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Iron Oxide Modified Carbon Paste Electrode Sensor for Guanine and Dopamine: A Voltammetric Technique

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Abstract- This article reports a detailed report of the synthesis and depiction of iron oxide nanomaterials (FeONPs) and their electroanalytical application. Electrochemical examination of Guanine (GU) and Dopamine (DA) is performed by synthesized iron oxide nanoparticles modified with carbon paste electrode (CPE) using differential pulse voltammetry (DPV) and cyclic voltammetry (CV) techniques. Peak potential of GU along with DA at FeOMCPE are 0.76 and 0.14V respectively. A good linear response was obtained by CV of GU and DA, in the concentration ranges 10-70 and10-70 μ M respectively and the LOD value for GU along with DA was observed to be 5.3 and 4.6 μ M. The result shows that the FeOMCPE exhibited good analytical performance and high electro-catalytic activity of DA and GU.

Keywords- Iron oxide nanoparticles; Modified carbon paste electrode; Guanine and Dopamine; Cyclic voltammetry

1. INTRODUCTION

Nanoscience will bring more benefits and improvements to the modern world. It has wide applications; such as medicines, electronics, cosmetics, paints, textiles, solar cells, study of tumors, anticarcinogen drugs, nanowires, nanofibers, textiles, and catalysts etc. Focusing on "nanomedicine," nanoparticles are a popular topic in the study of ophthalmic preparations because they function as nano-carriers in ocular medication delivery systems. In a rapid Covid

test, magnetic nanoparticles are used to detect coronavirus antibodies. Nanoparticles, such as metal, oxide, semiconductor, and composite nanoparticles, have been used in electrochemical sensors and biosensors. Their unique properties include catalysis of electrochemical reactions, immobilization of biomolecules, acting as a reactant, labeling biomolecules, and enhancement of electron transfer. Nanotechnology has received much awareness in a wide spectrum of research areas and industrial activities. Nanomaterials have catalytic properties different from those of bulk materials due to their unusual electronic properties, high edge concentration, and high surface area, leading to their application in bioelectronics, biophotonics, and biosensing.

Metal nanoparticles have wide applications. For example, they are used in biomedical and sensor fields because of their smaller size, mechanical, magnetical, and optical properties. The metal nanoparticle-MCPE is used to improve the sensitivity, selectivity, and electron transformation between the electroactive species and the electrode surface. FeONPs are non-toxic and are used as a drug in the treatment of cancer and diagnosis. Due to their magnetic and semiconductor property, they are used in the medical field [1-12].

GU is an organic compound containing the purine group. It is one of the building blocks of DNA and RNA as it is paired with cytosine in the double helix. It is used in the cosmetic industry, paints, pearls, and plastics. A high purine diet leads to gout and kidney stones. People with such conditions avoid the consumption of alcohol and fats, as they block the metabolism of purines [13-20].

DA is called a chemical messenger of our body because it plays an important role in human feelings and the ability to think. It helps us strive, focus and find things interesting. It is also called as happy hormone [21-45].

A lot of work has been put into developing chemically modified electrodes for direct electrochemical analysis of Nucleic acids and the associated nucleosides over the past few years. For instance, Fan et al. proposed a simple method for making uniformly embedded TiO₂ nanocomposite on graphene, which creates a favorable microenvironment for the electrochemical interaction of these purine bases [46]. In order to quickly create a CNT ceramic electrode formed from a sol-gel for the sensitive detection of guanine and adenine in DNA, Abbaspour et al. used microwave irradiation [47]. Guanine and adenine detection techniques that had previously been tried suffered from surface fouling caused by the purine bases' irreversible adsorption to the electrode surface. For use in nanoscale devices and nanoelectronics, nanoparticulate hybrid materials consisting of inorganic solids hold great promise. Fe₃O₄ nanoparticles have attracted interest due to their biocompatibility, strong superparamagnetic properties, low toxicity, easy preparation, and high adsorption ability.

