

Full Paper

Pharmaceutical Electrochemistry: the Electrochemical Behaviour of Paracetamol at ZnO Nanoparticules/1, 2-Napthaquinone-4-Sulphonic Acid Glassy Carbon Modified Electrode

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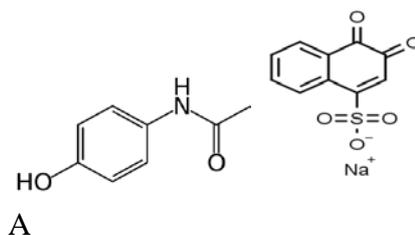
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Abstract- The development of a new modified glassy carbon electrode with ZnO nanoparticules and a polymer film of 1, 2-napthaquinone-4-sulphonic acid have been reported. The electrochemical behaviour of paracetamol in pharmaceutical form at ZnONPs-Nq/GC modified electrode using cyclic and linear sweep voltammetry was investigated. The modified electrode exhibited excellent electrocatalytic activity for the behaviour of PA. The anodic and cathodic peak currents of PA at ZnONPs-Nq/GCME increased significantly when compared with that at a bare glassy carbon electrode and ZnONPs/GCE, also its potential shifted to more negative value. The effect of pH, concentration and scan rate were also investigated. The anodic peak current was linearly proportional to the concentration of PA in the range from 0.088 to 1.1 mM with detection and quantification limits of 30.03×10^{-6} and 10.01×10^{-5} M respectively. The pH 7.6 is the gives value for studying the behaviour of PA at the modified electrode. Some parameters such as the diffusion coefficient (D), the electron transfer rate constant (k_s) and the electron transfer coefficient (α) were calculated and found for the PA oxidation to be 5.998×10^{-6} cm² s⁻¹, 11.279 s⁻¹ and 0.721 respectively. The modified electrode shows high selectivity, satisfied stability and good reproducibility for the detection of PA. This modified electrode was successfully applied to PA determination in pharmaceuticals tablets samples.

Keywords- Paracetamol, ZnO nanoparticules, 1, 2-napthaquinone-4-sulphonic acid sodium, Modified-glassy carbon electrode, Electropolymerization

1. INTRODUCTION

One of the most commonly headaches drugs and relieve pain is paracetamol (PA). Paracetamol (known also acetaminophen) is N-acetyl-p-aminophenol (scheme 1) and works as a pain removal by affecting chemicals in the body called prostaglandins what relieves headache, backache, arthralgia, neuralgia arthritis and postoperative pain. It also reduces temperature by acting on the area of the brain that is responsible for controlling temperature. The over dosage of paracetamol causes liver and kidney damage, skin rashes, inflammation of the pancreas and in rare cases lead to death [1,2].



Scheme 1. Chemical structure of paracetamol (A) and 1, 2-naphthaquinone-4-sulphonic acid sodium (B)

Metal oxides nanoparticales such as ZnO, NiO, TiO₂, Fe₃O₄, Al₂O₃, SnO₂, WO₃ and others play a very important role in chemical modification of electrodes due to it can exhibit unique physical and chemical properties into electrodes surface such as large specific surface area, high electrocatalytic activity, low overpotentials, investigate the reversibility of some redox reactions that are irreversible at most bare electrodes, high selectivity, good stability and enhancement of electron transfer, high surface free energy, and adsorbing biomolecules strongly [3].

Some electrodes can be electrochemically modified using electropolymerization methods. There are three methods to form a polymer film on a surface of electrode: galvanostatic (constant current) method, potentiostatic (constant potential) method and potentiodynamic (potential scanning or cyclic voltammetric) method [4]. The polymer film modified electrodes have large surface area, accelerated of electron transfer, increased active sites, high selectivity and sensitivity [5].

It is very useful to establish a simple, fast, inexpensive, sensitive and accurate method to detect paracetamol in its pharmaceutical forms. There were many methods have been reported for determination PA such as FT-IR Raman spectrometry [6], spectrophotometry [7], spectrofluorometry [8], liquid chromatography [9], liquid chromatography-tandem mass spectrometry [10], micellar electrokinetic chromatography [11], automatic sequential injection analysis [12], colorimetry [13] and capillary electrophoresis [14]. However, these methods have some disadvantages, such as high costs, time-consuming procedures and requirement for the complicated sample pretreatment.

