology and the sample's particle size have been investigated. The synthesized nanostructures were characterized by X-ray diffraction and scanning electron microscopy. The optimized condition for time and power consumption to prepare CdS nanoparticles was obtained 4 min and 360 W. Cyclic voltammetry study of the modified electrode indicated that the oxidation potential shifted towards a lower potential by approximately 106 mV and the peak current was enhanced by 2 fold in comparison to the bare CPE. The effect of pH and interferences from some inorganic salts and organic compounds were studied. The usability of this method for the quantification of pyrogallol was investigated with differential pulse voltammetry (DPV). Under the optimal conditions, the peak current was proportional to pyrogallol concentration in the range of 7.0×10^{-7} to 3.0×10^{-4} mol L⁻¹ with a detection limit of 4.8×10^{-7} mol L⁻¹. These values are satisfactory for application to real samples. Finally, the developed method was successfully used for the analysis of real samples.

1. Introduction

Pyrogallol is an important kind of polyphenol with a strong reduction property and has been widely used as a useful antioxidant and scavenger free radicals. Free radicals are chemical species possessing an unpaired electron that can be considered as fragments of molecules that are generally very reactive [1]. Oxidative stress is a phenomenon caused by an imbalance between the production and accumulation of oxygen reactive species (ROS) in cells and tissues and the ability of a biological

system to detoxify these reactive products. Oxidative stress is the cause of many human diseases like diabetes, thyroid disorders, hypertension, arthritis, etc [2, 3]. Antioxidants are compounds that act as inhibitors of oxidative damage [1]. Therefore, the determination of pyrogallol is very important in chemistry, environment, clinic, and biological system. There are several methods have been developed for the determination of pyrogallol, such as UV spectrophotometry [4], chromatography [5], electrochemiluminescence [6]. However, these instrumental methods have suffered some disadvantages such as being time-consuming, solvent usage intensive, and requiring expensive devices and maintenance [1]. Electroanalytical methods

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represent a cheaper alternative with the possibility of field analysis. Modified electrodes have been widely used in sensitive and selective analytical methods for the detection of trace amounts of antioxidants [7-10]. These modified electrodes have shown good electrocatalytic properties, high surface-to-volume ratios, high stabilities, and fast electron transfer rates [8]. Nanomaterials may be mixed with electrode materials or may be attached to the electrode surface [11]. Carbon paste electrode has been extensively modified with nanomaterials and has been used to measure a wide range of compounds. This is due to modified electrodes with nanoparticles that can enhance the electrocatalytic property, stability, fast reaction rate, and reproducibility in the results. Various types of nanoparticles such as metal nanoparticles [12], metal oxides [13], and even composite nanoparticles [14-16] have been used to modify the electrodes. Several strategies based totally on bodily and chemical methods have been advanced for the synthesis of controlled size and form nanoparticles [17]. Examples of these procedures involve solvothermal techniques, template-assisted, kinetic increase management; sonochemical reactions, and thermolysis of unmarried-supply precursors in ligating solvents [17]. Microwave Irradiation (MWI) methods provide simple and speedy routes to the synthesis of nanoparticles on account that no excessive temperature or excessive strain is needed. The heating effect is generated by the interaction of the dipole moment of the molecules with electromagnetic radiation at high frequency. Moreover, MWI is specifically beneficial for a managed huge-scale synthesis that minimizes the thermal gradient consequences [18-20]. To our knowledge, no study has reported the electrocatalytic oxidation of pyrogallol by using CdS modified carbon paste electrode.

Thus, in the present work, CdS nanoparticles have been synthesized using a microwave irradiation process. In this process, changes in power and time of microwave irradiation caused different CdS morphologies. A modified carbon paste electrode is fabricated by using CdS nanoparticles for the determination of pyrogallol.

2. Experimental

2.1. Chemicals and Reagents

Pure pyrogallol, thioacetamide (TAA), sodium dihydrogen orthophosphate (NaH_2PO_4), disodium hydrogen phosphate (Na_2HPO_4), sodium phosphate (Na_3PO_4), orthophosphoric acid (H_3PO_4), sodium hydroxide (NaOH), hydrochloric acid (HCl), $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and graphite powder were obtained from Merck. The buffer solutions were prepared from orthophosphoric acid and its salts in the pH range of 1 to 7. All the aqueous solutions were prepared by using double distilled water. High viscosity paraffin ($d = 0.88 \text{ kg L}^{-1}$) from Merck was used as the pasting liquid for the preparation of the carbon paste electrodes.