In this work, FeONPs are synthesized by the co-precipitation method. GU and DA are studied individually and simultaneously utilizing CV and DPV techniques. Sensitivity, selectivity, and electroactive species for the estimation of GU and DA are detected by FeOMCPE as an electrochemical sensor. The drawback of purine bases adhering permanently

to the electrode surface is removed by the redesigned electrode using magnetite nanoparticles, the results presented here show that the modified electrode could give electrochemical devices new capabilities. It can be used for the simultaneous determination of GU and DA with extreme sensitivity.

2. EXPERIMENTAL SECTION

2.1. Chemicals and equipment

From Himedia the reagents ferrous sulfate heptahydrate, ferric chloride, ammonium hydroxide, potassium chloride, and Potassium ferrocyanide are purchased. Silicon oil and graphite powder are purchased from nice chemicals. Disodium hydrogen orthophosphate and Sodium dihydrogen orthophosphate are buying from Karnataka fine chemicals. Aqueous solutions are prepared by utilizing deionized water. Guanines along with Dopamine are purchased from Sigma and the solutions are prepared by using NaOH and perchloric acid respectively.

CV contains an electrolytic cell, a data collection system, a current-to-voltage converter, and a potentiostat. The cell contains a reference, a counter, and a working electrode, and an electrolytic solution is made to perform CV and DPV techniques. The synthesized nanomaterials are analyzed by SEM, EDAX, and XRD techniques.

2.2. Synthesis of FeONPs

The co-precipitation method was employed to prepare FeONPs. The procedure of the experiment is as follows, 1M ferrous sulfate heptahydrate and 0.05M ferric chloride are dissolved in 100ml distilled water. Both the solutions are mixed utilizing a magnetic mixer for half an hour. Add 1M ammonium hydroxide solution, slowly under constant stirring for 2 hours. Brown color precipitate was formed. Filtered the settled down brown precipitate and washed it several times by deionized water. The product was calcinated in a muffle furnace at 500^oC for one hour. The synthesized FeONPs are further utilized for characterization [9-12].

2.3. Amalgamation MCPE and BCPE

Homogenous paste of silicon oil and graphite powder is mixed in the ratio of 70:30 with the help of mortar and pestle to prepare BCPE. The resultant paste was packed in a homogenous Teflon cavity. Utilizing butter paper, BCPE was polished mechanically until shiny working surface was formed. The same process follows to the preparation of FeOMCPE but graphite powder, silicon oil with different amount of FeONPs in the ratio 70:30 :(2, 4, 6, 8 mg).

3. RESULTS AND DISCUSSION

3.1. Characterization of synthesized FeONPs

XRD analysis is an important tool to study purity and size of synthesized nanomaterials. XRD pattern of synthesized FeONPs as shown in Figure 1A, the d-space value of the sample matches with standard JCPDS file number 39-1346. Sharp peak shows the crystallinity of the sample. No impurity peaks of FeONPs are noticed, which shows that the high purity of products. Size of the nanoparticle calculated by using Debye-Scherrer formula and it was 14.38 nm. Figure 1B shows SEM image of the nanoparticle. Shape of the nanoparticles is assessed using SEM analysis. The synthesized FeONPs are crystal shaped and arranged without aggregation. Analysis of EDAX shows energy peak of FeO nanoparticle and it contains iron, carbon and oxygen [36-40] (Figure 1C).



Figure 1. a) FeONPs X-ray diffractograms pattern; b) FeONPs SEM image; c) FeONPs EDAX spectrum

3.2. Quantified analysis for certain quantity of FeOMCPE

Figure 2a shows certain weights of FeOMCPE and BCPE were analyzed using PBS (0.2 M) at pH 7.4 for 10 μ M GU with scan rate of 50 mV/s by CV technique. The plot of anodic peak current (I_{pa}) versus different weight of FeONPs are shown in Figure 2b. The weight of

FeOMCPE increases (2, 4, 6, 8 mg) simultaneously, peaks current decreases. 2 mg of FeONPs MCPE shows enhanced peak current and it was optimized for more detailed study.



