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Development of simple and accurate voltammetric method based on Surfactant Modified Graphene for the Determination of Rutin

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This examination relates to the electrochemical analysis of Rutin (RT) at anionic surfactant Sodium dodecyl sulfate chemically modified graphene paste electrode (SDSMGPE) and its electrocatalytic oxidation or reduction were analyzed using the cyclic voltammetry (CV). The fabricated electrode showed an impressive electrocatalytic movement for RT with a decent electrochemical performance, low over potential and high conductivity. It was found that under ideal conditions (pH7.0) in CV, the oxidation potential of RT decrease to291 mV at the SDSMGPE as compared to a bare graphene paste electrode (BGPE). Calibration plot showed two direct unique reaches of 2.0-10 μ M and 10-60 μ M with a calculated detection limit (LOD) and calculated limit of quantification (LOQ) are found to be 1.17×10^{-8} M and 3.91×10^{-8} M, respectively. Simultaneous investigation was completed with paracetamol (PC), effectively shows two obvious sharp tops with improved current as compared to the BGPE. The SDSMGPE has excellent reproducibility and long term stability with fine recovery in real sample of RT. Field emission scanning electron microscopy (FESEM) examination was performed to check the surface morphology of the BGPE and SDSMGPE and EIS was utilized to explore the electrode and solution interface properties.

Keywords: Rutin; Paracetamol; Graphene sensor; Electroanalysis.

1. INTRODUCTION

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Rutin place a significant function in medical services, for example, hostile to irritation, antioxidation, antidiabetic and so on [1-3]. It has gotten expanding consideration from established researchers due to its putative helpful exercises saw in vivo and in vitro, including cancer prevention agent, antidiabetic, anticarcinogenic, neuroprotective, cardioprotective, vasoprotective, cytoprotective and antiosteopenic impacts [4-6]. Over the previous years, rutin have increased huge intrigue in view of their free radical scavenging activities [7], their protective effects on DNA harm [8], and the physiological effects, for instance, threatening to cancer [9,10], enemies of microbes [11,12], calming [13] and hindrance of human platelet collection [14]. Up to the present, rutin has been used clinically as the therapeutic medicine [15, 16], and over numerous arrangements containing rutin are enrolled as medications overall [17]. In this manner, it is huge to build up specific techniques to decide rutin in drugs, nourishments and facility tests. Up until this point, numerous diagnostic techniques, for example chemiluminescence [18], high performance liquid chromatography [19], spectrophotometric detection [20] and capillary electrophoresis [21] have been utilized. Nonetheless, disadvantages of significant expense, monotonous, exorbitant instruments and talented administrator happen in a portion of these strategies. Electrochemical detecting is a fair candidate against these obstacles with high affectability, insignificant exertion, and simple activity [22]. Among of various scientific strategies, electroanalytical techniques perform with them significant focal points, for example, high affectability, low discovery limits, relative straightforwardness, low expenses, less obstructions and convenient field based hardware [23-27]. Since RT is electrochemically dynamic, its electrochemical estimation has been accounted for and some changed cathodes have been utilized to decide it. Particularly, the electrochemical systems subject to different carbon materials have been created to build the affectability and selectivity [28]. Of course, the helpless capacity of the exposed anodes in direct electrochemical movement of various electro dynamic materials has prompted interest in the utilization of go between and changed terminals to catalyze the electrochemical oxidation of RT. Hence, changed cathodes have been extensively used for electrochemical assurance of different mixtures since alteration of the terminal surfaces fundamentally expands the affectability close by noteworthy decline in identification limit and meddling impacts. The use of graphene in the field of electrochemical sensors has prompted the improvement of a thorough stage for expansive electro insightful assessments. The surface change of anodes utilizing electron move go between is a fascinating field with regards to electro insightful science [29-36]. One of the main effects of any arbiter is a decrease of the over potential needed for electrochemical response and improvement of the affectability and selectivity of the technique.