2.2. Apparatus

Electrochemical studies were performed using a Metrohm polarograph potentiostat-galvanostat (Metrohm Computrace 797-VA). Three-electrode system consisted of a bare CP and CdS/CP electrode as the working electrode, Ag/AgCl (3 M KCl) as the reference electrode, and a platinum wire as the auxiliary electrode. A Metrohm 691 pH/Ion meter was used for pH measurements. Solutions were degassed with nitrogen for 10 min prior to the recording of the voltammogram. X-ray diffraction (XRD) patterns were recorded by a Philips, X-ray diffractometer using Ni filtered Cu K α radiation. Scanning electron microscopy (SEM) images were obtained from LEO instrument model 1455VP.

2.3. Synthesis of CdS Nanoparticles

To synthesize CdS nanoparticles, 1mmol of $\text{Cd}(\text{NO}_3)_2$ was solubilized in 10 mL of water and stirred for 10 min. Subsequently, 0.1 mmol of thioacetamide has added to the solution and stirred for 15 min. Then the mixture was left for the reaction to proceed by cyclic microwave radiation at 360 W power for 4 min. Each cycle was 90 s long, and composed of 30 and 60 s for the on and off periods, respectively. The final precipitate was washed with water and ethanol, dried at 80 °C for 24 h.

2.4. Preparation of bare carbon paste electrode and modified carbon paste electrode

The modified carbon paste electrode was prepared by hand mixing 0.2 g of CdS nanoparticles with 0.9 g graphite powder with a mortar and pestle. Then paraffin was added to the above mixture and mixed for 30 min until a uniformly wetted paste was obtained. This paste was then packed into the end of a glass tube (ca. 3.35 mm i.d. and 10 cm long). Electrical connection implemented by a copper wire leads fitted into the tube. A fresh electrode surface was obtained by squeezing out a small portion of paste and polishing it with filter paper until a smooth surface was obtained. Unmodified CPE was prepared in the same way without adding CdS nanoparticles.

2.5. General procedure for the determination of pyrogallol

A 25 ml aqueous solution of analyte-containing 5 ml of 0.1 M phosphate buffer at pH 6 and a

specific amount of sample solution was added to the cell and purged with purified nitrogen for 5 min to remove oxygen (Fig 1) and CV and DPV voltammograms were recorded. The scanning potential was varied from 0 to 0.8 V. Cyclic voltammogram was recorded by the anodic potential scanning at scan rate 50 mVs⁻¹. A renewed surface electrode was used for each measurement.

3. Results and discussion

3.1. XRD analysis

The XRD pattern of the as-obtained CdS nanoparticles was shown in Figure 2. The diffraction peaks observed can be indexed to pure cubic phase CdS with cell constants $a = b = c = 5.3580 \text{ \AA}$ (JCPDS No. 05-0731). The XRD results proved the high crystallinity and purity of the products synthesized by this method. According to XRD data, the crystallite size