Figure 2. a) CV for 2 mg, 4 mg, 6 mg, 8 mg of FeOMCPE and BCPE was analyzed at pH 7.4 for 25×10^{-3} M GU with sweep rate of 50 mV/s in 0.2 M PBS; b). Different weight of FeO nanomaterials taken in milligram versus I_{pa}

3.3. Electrochemical interactivity of BCPE and FeOMCPE using potassium ferrocyanide

Electroanalytical interaction of $K_4[Fe(CN)_6]$ (0.2 M) and KCl (1M) at BCPE and FeOMCPE with scan rate 50mV/s were analysed using CV technique. Figure 3 shows the CV of FeOMCPE (solid line) and BCPE (dashed line) in 1 M potassium chloride and 0.2 M Potassium ferrocyanide solutions. The increase in peak height of the FeOMCPE strongly depends on KCl (supporting electrolyte) and gave well-defined peak.



Figure 3. CV of 0.2 M K₄[Fe(CN)₆] in existence of 1 M KCl at a sweep rate of 50 mV/s: a) BCPE (dashed line) and b) FeOMCPE (solid line)

The voltammogram shows peak current 0.74 μ A and 1.2 μ A at BCPE and FeOMCPE respectively. FeOMCPE shows maximum enhanced peak current and it indicates that FeOMCPE is having more charge transfer than BCPE. The increasing peak current shows that FeOMCPE is having a higher surface area than BCPE. The surface area of FeOMCPE and BCPE are calculated employing Randle-Sevcik equation with various sweep rates (50 to 500 mVs⁻¹):

$$I_{p} = 2.687 \times 10^{5} \text{ n}^{3/2} \text{ } \text{D}_{0}^{1/2} \text{ } \nu^{1/2} \text{ } \text{A} \text{ } \text{C}_{0} \tag{1}$$

where peak current (A) is I_p , diffusion coefficient (cm²/s) is D₀, number of electron transfer is n, electroactive surface area (cm²) is A, scan rate (V/s) is v, and concentration of electroactive molecules (mol/cm³) is C₀. The surface area of BCPE and FeOMCPE were obtained to be 0.0238 and 0.039 cm² respectively.

3.4. Electrochemical interactivity of GU and DA at BCPE and FeOMCPE

The amalgamation of carbon paste with various sort of metal oxide nanoparticles significantly enhances the sensitivity of CPE. In this work, FeONPs was examined as a modifier, because it gives superior adsorptive properties. Figure 4a and 4b shows, CV response for electrochemical interactivity of 10 μ M GU and 10 μ M DA of FeOMCPE and BCPE in 0.2 M PBS at pH 7.4 with a sweep rate of 50 mV/s. 2 mg FeOMCPE gives extreme peak intensity values and BCPE give decrepit signals.



Figure 4. CVs of 10μ M a) Guanine b) Dopamine at BCPE (dashed line) and FeOMCPE (solid line) at pH 7.4 with a sweep rate of 50 mV/s in 0.2 M phosphate buffer

At BCPE, GU and DA exhibits electrochemical behavior (Scheme 1), anodic peak was observed with the potential 0.69 V and 0.14 V, and current were found to be 6.92 μ A and 17.4 μ A. At MCPE, GU and DA also exhibit electrochemical behavior, anodic peak was observed

with the potential 0.76 V and 0.15 V, and current were found to be 1.52 μ A and 4.04 μ A. The above result exhibits, the electron transfer reaction of MCPE is more compared to BCPE. Hence the modified electrode shows enhanced conductivity, larger active surface area and good electrocatalytic activity than BCPE.



Scheme 1. Redox mechanism for DA and GU

3.5. Effect of pH on GU at FeOMCPE

In aqueous solutions voltammetric estimations were carried out and are suitable for pH dependent since removal or addition of electron from an analyte especially instigate the loss or uptake of proton. At different pH, the oxidation of 10 μ M GU in PBS with 50 mV/s scan rate were studied over pH from 6.2 to 7.8 using FeOMCPE.