In the current work, the use of surfactant changed GPE electrode for the assurance of RT within the sight of PT. This examination has provoked the improvement of speedy, straightforward and precise voltammetric technique with extraordinary characteristics, for instance, basic arrangement of anode, high affectability, steadiness, and surface recovery with phenomenal reproducibility, high selectivity, wide direct working reach and lower recognition limit stood out from as of late made sensors for RT confirmation.

2. MATERIALS AND METHODS

2.1 Chemicals and reagents

Graphene, Rutin and Paracetamol were acquired from Sigma (USA).Silicone oil. Disodium phosphate, Monosodium phosphates were acquired from Himedia synthetics organization (USA). Phosphate support (PBS) was ready by blending various volumes of 0.1 M monosodium phosphate and 0.1 M disodium phosphate with the fitting measure of 0.2 M NaOH to acquire the ideal pH 5.5–8.0. All arrangements were ready from scientific grade synthetic substances and refined water.

2.2. Preparation of the Bare and Modified electrode

Graphene paste electrode was ready by mixing graphene and silicone oil as staying liquid in a mortar. The accomplished paste was filled in a Teflon tube. The electric contact was set up by a copper wire through the rear of the electrode. The outer layer of electrode was cleaned on a spotless tissue paper to get a smooth surface was noticed [31]. Modified electrode manufactured by immobilization of surfactant.

2.3 Apparatus

Electrochemical estimations were conducted utilizing a electrochemical system. The tri electrode system is utilized in all experiments contained a working electrodes (SDSMGPE and BGPE), a platinum wire as a counter and saturated calomel electrode as a reference electrode with a standard one cell of 25 mL limit.

3. RESULTS AND DISCUSSION

3.1 Surface characterization

Surface modification, active surface area and conductive nature of the BGPE and SDSMGPE were analyzed by using FESEM, CV and EIS methods, respectively.

The FESEM image of BGPE showed that its surface was depicted by a reduced surface and unpredictably formed surface (Fig. A.1). While the FESEM image of SDSMGPE (Fig. B.1) showed entirely unexpected morphology diverged from BGPE, showing a routinely covered surface demonstrating that the fuse of surfactant which very improved the dynamic surface space of adjusted anode and advanced electron move.

The active surface area of SDSMGPE and BGPE was determined by the use of Randles-Sevcik equation [37, 38]. Appropriately, the electrochemically active surface area was analyzed using 1.0 mM [Fe(CN)6] in 0.1 M KCl through CV (Fig. C.1). The obtained active surface area values are found to be 0.0123 cm² (BGPE) and 0.0869 cm² (SDSMGPE). In view of the obtained results, the SDSMGPE

surface area is 7.06 times more than that of the BGPE. Due to the improved surface area the oxidation peak current of the electro active molecule is improved.

EIS is the important and simple approach for the elucidation of surface conductance and resistance behaviors of the equipped electrodes. The EIS scrutiny of BGPE and SDSMGPE was done in the presence of 1.0 mM [Fe(CN)6]⁻in 0.1 M KCl at an optimum conditions. The Nyquist plots of BGPE (curve b) and SDSMGPE (curve a) are plotted using equivalent circuit (Fig. D.1) [39]. The Nyquist plot of BGPE shows a loftier semicircle, however, SDSMGPE displays a smaller semicircle, characterizing that the electron transfer nature electro active molecule at BGPE is significantly improved by the adsorption of a surfactant SDS on its surface. Hence SDSMGPE reveals a high current sensitivity and lower resistance than BGPE.



Figure 1. FE-SEM pictures of, (A) BGPE and (B) SDSMGPE. (C) CVs of [Fe(CN)6] ⁻in 0.1 M KCl at BGPE and SDSMGPE at the scan rate of 0.1 V/s. (D) EIS of 1.0 mM [Fe(CN)6] ⁻in 0.1 M KCl at BGPE (b) and SDSMGPE (a).