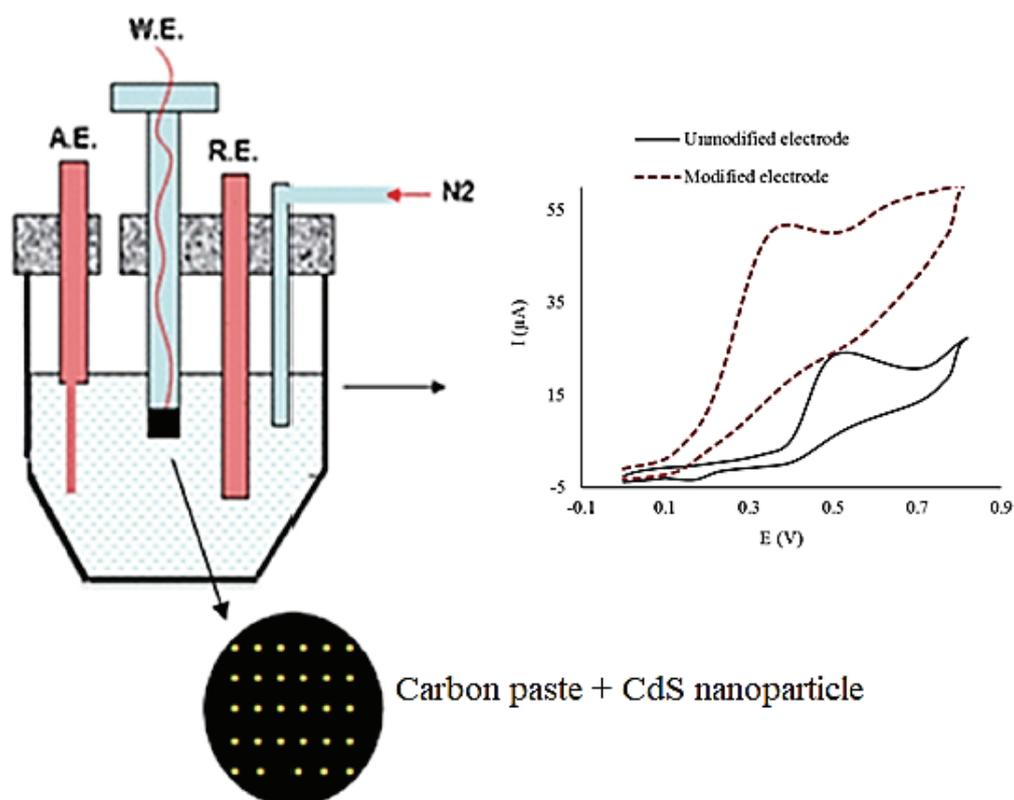


Fig 1. The procedure of voltammetry for determination of pyrogallol

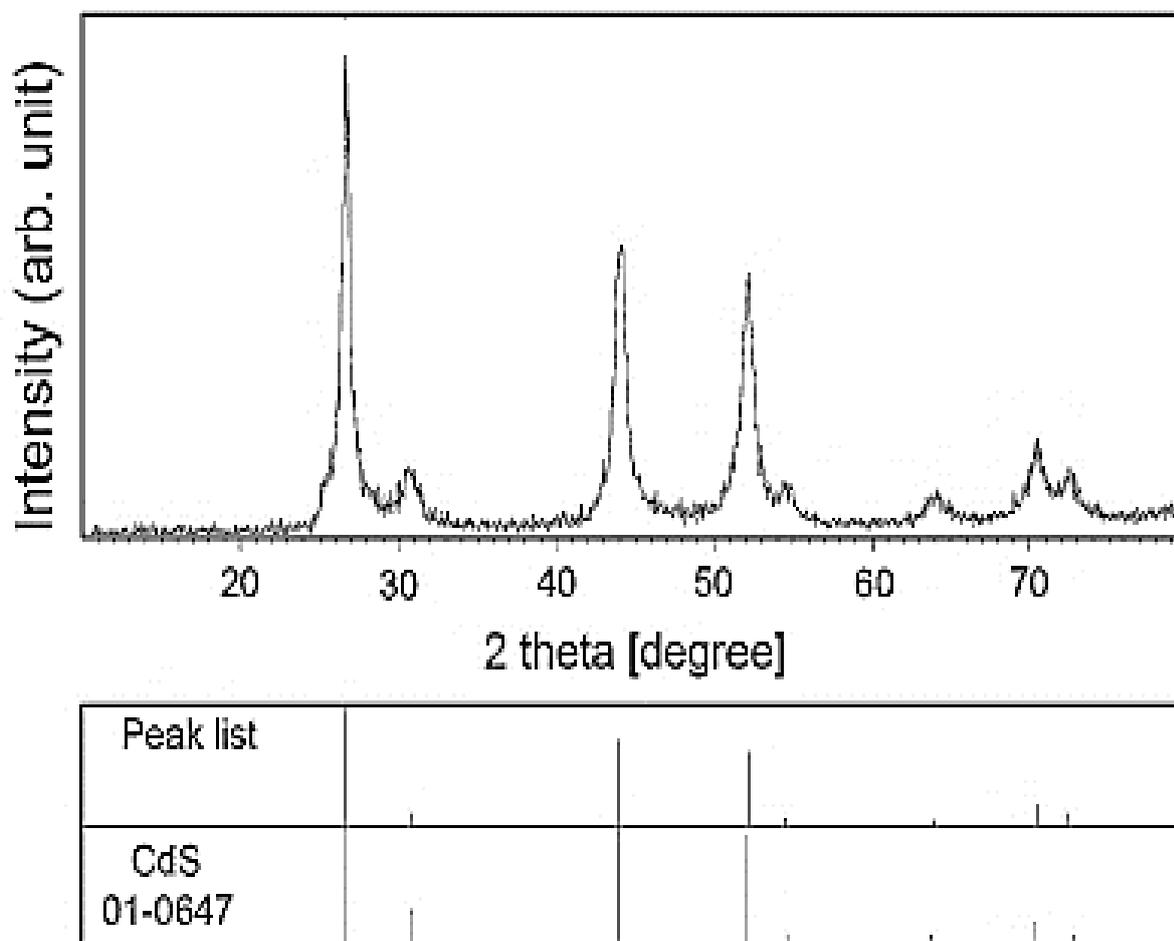


Fig. 2. XRD patterns of the synthesized CdS prepared by microwave irradiation

(Dc) of CdS nanoparticles can be determined by using the Debye-Scherrer formula. The obtained average particle size was found to be 45 nm. The dependence of morphology and the average particle sizes of the products on the irradiation power was also investigated.