Voltammetry has the advantages of simple, inexpensive, rapid, environmental-friendly, high sensitivity, good selectivity, and it's widely used for the detection and determination of various pharmaceutical compounds. There are many efforts have been made in electrochemical modification of the electrodes to the determine PA such as [4,15-18]. To the best of our knowledge, there is no report based on using ZnONPs-Nq/GCME for the determination of paracetamol.

In this paper, we are developing a new modified glassy carbon electrode with ZnO nanoparticales (ZnONPs) mechanically followed electropolymerization formed a polymer film of 1, 2-napthaquinone-4-sulphonic acid (Nq) scheme 1. Finally, use this modified electrode (ZnO NPs-Nq/GCME) to determine paracetamol in pharmaceutical forms in 0.1 M PBS.

2. EXPERIMENTAL

2.1. Reagents and Materials

Stock solutions of 0.0132 M paracetamol (Abimol tablets 500 mg from local market) were prepared by weighed and powdered one tablet and dissolved in doubly distilled water and filtered. 1, 2-napthaquinone-4-sulphonic acid (Sigma Aldrich) solutions were prepared by dissolved 0.44 gm in doubly distilled water and used without any further purification. Phosphate buffer solutions were prepared using 0.1 M NaH_2PO_4 - Na_2HPO_4 and by adjusting the pH with 1 M H_2SO_4 and 1 M NaOH. ZnO nanoparticales were prepared in our lab by precipitation method [19]. All aqueous solutions were prepared in doubly distilled water at room temperature and used analytical grade chemicals.

2.2. Apparatus and Cell

A computer-aided electrochemistry system was used in the voltammetric studies. The apparatus used in these experiments is a potentiostat model 263 (EG δ G PARC) Princeton applied research corporation (made in USA). The cell consists of three electrodes (MODEL K0264 MICRO-CELL), the counter (model K0266) electrodes were of high purity platinum wire, while an Ag/AgCl saturated KCl (model K0265) electrode was used as the reference electrode and a glassy carbon electrode (1 millimeter in diameter) was using as working electrode. The pH-meter was used for adjusting pH values.

2.3. Procedures

For all the cyclic and linear sweep voltammetric the supporting electrolyte was degassed in a 25 ml cell. Then the voltammogram was recorded after applying the required potential for a period of time at the definite scan rate. The analyte under investigation was added to a

suitable PBS in a definite concentration and the peak response was characterized with respect to concentration, pH and scan rate. All the experiments were carried out at room temperature and triplicates.

2.4. Preparation of ZnONPs-Nq/GCME

A glassy carbon electrode (1 mm diameter) was polished to a mirror finished with polish paper, Al₂O₃ slurry 0.05 μ m on a polishing cloth and alumina suspension solution, followed by sonication in nitric acid, acetone and redistilled water. Then, the GCE was electrochemically cleaned by cycling the electrodes potential between -1.0 to 1.0 V in 1.0 M H₂SO₄ [20], until a stable voltammogram was obtained. After it was washed with redistilled water and dried in air, the modification carried in two steps: 1- Mechanically through abrasive immobilization [21]: the solid compound zinc oxide nanoparticles was transferred to the surface of glassy carbon electrode as follows: Sample amounts of 1-3 mg of nanoparticles ZnO were placed on a coarse grade filter paper. The glassy carbon electrode was pressed onto the substance and rubbed over the material, causing some compound to adhere to the electrode surface. 2- Electropolymerization: the ZnONPs/GC modified electrode was immersed in 0.1 M Na₂HPO₄ buffer solution containing 3.33 mM of Nq and was conditioned by cyclic sweeping between for 50 cycles. Finally, the modified electrode was carefully rinsed with distilled water to remove the physically adsorbed material and was transferred to an electrochemical cell.

2.5. Sample preparation

Linear sweep voltammetry was applied to the analysis of two different pharmaceutical PA tablets (Abimol 500 mg and Adol 500 mg). Each one was weighed, ground into powder and dissolved in 250 mL doubly distilled water. Then filtered through filter paper and diluted to 10 ml of standard solution diluted to 100 mL with 0.1 M PBS.