3.2 Effect of surfactant concentration, accumulation potential and accumulation time

Optimization of key factors such as surfactant concentration, accumulation potential and accumulation time is needful to provide the high sensitivity for the sensing of RT on the surface of SDSMGPE.

The impact of surfactant concentration on the electrochemical nature of RT was investigated using CV. Form Fig. A.2, we can see that there was an intense improvement in the oxidation peak currents of RT on the surfactant modified electrode compared to BGPE. Although all the four different concentrations show a less enhancement in the oxidation of RT than 10 μ L (Fig. B.2).Hence, the highest oxidation peak current of10 μ L was utilized as optimum for RT analysis. The difference in structure and the corresponding hydrophobic interaction with RT for different concentrations might

explain their different improvement impacts. Other than the hydrophobic interactions electrostatic interactions might also exist, which assumed a significant function in the improved electrochemical reaction of RT at various concentrations of the modifier.

The effect of accumulation potential on the oxidation peak current of RT was studied by the CV method. The accumulation potentials were varied between -0.4 V to 0.0 V (Fig. C.2), here the oxidation peak current of RT is maximum at -0.1 V compare to other potentials (-0.4 V, -0.3 V, -0.2 V and 0.0 V). Thus -0.1 V was employed as the optimum accumulation potential for further experimentation.

The accumulation time is a great influence on the peak current of RT at SDSMGPE. The CV experiment was conducted for RT in PBS at SDSMGPE by varying the accumulation time from 0 to 80 s (Fig. D.2). The oxidation peak current of RT is high at 40 s, indicating that the accumulation of RT sample was sufficient on the surface equipped electrode. Hence, 40 s was selected as optimum accumulation time for further study.



Figure 2. (A) CVs for 0.1 mM RT in 0.1 M PBS (pH 7.0) at different concentrations of SDS on the surface of BGPE. (B) Plot of Concentration of SDS v/s Peak current of RT. (C)Plot of accumulation potential v/s peak current of 0.1 mM RT at SDSMGPE. (D) Plot of accumulation time v/s Peak current of 0.1 mM RT at SDSMGPE.

3.3 Electrochemical oxidation of RT on BGPE and SDSMGPE

Fundamental investigations of the electro catalytic activity of SDSMGPE toward the electro oxidation of RT were performed utilizing the CV method. Fig. 3 shows the CVs for the presence and the absence (c) of 0.01 M RT in PBS (0.1 M with pH 7.0) at the BGPE (b) and SDSMGPE (a). The

proposed electrode do not shows any electrochemical actions in the absence of RT. At the surface of the BGPE, the direct oxidation of RT produces not well defined an extremely feeble and wide anodic top at a moderately high certain potential. Be that as it may, an extremely sharp anodic top at a lower positive potential is acquired at the SDSMGPE. A huge upgrade of the anodic peak current related to the sharpness of the peak, which is identified with a decrease of the over potential, uncovered that the modified could go about as an amazingly convincing advertiser to work on the kinetics of the electrochemical interaction.



Figure 3. CVs of the presence (Curve a) and absence (curve b) of 0.1 mM RT at SDSMGPE and BGPE (Cycle c) in 0.1 M PBS of pH 7.0 at the scan rate of 0.1 V/s.

3.4 pH influence

The effect of pH on the oxidation peak current of 0.1 mM RT in factor supporting electrolyte pH was contemplated by CV. It was considered that to be the pH of the medium expanded dynamically, the peak potential liable to moves towards the more negative side, proposing the association of proton in the oxidation response (Fig. A.4). The Epa for the oxidation of RT at the SDSMGPE showed a straight relationship with the pH with the accompanying condition: Epa(V) = 0.597-0.043 pH (V/pH) (R2 = 0.988) [40]. The linearity in the plot can be clarified by the progressions in changes in protonation of the acid-base functions in the particle concerning the noticed slant of 43mV/pH for RT. The investigation of the impact of pH on CV anodic peak current was likewise done to decide the pH an incentive for the greatest sign (Fig. B.4). The stature top current and the better state of bends were observed at 7.0 pH PBS, subsequently which was picked as ideal for this experimentation.