3.2. Scanning electron microscopy

SEM images of the CdS obtained with thioacetamide in different powers of 180, 360, and 540 W are shown in Figure 3a–d respectively. As can be seen from SEM images, at 180 W power, due to the insufficient heat of the reaction, cohesive particles are formed. At 540 W, due to the very high energy produced in this power,

the nucleation of the particles is increased, and since the particles have a very active surface, large and cohesive masses are obtained in all test conditions. However, at 360 power, the reaction is faster due to the generation of more free radicals in solution and increased heat production due to the rotation of these active species. The formed nanoparticles have relatively smaller sizes and better distribution. Therefore, the sample prepared in 360 W power and 4 min time due to the creation of nanoparticles in nanometer size according to the scale of images and homogeneous distribution is an optimized condition for time and power consumption to prepare of CdS nanoparticles.

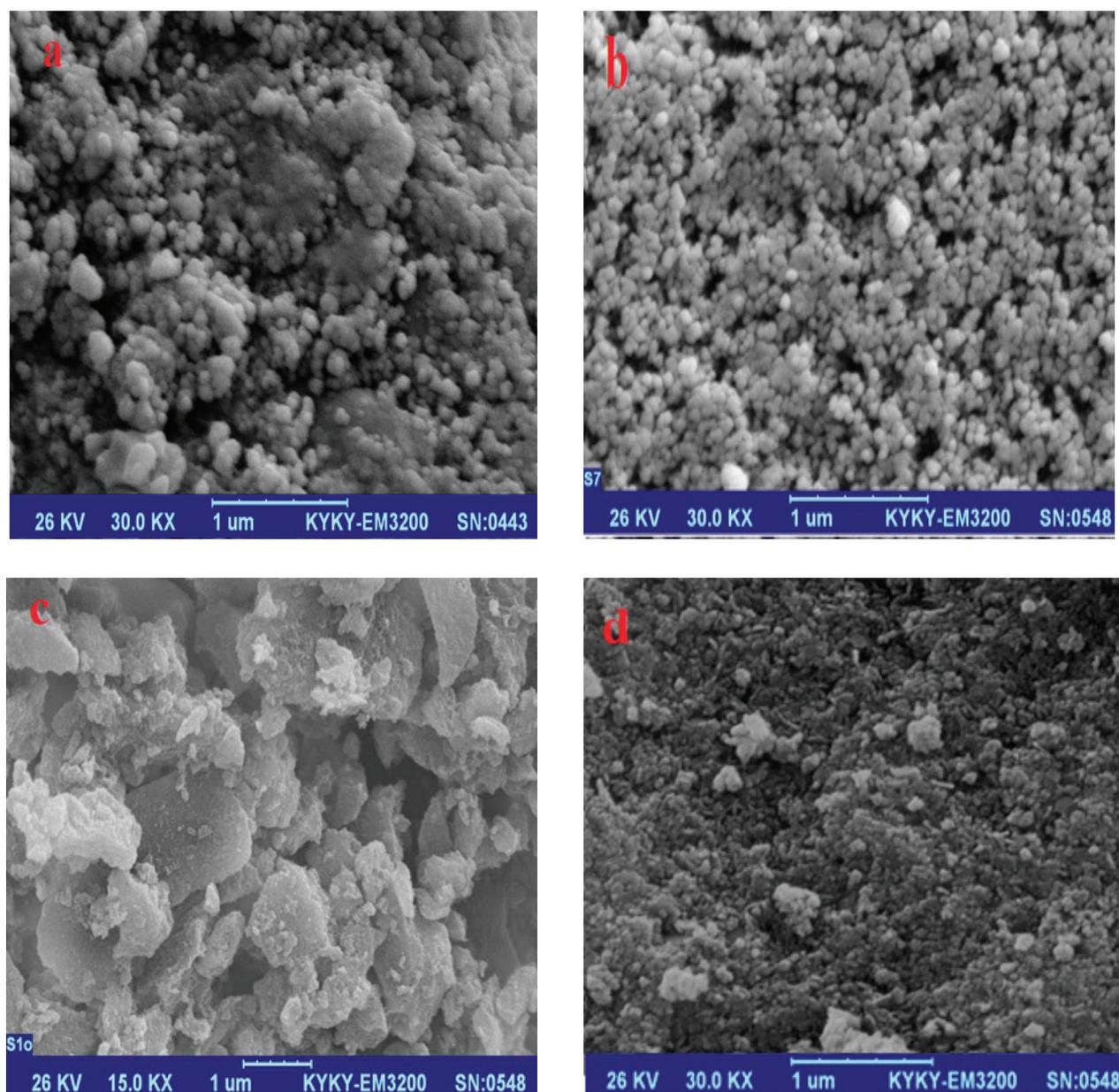


Fig. 3. SEM images of the CdS nanoparticles at (a) 180 W, (b) 360 W and (c, d) 540W

3.3. Electrochemical behavior of pyrogallol at the CdS/CPE

The electrochemical behavior of pyrogallol has been studied in two electrodes. Cyclic voltammetry (CV) was applied to investigate the electrochemical behavior of 0.4 mM pyrogallol in 0.1 M phosphate buffer at pH 6 with a bare CPE and a CdS/CPE (Fig. 4).