3. RESULTS AND DISCUSSION

3.1. Electrochemical behavior of the Nq film on ZnONPs/GCME

The electropolymerization of 1, 2-napthaquinone-4-sulphonic acid on a glassy carbon electrode modified with ZnO nanoparticales was carried out using cyclic voltammetry. Fig. 1 shows cyclic voltammetry of 0.1 M PBS containing 3.33 mM of Nq at scan rate 100 mV/s. as can be seen from this figure (curve a) an anodic peak at -0.104 V and a corresponding cathodic peak at -0.182 V (*vs.* Ag/AgCl) belong to the bare glassy carbon electrode immersed on phosphate buffer solution. At the same condition, from curve b an anodic peak at -0.263 V and a corresponding cathodic peak at -0.389 V with peak separation potential $\Delta E_p=126$ mV (*vs.* Ag/AgCl) was observed in the first scan. This can be attributed to the molecules of Nq

which attached on the surface of ZnONPs/GC modified electrode. The electrochemical behavior of Nq at ZnONPs/GCME was a quasi-reversible similar to the references reported [22,23].

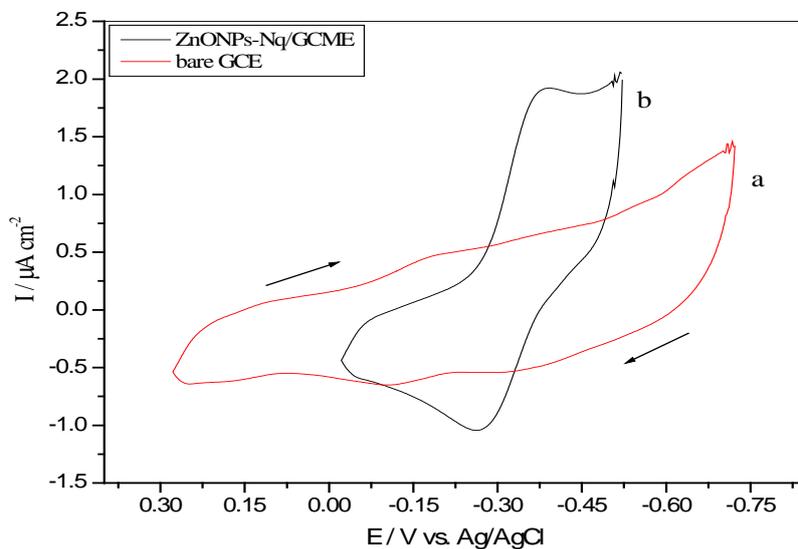


Fig. 1. CVs of 0.1 M PBS at bare GCE (a) and at ZnONPs-Nq/GCME (b) in scan rate 100 mV/s

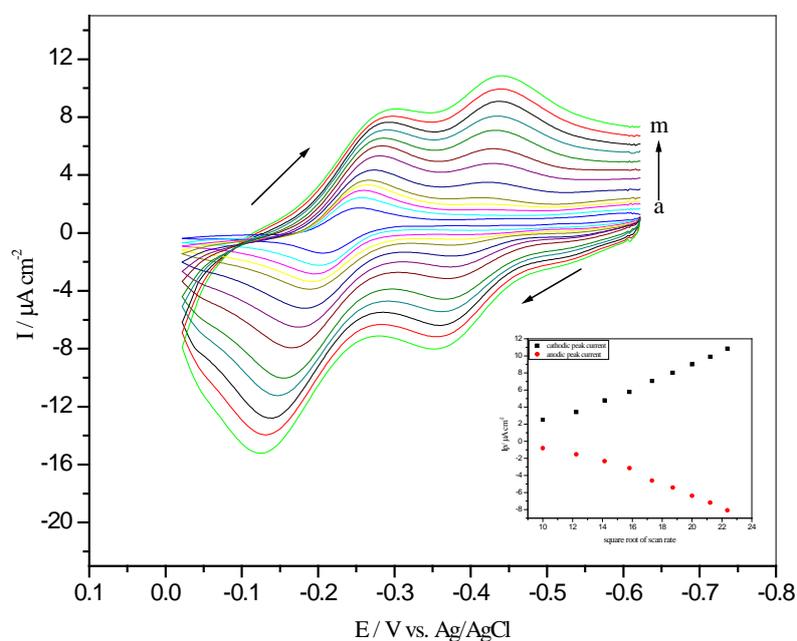


Fig. 2. CVs of ZnONPs-Nq/GC modified electrode in 0.1 M PBS contains 3.33 mM Nq at different scan rate: (a) 20, (b) 40, (c) 60, (d) 80, (e) 100, (f) 150, (g) 200, (h) 250, (i) 300, (j) 350, (k) 400, (l) 450 and (m) 500 mV/s. Inset shows the corresponding I_{pa} and I_{pc} vs. $\nu^{0.5}$