Figure 4. (A) CVs of 0.1 mM RT at SDSMGPE in 0.1 M PBS of different pH values (6.0 to 8.5) at the scan rate of 0.1 V/s. (B) Plot of pH v/s E_{pa}.

3.5 Scan rate impact

The impact of differing scan rate on the peak potential and current of RT was analyzed using CV method. The outcomes in Fig.A.5 and B.5, revealed that the peak currents of RT increased linearly with increased scan rate over the range from 50 to 250 mVs⁻¹ with the liner regression equation of $I_{pa}(A) = 5.961 \times 10^{-6} + 4.440 \times 10^{-6} \upsilon (V/s)(R^2 = 0.997)$, suggesting that the oxidation of RT at the SDSMGPE is adsorption controlled. The plot (Fig. C.5) of E_{pa} v/s the log v shows a fine linearity with the linear regression equation of $E_p(V) = 0.362 + 0.096 \log \upsilon (V/s) (R^2 = 0.977)$ indicating that the electrochemical reaction of RT continues through the electron transfer nature. The number of transferred electron in the redox nature of RT is calculated by the following Laviron's relations [41],

$$E_{p} = E^{0} + \left(\frac{2.3 \text{ R T}}{\alpha \text{ n F}}\right) \log\left(\frac{\text{R T k}^{0}}{\alpha \text{ n F}}\right) + \left(\frac{2.3 \text{ R T}}{\alpha \text{ n F}}\right) \log v \quad \text{Eq. (A. 1)}$$

Slope of $E_{pa} v/s \log v = \frac{2.3 \text{ R T}}{\alpha \text{ n F}} \quad \text{Eq. (A. 2)}$

Where, E^0 is the standard potential, T is the temperature, α is the charge transfer coefficient and n is the number of the electron transfer in RT redox nature, k^0 is the electrochemical rate constant and F is the Faraday constant. The calculated value of n is found to be 1.64 (nearly equal to 2) and the probable reaction mechanism is shown in Scheme 1.



Figure 5. (A) CVs of 0.1 mM RT at SDSMGPE in 0.1 M PBS of pH 7.0 at different scan rates from 0.05 to 0.25 V/s. (B) Plot of υ v/s I_{pa}. (C) Plot of log υv/s E_{pa}.



Scheme 1. Probable redox reaction of RT at SDSMGPE.

3.6 Repeatability and reproducibility

The repeatability of the SDSMGPE was measured by the CV technique for changing RT solution (five times) in PBS at constant SDSMGPE and the RSD of 2.09% displays an acceptable repeatability. The reproducibility of the SDSMGPE was assessed by the CV method at unchanged RT solution in PBS on five distinctly modified electrodes. Here the SDSMGPE delivers the relative standard deviation (RSD) of 2.09%, exhibits an adequate reproducibility.

3.7 Calibration plot

The CV method was used for the estimation of the detectable amount of various concentration of RT in PBS at optimized measuring conditions (Fig. A.6). The calibration graph for RT concentration and peak current at SDSMGPE (Fig. B.6) shows that, the peak current (I_{pa}) increased

continuously with the increase of RT concentration in the range of 2.0-60 μ M. Also, here we observed a two linear regions; primary one is in the range of 2.0-10 μ M and secondary one the range of 10-60 μ M. The LOD and LOQ for both the linear ranges are calculated using the following relations; LOD=3 σ /m and LOQ=10 σ /, respectively [42,43]. The primary linear was selected as main calibration plot and it provides a fine LOD and LOQ values of 1.17×10⁻⁸ M and 3.91×10⁻⁸ M, respectively.



Figure 6. (A) CVs of various concentrations of RT at SDSMGPE in 0.1 M PBS of pH 7.0. (B) Plot of concentration of RT v/s peak current of RT.