As shown in this Fig 3, in the presence of pyrogallol, an irreversible oxidation peak at 0.520

V on the bare CPE attributed to the electrochemical oxidation of pyrogallol. In the case of the CdS / CPE, the oxidation peak of pyrogallol decreased to 0.355 V and the peak current increased by 2.0 times compared with that for the bare CPE. These results suggested that CdS obviously accelerate the electron transfer at the electrode surface and improve the electrochemical performance accordingly.

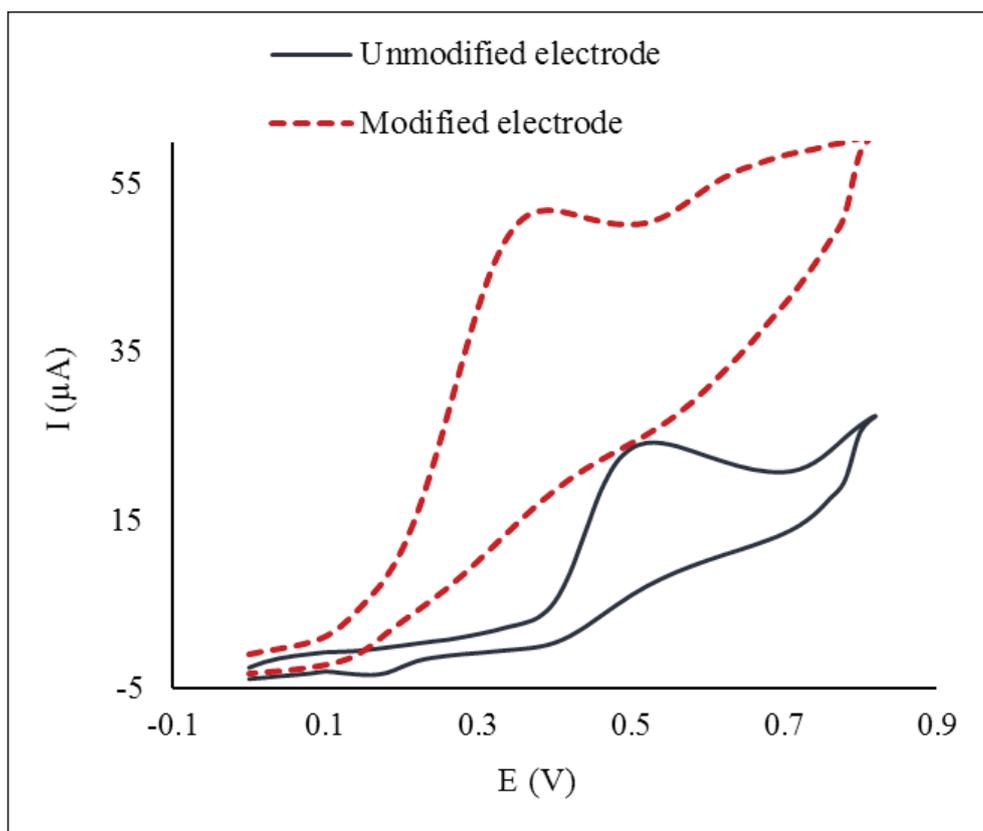


Fig. 4. Cyclic voltammograms of CPE and CdS/CPE at the presence of 0.4 mM pyrogallol in 0.1 phosphate buffer solution (pH 6) at scan rate 50 mVs⁻¹

3.4. Effect of pH

The effect of the pH of the solution on the electrochemical response of pyrogallol was investigated from pH 1.0 to 7. Due to [Figure 5](#), the anodic peak current of pyrogallol increased with increasing pH and the highest peak current was obtained at a pH of 6.0. Additionally, anodic potential peaks shifted by 165 mV to a more negative potential upon increasing pH, which is better for pyrogallol oxidation. Thus, a buffer solution with a pH of 6.0 was selected for further studies. In addition, the relationship between oxidation peak potential (E_{pa}) and pH value was also investigated and the results are shown in [Figure 5](#). As pH value increases from 1.0 to 7.0, the E_{pa} shifts linearly to a more negative potential, obeying the following equation: $E_{pa} = 631.3 - 55.4 \text{ pH}$, $R^2 = 0.9979$. (1) The slope of E_{pa}/pH is -55.4 mV , suggesting that the number of electrons and protons involved in the oxidation of pyrogallol is equal.