Figure 5. a) CVs of 10 μ M GU at different pH (6.2, 6.6, 7.0, 7.4, and 7.8) with scan rate 50 mVs⁻¹ at FeOMCPE; (b) plot of pH versus anodic E_{pa}; (c) plot of pH versus I_{pa}

For wide range of pH ,the peaks were well defined (Figure 5a). The graph was plotted between pH versus peak current (Figure 5b) and $E_{pa}=0.06055+0.975$ pH (R²=0.9964) is the

linear regression equation. The current of anodic peak increases linearly with increase in pH of the solutions. The oxidation of GU to form 8-oxo-guanine of FeOMCPE was a pH-dependent process, according to the final electrode reaction, and a graph plotted between pH versus peak potential (Figure 5c). Since peak potential increases with increasing pH of the solution and decreases after reaching pH=7.4, pH=7.4 was chosen as the ideal pH for further experimentation.

3.6. Effect of scan rate on GU and DA at FeOMCPE

Effect of sweep rate depends upon how fastened the applied potential is sweeped and it evaluates the process of an electrode reaction (adsorption or diffusion controlled). The scan rate effect of GU and DA of FeOMCPE was scruntinized using CV technique. Figure 6a demonsrates the GU at CV of FeOMCPE with scan rates from 50 to 500 mV/s. In GU, as sweep rate increases, peak currents pogressively increases and potential negligible shift towards positive side. The potential shift is mainly due to the development of adsorption layer at the electrode surface. The graph plotted between scan rate versus peak potential (Figure 6b) and Ipa =0.5606+1.185pH (R²=0.9804) is the linear regression equation. Therefore, this result suggests that the electron transfer process of GU is adsorption controlled and the graph of log scan rate versus log peak current is shown in Figure 6c. The result gave good linearity with correlation coefficient value (R²) and was found to be 0.999.



Figure 6. a) CVs of 10 μ M GU and d) 10 μ M DA of pH 7.4 with various scan rates (50-500mVs⁻¹) in 0.2 M PBS at FeOMCPE; (b and e) Plot of scan rate versus I_{pa}; (c and f) Plot of log of scan rate versus log of I_{pa}

To identify the process of electrode takes place at the electrode surface using CV were recorded on FeOMCPE with scan rates ranging from 50 to 500 mV/s as shown in Figure 6d. In DA, with increase in sweep rate the peak current progressively enhanced and potential

minuscule shift towards both negative and positive side. The graph was plotted between the scan rate versus peak potential (Figure 6e) and $I_{pa} = 0.3528 + 0.7460$ pH (R²=0.9846) is linear regression equation. Therefore, this result suggests that the process of electron transfer was adsorption controlled for DA. Figure 6f is the plot of log of scan rate versus log of peak current. This result gave good linearity with a correlation coefficient value (R²) and was found to be 0.999.

3.7. Effect of concentration on GU and DA at FeOMCPE

Utilizing CV technique the effect of the concentration of GU was studied (Figure 7a). The effective concentration of GU differs in the range 10-70 μ M at pH 7.4 with 50 mV/s scan rate in 0.2 M PBS at FeOMCPE. As the concentration increases, peak current was enhanced gradually and potential gains negligible shifts towards positive side. Figure 7c displays the CV of DA at FeOMCPE with various concentrations between 10-70 μ M, the concentration increases both anodic and cathodic peak current was progressively enhanced with potential negligible shift towards both positive and negative side. Figure 7b and 7d reflect the linearity between Ipa and concentration of both analytes. The corresponding linear regression was expressed as follows: I_{pa}=0.05278+0.02964pH (R²=0.9971) and I_{pa}=0.07512+0.04821pH (R²=0.9978) for both GU and DA respectively.



Figure 7. a) CV of GU and c) DA of pH 7.4 with different concentrations (10-70 μ M) in 0.2 M PBS at FeOMCPE; (b and d) plot of anodic peak current versus concentrations of GU and DA

The limit of detection (LOD) and limit of quantification (LOQ) were calculated using the equations 2 and 3 [41].

$$LOD=3S/M \tag{2}$$

$$LOQ=10S/M \tag{3}$$

Where M is slope of the graph and S is standard deviation.LOD and LOQ values are found to be 5.3 μ M and 17.8 μ M for GU,4.6 and 15.65 μ M for DA respectively. It shows that, FeOMCPE obtain good linearity and also give a low value of LOD and LOQ. The differentiation of LOD for GU and DA with other modified electrodes is shown in Table1.