The effect of scan rates on the response of the ZnONPs-Nq/GCME shows in figure 2. Where, both the anodic and cathodic peak currents were increasing with increase in scan rate (20-500 mV/s) suggesting a surface-controlled process [22,23]. At scan rate higher than 100 mV/s appear anodic peak at -0.374 V and cathodic peak at -0.41 V vs. Ag/AgCl. These peaks current is directly proportional to square root of scan rate from 100 - 500 mV/s (Inset figure 2) which enhanced the adsorption of Nq molecules on the surface of ZnONPs/GCE under diffusion control process [24].

3.2. Electrochemical behaviour of PA

The modified electrode was used to investigate the electrochemical behaviour of PA in 0.1 M PBS (pH 7.6) at scan rate 100 mV/s as shown in Figure 3. The electrochemical behaviour of PA at the bare glassy carbon electrode (Fig. 3, curve a) exhibited one anodic peak potential at 0.635 V and one cathodic peak potential at 0.449 V (vs. Ag/AgCl) with $\Delta E_p=186$ mV. For the ZnONPs/GC modified electrode (Fig. 3, curve b) appear both anodic and cathodic peaks potential at 0.419, 0.276 V (vs. Ag/AgCl) respectively with $\Delta E_p=143$ mV. But, for the ZnONPs-Nq/GC modified electrode (Fig. 3, curve c) appear at 0.207, 0.078 V (vs. Ag/AgCl) respectively with $\Delta E_p=129$ mV. As can be observed, a substantial negative shift of the oxidation potential for PA indicated the catalytic ability of ZnONPs-Nq/GCME to PA oxidation due to overpotential decreased 0.428 V.

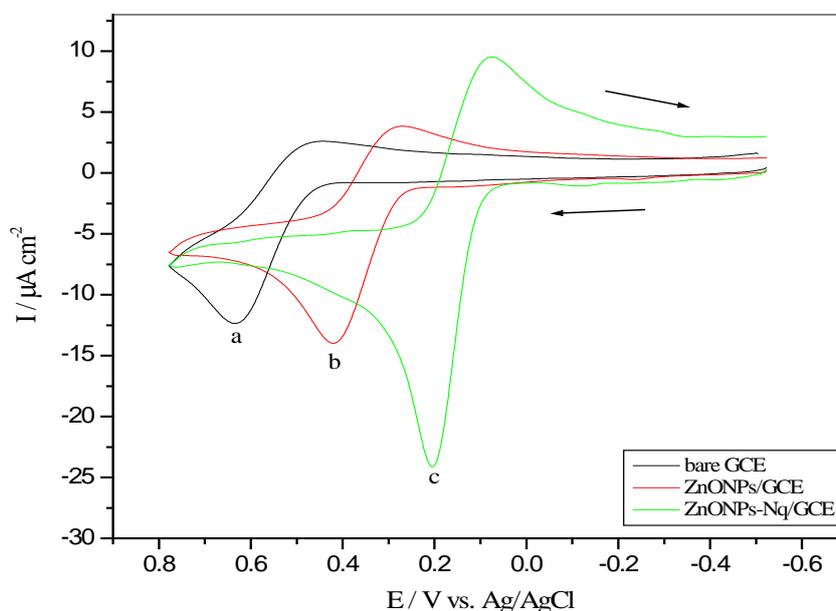


Fig. 3. CVs of 4.29 mM of PA in 0.1 M PBS on bare glassy carbon electrode (a), ZnONPs/GC modified electrode (b) and ZnONPs-Nq/GC modified electrode at scan rate 100 mV/s

In addition to, the ZnONPs/GC modified electrode (Fig. 3, curve b) enhanced the current response for PA by 1.6 folds compared to the bare GCE (Fig. 3, curve a). The addition of Nq to the modified electrode (Fig. 3, curve c) caused a 3.5 folds increase in the anodic peak current which indicate the electrocatalytic activity of ZnONPs-Nq/GCME towards the oxidation of PA.