3.5. Effect of scan rate

The effect of scan rate on the electrocatalytic oxidation of pyrogallol at the CdS/CPE was investigated by cyclic voltammetry. As can be seen in [Figure 6a](#), the scanning potential increases the peak pyrogallol oxidation shifts to more positive potentials, which imposes a kinetic constraint on the electrochemical reaction. [Figure 6b](#) illustrates that a linear relationship existed between the oxidation peak currents of pyrogallol and the square root ($v^{1/2}$) of the scan rate in the range from 10 to 150 mVs⁻¹, indicating a diffusion-controlled process. The linear regression equation was expressed as $I_{(\mu\text{A})} = 4.178v^{1/2} + 10.657$ ($R^2 = 0.9925$).

3.6. Calibration curve

In order to develop a voltammetric method for the determination of the drug, the DPV mode is selected, because the peaks are sharper and better defined at lower concentrations of pyrogallol

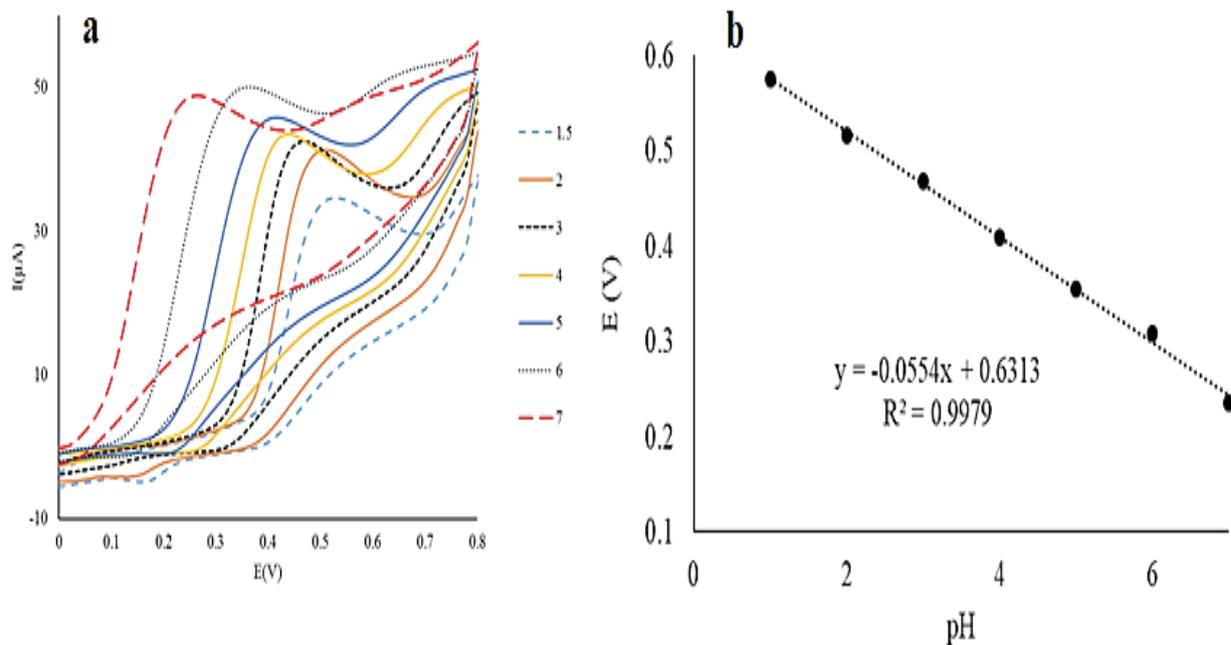


Fig.5. a) Cyclic voltammogram of pyrogallol at different pH
b) Relationship between the peak potential of pyrogallol and pH