SI No	Working Electrode	Electrochemical Technique	Detection Limit		Linear range	Ref.
			GU	DA		
1	SWCNT/GCE	DPV		7.0 µM	0.5-100 μΜ	[31]
2	F-MWCNT/GCE	DPV		6.0 µM	0.2-250 μM	[42]
3	MWCNTs/Poly (new fuchsin)MCPE	CV	18.2 μM		0.02-3.1 mM	[43]
4	CILE	CV	7.87×10 ⁻⁸ M		3.0×10 ⁻⁷ - 5.0×10 ⁻⁵ M	[44]
5	MWNTs	DPV	7.5×10 ⁻⁹ M		2.0×10 ⁻⁸ - 5.4×10 ⁻⁶ M	[45]
6	FeOMCPE	CV	5.3 μM	4.6 µM		This work

Table 1. Comparison of LOD of GU and DA with different electrodes

3.8. Simultaneous detection and interference study of GU and DA at FeOMCPE

Compare to CV, DPV demonstrates high current sensitivity. Because of the lower concentration detection, well-defined peak current and magnefied resolution. DPV was used to detect sensitive and selective of GU and DA at FeOMCPE.



Figure 8. a and b) CV and DPV of GU and DA at bare (dashed line) and FeOMCPE (solid line) using 0.2 M PBS at sweep rate of 50 mV/s

Figure 8a and 8b shows simultaneous analysis of GU and DA in PBS (0.2 M) with 50 mV/s sweep rate at BCPE and MCPE utilizing DPV and CV techniques. At BCPE, CV and DPV response peak current and potential of GU and DA were not well separated. In the case of FeOMCPE; it gives two well-defined peaks of current 0.24 μ A and 0.43 μ A and potential 0.281 V and 0.638 V for GU and DA respectively. Hence, MCPE acts as a good electrochemical sensing element for GU and DA.

DPV was used for the analysis of GU and DA at FeOMCPE was studied under optimized conditions. These experiments were carried out by varying the concentrations of GU (10-60 μ M) in the presence of constant concentration DA (10 μ M) in PBS at pH 7.4 using FeOMCPE as shown in Figure 9a.Then, vary the concentration of DA (10-40 μ M) while of GU (10 μ M) concentration was maintained as shown in Figure 9b.To increase the concentration of one analyte increases as peak current was progressively enhanced and negligible shift in potential towards positive side and fixed analyte concentration remains constant.The aforementioned result demonstrates that peak current and potentials for constant analyte did not vary. This finding demonstrates that FeOMCPE have outstanding selectivity and also individually analysed in the mixed solution.



Figure 9. a and **b**) DPVs of different concentration of GU (10–60 μ M) in presence of DA (10 μ M), DA (10–40 μ M) in presence GU (10 μ M) at FeOMCPE respectively with sweep rate 50 mV/s in 0.2 M PBS

4. CONCLUSION

In this work, FeONPs were prepared utilizing co-precipitation method and analyzed by EDAX, SEM and XRD. Electrochemical interactivity and surface area were analyzed using CV technique. The simultaneous and interference study of GU and DA was examined using the DPV technique. The FeOMCPE enhances sensitivity, selectivity, and good electron transferability. From the study of sweep rate, FeOMCPE was recognized as a process of electrode was adsorption-controlled for GU and DA. The redox peak current increases with an

increase in concentration the concentration of analyte and LOD and LOQ values are found at 5.3 and 17.8 μ M for GU,4.6 and 15.65 μ M for DA respectively. It is clear that FeOMCPE obtains good linearity and also gives a low value of LOD and LOQ. Therefore, the preferred method was authenticated for the individual and simultaneous determination of DA and GU.

Declarations of interest

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

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