3.3. Effect of pH

The electrochemical behavior of PA strongly affected by the variation of pH, due to the presence of non-bonding electron pairs located on $-O$, $-NH-$, and $-COCH_3$ functional groups. Figure 4 shows cyclic voltammograms of 2.2 mM PA on ZnONPs-Nq/GCME for pH range (2.4-13.0) at scan rate 100 mV/s. In acidic medium the anodic peak current has high values, but cathodic peak current has low ones due to the participation of proton(s) in the oxidation reaction of PA. In basic medium there are poor values for anodic and cathodic peaks currents because PA is converted to the phenoxide ion [4]. The suitable of anodic and cathodic peaks currents were obtained at pH 7.6 suggested it as optimal pH value.

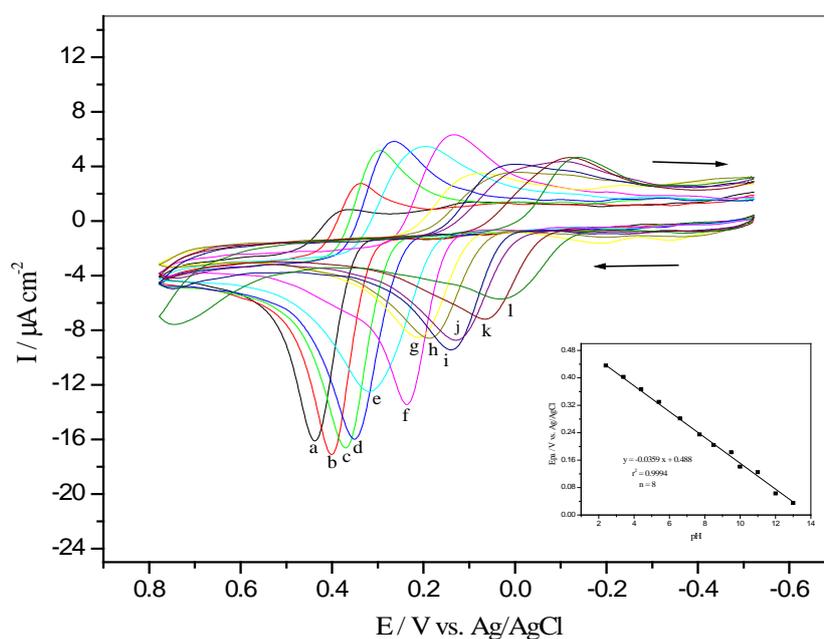


Fig. 4. CVS of 2.2 mM of PA on ZnONPs-Nq/GC modified electrode at scan rate 100 mV/s in 0.1 M PBS with different pH: (a) 2.4, (b) 3.4, (c) 4.4, (d) 5.4, (e) 6.6, (f) 7.6, (g) 8.5, (h) 9.5, (i) 10, (j) 11, (k) 12 and (l) 13. Inset shows the corresponding pH vs. E_{pa}

Each oxidation and reduction peaks potentials shifted to the negative potential with increase of pH as shown in figure 4. A linear relationship was obtained (inset figure 4)

between the anodic peak potential of PA and pH of PBS over the pH range (2.4-13.0) with a linear regression equation $E_{pa} = -0.035\text{pH} + 0.49$ (correlation coefficient $r^2 = 0.9994$). This value of slope 0.035 indicates the electrochemical reactions of PA on ZnONPs-Nq/GC modified electrode are two-electron and one-proton process [25]. This means the transformation of paracetamol to N-acetyl-pquinoneimine and vice versa is a quasi-reversible with participation of proton.

3.4. Effect of scan rate

Scan rate effect for 2.2 mM of PA in 0.1 M PBS (pH 7.6) performed by increase in the scan rate from 20 to 1000 mV/s at ZnONPs-Nq/GC modified electrode. As can be seen from figure 5 there is increase in each anodic and cathodic peak current with increasing in scan rate. On the other hand, the oxidation peaks potentials shifted positively with the increase of scan rate, indicating the quasi-reversible electrochemical reaction process [4]. Inset figure 5 a linear relationship between $\log I_{pa}$ and $\log \nu$ was found for PA ranging from 20 to 1000 mV/s ($I_{pa} = 0.6 \log \nu - 0.23$ with correlation coefficient $r^2 = 0.9997$). A slope of 0.6 with close to the theoretical slope of 0.5 for diffusion controlled process [26].