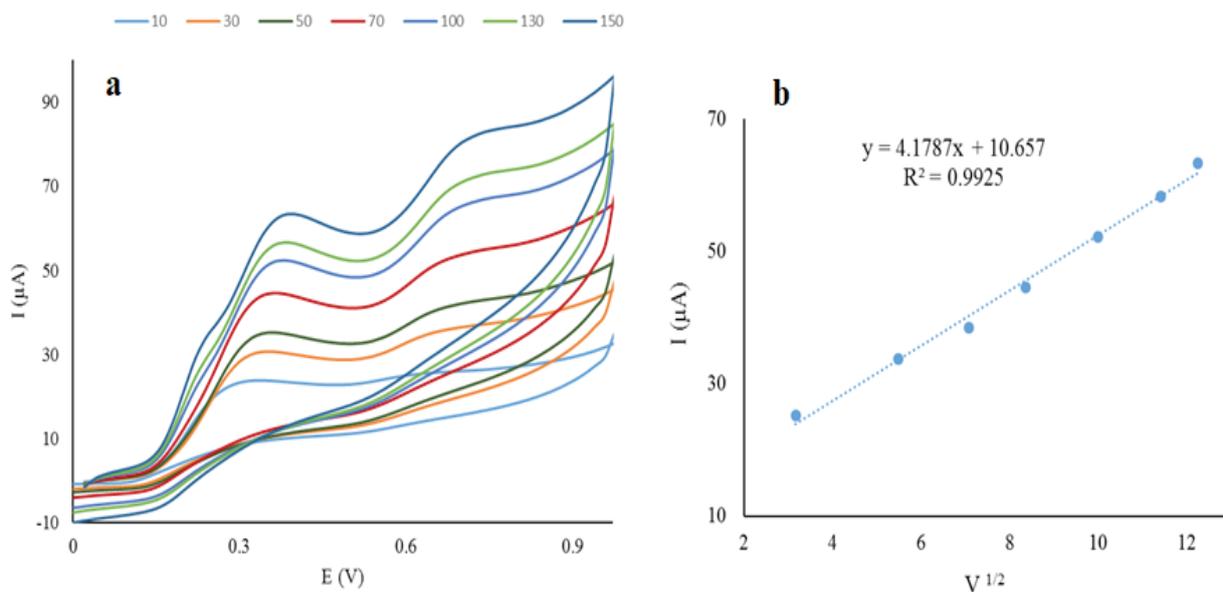


Fig. 6. (a) Cyclic voltammograms of Cds/CPE in the presence of 0.4 mM of pyrogallol in 0.1 phosphate buffer solution (pH 6) at different scan rates (from inner to outer): 10, 30, 50, 70, 100, 130 and 150 mV s⁻¹.
(b) peak current vs. square root of scan rate ($v^{1/2}$).

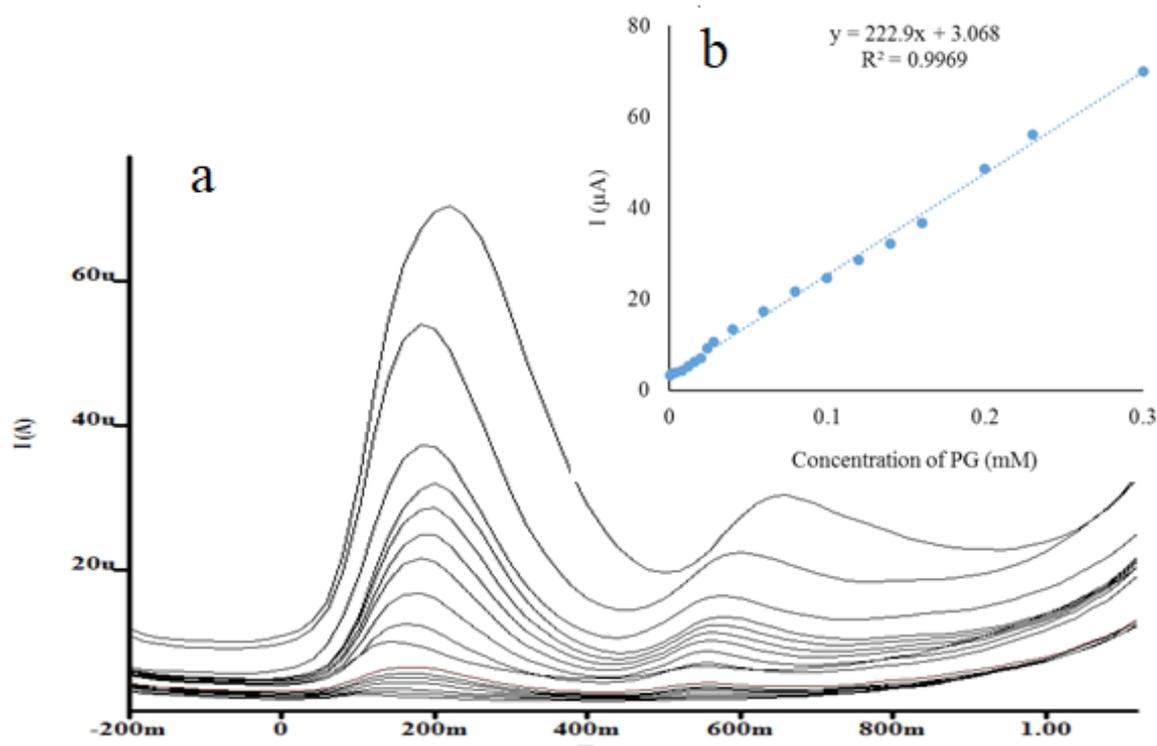


Fig. 7. (a) DPV obtained at a CdS/CPE for different concentrations of pyrogallol (0.7 to 300 μM). Inset: (b) linear relationship between the peak current and concentration of pyrogallol, scan rate: 50 mV s^{-1}