3.8 Comparison SDSMGPE with other reported sensors for RT detection

In Table1, the performance of the fabricated sensor is contrasted with different electrodes utilized for RT determination [44-50], reveals that the performance of the SDSMGPE is better or comparable to the reported electrodes.

Modified electrode	Linear range	Limit of detection	References
	(µM)	(µM)	
Poly(p-aminobenzene sulfonic acid)	0.25-10.0	0.7000	[44]
modified glassy carbon electrode			
Glassy carbon electrode coated with	0.001-2.0	0.0006	[45]
graphene nanosheets, chitosan, and a			
poly(amido amine) dendrimer			
Multi-walled carbon nanotube	28.0-210.0	0.7000	[46]
modified glassy carbon electrode			
Carbon nanotube modified glassy	2.5-100.0	0.5000	[47]
carbon electrode			
Gold nanoparticles/ethylenediamine/	0.048-0.97	0.0320	[48]
carbon nanotube modified glassy			
carbon electrode			
Carbon ionic liquid electrode modified	0.08-100.0	0.0160	[49]

Table 1. Comparison of earlier reported methods and LODs of RT with the present work.

by nafion, graphene oxide and ionic			
liquid composite			
Carbon nanotube paste electrode	0.199–9.99	0.0339	[50]
Sodium dodecyl sulfate modified	2.0-10.0	0.0117	Present
graphene paste electrode			analysis

3.9 Simultaneous determination of RT and PC

The principle target of this work is the simultaneous determination of RT in presence PCat SDSMGPE. Fig. 7 shows the CVs obtained for the mixture of 0.05mMPC and 0.05 mMR Tin PBS (pH 7.0) at the BGPE (curve b) and SDSMGPE (curve a). The peaks obtained at BGPE are broad and less sensible for RT and PC. However, SDSMGPE displays two well defined sharp peaks with improved current response as compared to the BGPE with the anodic peak potentials 217 mV and 458 mV for RT and PC, respectively. The obtained result emphatically suggests that SDSMGPE can be effectively utilized for the detection of RT and PC simultaneously.



Figure 7. CVs of 0.05 mM RT and 0.05 mM PC in PBS of pH 7.0 at SDSMGPE (Curve a) and BGPE (Curve b) at 0.1 V/s scan rate.

3.10 Determination of RT in fruit juice sample

The adaptableness of the equipped sensor was verified by the sample spiked method for the examination of RT in fruit juice sample. Here the real sample with different concentrations were diluted with PBS (0.1 M and pH 7.0) and CV was cycled individually at SDSMGPE in improved experimental conditions. The fruit juice sample of RT at SDSMGPE displays an acceptable recovery range from 93.82 to 98.32%. The outcomes are presented in Table 2.

Analytical	Added	Spiked	Found	Recover
Sample	Concentration	Concentration	Concentration	y (%)
	(µM)	(µM)	(µM)	
Fruit Juice	0.10	2.00	1.91	95.97
(n=4)	0.16	4.00	3.83	95.99
	0.24	6.00	5.89	98.32
	0.32	8.00	7.50	93.82

Table 2. Calculated values of percentage of recovery in fruit juice sample.

4. CONCLUSION

The voltammetric behavior and redox mechanism of RT were examined at SDSMGPE by CV in PBS (pH 7.0). During the examination, impact of a few physicochemical parameters like potential scan rate, pH and concentration were investigated. The redox reaction of RT was found to be a reversible with adsorption character. The redox response of RT was viewed as a reversible with adsorption character. SDSMGPE showed a fine electrocatalytic activity for the redox activity of RT, describing by the upgrade of the peak current and the decrease of the over potential, which was apparently a direct result of the bigger compelling surface space of graphene and the more grounded adsorption of surfactant. The proposed method has been successfully used to decide RT in natural product juice test. The proposed method offered an effortlessness and fine exactness with minimal expense

DATA AVAILABILITY

The data presented in this study are available on request from the corresponding author. The data are not publicly available due to privacy

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CONFLICTS OF INTEREST

The authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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