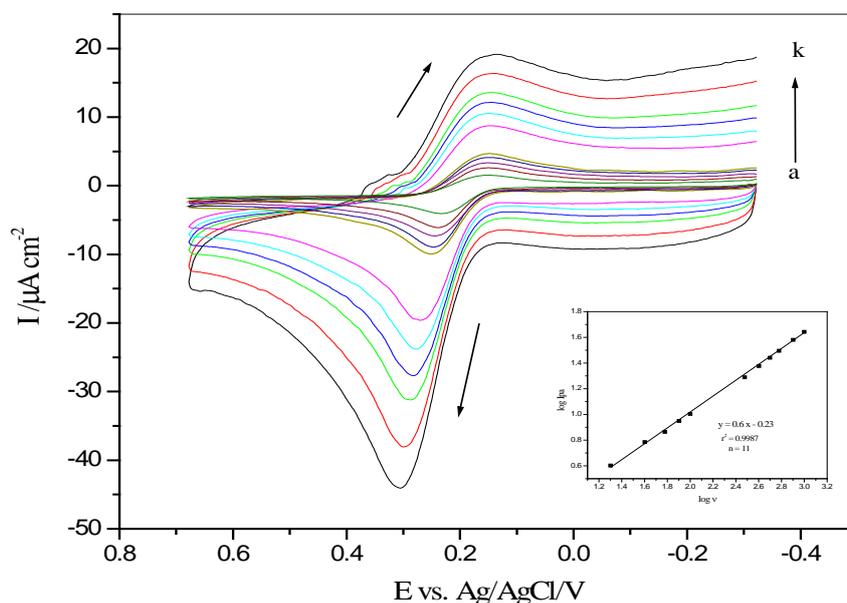


Fig. 5. CVs of 2.2 mM of PA in 0.1 M PBS on ZnONPs-Nq/GC modified electrode at different scan rate: (a) 20, (b) 40, (c) 60, (d) 80, (e) 100, (f) 300, (g) 400, (h) 500, (i) 600, (j) 800 and (k) 1000 mV/s. inset shows the corresponding $\log I_{pa}$ vs. $\log \nu$

The value of diffusion coefficient (D) can be obtained from the slope of anodic peak current against square root of scan rate as figure 6A ($I_{pa}=1.73 v^{0.5}-9.24$ with correlation coefficient $r^2=0.9987$) and using the formula of Randles–Sevcik [24]:

$$I_{pa}= 0.4463nFAC (nFDv/RT)^{1/2}$$

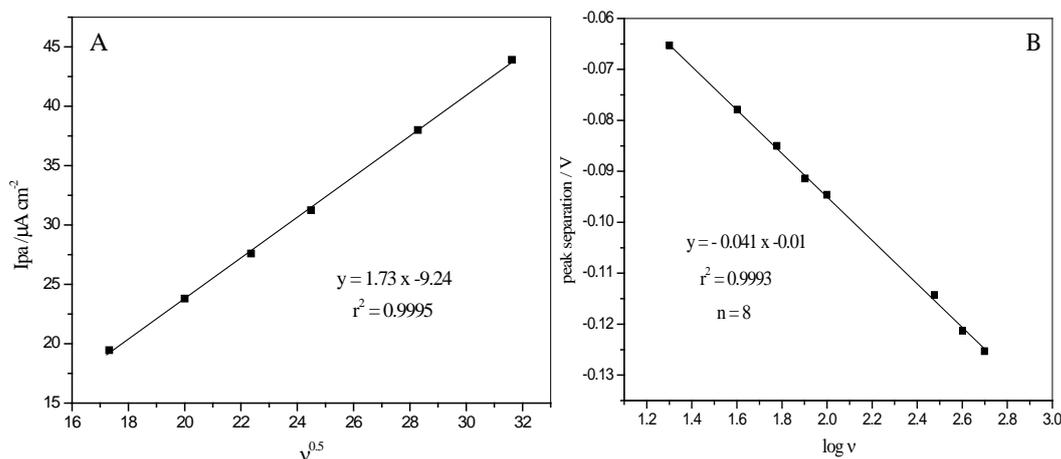


Fig. 6. Plot of I_{pa} vs. square root of scan rate (A) and plot of ΔE_p vs. log scan rate (B)

Where n is the number of electrons transferred (2), F is the Faraday constant ($96,485 \text{ C mol}^{-1}$), A is the surface area of working electrode (cm^2) and C is the concentration of PA (2.2 mM), D is the diffusion coefficient, R is the gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$), T is the absolute temperature (298 K), the value of D for PA on modified electrode was found to be $5.998 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$.