than those obtained by cyclic voltammetry, with a lower background current, resulting in improved resolution. According to the obtained results, it was possible to apply this technique to the quantitative analysis of pyrogallol. The phosphate buffer solution of pH 6 was selected as the supporting electrolyte for the quantification of pyrogallol as it gave maximum peak current at pH 6. DPV obtained with increasing amounts of pyrogallol showed that the peak current increased linearly with increasing concentration, as shown in Figure 7. Using the optimum conditions described previously, linear calibration curves were obtained for pyrogallol in the range of 7×10^{-8} to 3×10^{-4} M. (Fig. 7 Inset). The linear equation $I = 222.9x + 3.068$ ($R^2 = 0.9969$). The limit of detection (LOD), defined as $DL = 3S_b/m$ (where DL , S_b , and m are the limits of detection, the standard deviation of the blank and slope of the calibration graph, respectively) was found to be 0.48 μM of pyrogallol.

3.7. Interference study

To assess the prospects of the electroanalytical assays, several possible interferences of organic and inorganic chemicals were added into the 100 μM pyrogallol solutions to evaluate their effects on the current responses. The results were shows no significant signal change (below 5%) when 1000-fold phenol and hydroquinone Ca^{2+} , urea, Mg^{2+} , and 500 fold of glucose, caffeine, Na^+ , K^+ , Cl^- , and 100 fold of Zn^{2+} , Fe^{2+} , 10-fold folic acid, ascorbic acid were added.

3.8. The repeatability and stability

The repeatability of the CdS/CPE was examined by the determination of 0.8 mM of pyrogallol in 0.1 M phosphate buffer solution at pH=6 with the same electrode 5 times. A relative standard deviation (RSD) value of 3.92% was observed, indicating good reproducibility of CdS/CPE for pyrogallol determination. Furthermore, the

operational stability of CdS/CPE was investigated by the CV method every 2 days in 23 weeks. Only a small decrease of current (about 4.0%) for 0.1 mM pyrogallol was observed, which can be attributed to the good stability of the modified electrode.

3.9. Analysis of real samples

In order to evaluate the applicability of the proposed method in the real sample analysis, it was used to detect pyrogallol in tap water and green tea. The concentration of pyrogallol in these samples was obtained according to the calibration curve, and the results are listed in Table 1. In addition, the accuracy was evaluated by performing a recovery test after spiking the samples. The recovery is between 96.0 and 103.7 %, indicating that the determination of pyrogallol CdS/CPE is accurate and feasible. Analytical parameters obtained here

were compared with results obtained by other methods which show that they are comparable or better than the values reported by other groups (Table 2).

4. Conclusion

The CdS nanoparticles were synthesized via the microwave method. This method is rapid, simple, and can be easily controlled. The influence of different power on the morphology of the products was investigated. Cyclic voltammetry and differential pulse voltammetry determination of pyrogallol was successfully performed using CPE modified with CdS, which has shown an electrocatalytic effect on the oxidation of pyrogallol. The proposed electrode exhibited good sensitivity and stability for the determination of pyrogallol, with reduced overpotential. The pyrogallol peak current is linear

Table 1. Results of the recovery tests for pyrogallol using the CdS

Row	Sample	Spiked (μM)	Found (μM)	Recovery (%)
1	Tap water	0	Not detected	-
		40	38.4	96
		150	155.6	103.7
2	Green Tea	0	Not detected	-
		40	41.3	103.25
		150	153.1	102

Table 2. Comparison of detection limits and linear ranges obtained with the proposed electrode for determination of pyrogallol with those obtained by others

Working system	Linear range (μM)	Detection limit (μM)	Ref.
Pretreated Pt electrode	1-100	0.6	21
Au/CNT/PPY/HRP	1.6-22.4	1.24	22
GCE/poly(PPY-CD)	1 - 10	1.8	23
SPCE	10-1000	0.33	24
SiO ₂ -CPE	2-300	0.7	25
CdS-CPE	0.7-300	0.485	This work

from a concentration range of 0.7 μM to 300 μM with an excellent R^2 value of 0.9969. The detection limit of this modified electrode was found to be 0.48 μM and good reproducibility, high stability was obtained for the determination of pyrogallol using this electrode. The content of pyrogallol in tap water and green tea was successfully determined with CdS/CPE, which indicated the modified electrode is useable for the determination of pyrogallol concentration in real samples.

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