According to the Laviron theory [27] the electron transfer rate constant (k_s) and charge transfer coefficient (α) can be determined by plotting the variation of peak potential against $\log v$ (figure 6B) with linear regression equations $\Delta E_p = -0.041 \log v - 0.01$ and correlation coefficient $r^2 = 0.9993$. Using the equation of:

$$\Delta E_p = K - 2.3030 (RT/\alpha nF) \log v$$

α value was found to be 0.721 and compensation this value in equation:

$$\log k_s = \alpha \log (1 - \alpha) + (1 - \alpha) \log \alpha - \log (RT/nFv) - \alpha (1 - \alpha) (nFE/2.3RT)$$

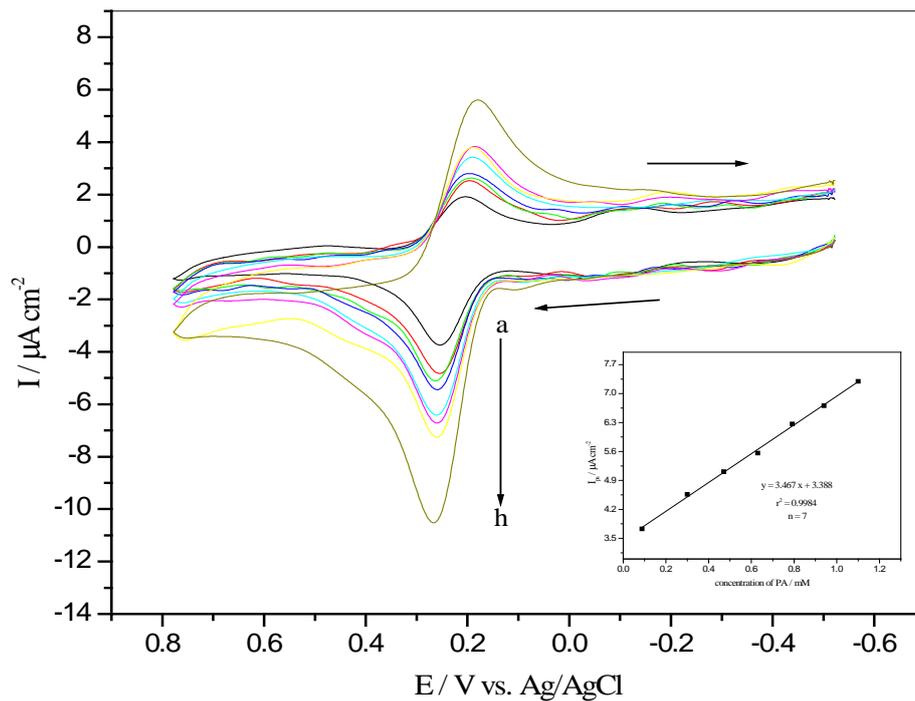


Fig. 7. CVs of PA in 0.1 M of PBS (pH 7.6) on ZnONPs-Nq/GCME at scan rate 100 mV/s with different concentration: (a) 0.088, (b) 0.3, (c) 0.471, (d) 0.63, (e) 0.792, (f) 0.94, (g) 1.1 and (h) 2.2 mM. Inset shows the corresponding calibration curve.

A surface electron transfer rate constant, $k_s = 11.279 \text{ s}^{-1}$, was obtained. This large value of electron transfer rate constant indicates the high ability of ZnONPs-Nq/GCME for promoting electrons between PA and the electrode surface.

3.5. Effect of concentration of PA

The effect of concentration of PA on ZnONPs-Nq/GC modified electrode has been studied in 0.1 M PBS (pH 7.6) at scan rate 100 mV/s as can be seen in figure 7. Where each anodic and cathodic peak current increase with increasing in concentration (8.8×10^{-5} – 2.2×10^{-3} M).

Table 1 shows the data used to calculate the value of standard deviation of the mean current (SD) from the following equation [28]:

$$SD = 1 / (n-2) \sum (i_j - I_j)^2$$

Here, i_j is the experimental value of the experiment number J and I_j is the corresponding recalculated value, at the same concentration using the regression line equation $I_{pa} = 3.467$

$C+3.388$ ($r^2=0.9984$) which obtained from plotting anodic peak current versus concentration of PA (inset figure 7).

Table 1. The data used to calculate the value of SD taken from calibration curve

X	i_p	I_p
0.088	3.727	3.693096
0.3	4.56	4.4281
0.471	5.11	5.020957
0.63	5.56	5.57221
0.792	6.265	6.133864
0.94	6.706	6.64698
1.1	6.294	7.2017

The calculated SD was used in the determination of the detection limit (DL, $3 \times SD/\text{slope}$) and the quantification limit (QL, $10 \times SD/\text{slope}$). From these values, the detection and quantification limits were, respectively, 30.03×10^{-6} and 10.01×10^{-5} M. A good sensitivity response of $3.467 \mu\text{A}/\text{mM}$ was obtained. The detection limit of ZnONPs-Nq/GCME for determination of PA were compared with previously published techniques in Table 2.

Table 2. Comparison of ZnONPs-Nq/GCME with other glassy carbon modified electrodes reported

Modified electrode	Detection limit (μM)	techniques	Ref.
Poly(caffeic acid)/GCE	0.026	SWV	[4]
NanoTiO ₂ /poly(AY)/GCE	2.00	CV	[29]
PEDOT/GCE	1.13	DPV	[30]
CF/GCE	0.036	CV	[30]
Fullerene /GCE	50.0	DPV	[31]
Nafion/TiO ₂ -Graphene/GCE	0.21	CV	[32]
ZnONPs-Nq/GCE	30.03	CV	Present work

3.6. Interference study

One of the most important advantages of modified electrode is having good selectivity. To test the selectivity of ZnONPs-Nq/GCME upon the determination of PA using linear sweep voltammetry, some interferents were investigated by the addition of various

interference species to 0.1 M PBS in the presence of 80.0 μM of PA. The results indicate that the common ions such as Na^+ , K^+ , NO_3^- , SO_4^- and Cl^- did not show interference with PA detection. Citric acid, tartaric acid and glucose have no obviously interference in PA determination as summarized in table 3.

Table 3. Interference of some foreign substances for 80.0 μM of PA

Interferents	Folds
Na^+ , K^+ , NO_3^- , SO_4^- , Cl^-	1000
Citric acid, tartaric acid	500
Glucose	500

3.7. Stability and reproducibility

Through two weeks the stability of ZnONPs-Nq/GCME toward the oxidation of PA was investigated. Where there is no significant shift observed for oxidation and reduction peaks potential of PA when kept the modified electrode in 0.1 M PBS at room temperature. In addition to, the current signals showed less than a 2.0% decrease relative to the initial current of oxidation peaks of PA. The reproducibility of ZnONPs-Nq/GCME toward the oxidation of PA was also tested. The reproducibility of the modified electrode was evaluated by measuring on seven solutions of 80.0 μM of PA in PBS leading to a relative standard deviation of 3.73%. These results indicated that this modified electrode has high stability and good reproducibility.

3.8. Applications for the determination PA in commercial drug samples

The ZnONPs-Nq/GC modified electrode was applied to determination of PA in two pharmaceutical drugs samples by standard addition method. I_{pa} was measured at the oxidation potential of PA by linear sweep voltammetry. For the analysis, 10 mL of the sample solution was diluted to 100 mL with 0.1 M PBS (pH 7.6) and the results are summarized in Table 4.

The PA recovery in the pharmaceutical drugs samples was in the range of (94.44-106.849) which indicate that the ZnONPs-Nq/GC modified electrode can be readily used to determine of PA in commercial tablets samples.

Table 4. Recovery of PA in 0.1 M PBS (pH 7.6) spiked with different paracetamol concentrations using an ZnONPs-Nq/GC modified electrode (n=4)

Commercial drugs samples	Original content (mg)	PA added(mg)	PA founded (mg)	Recovery (%)
Abimol tablet	500	-	499	99.8
		100	577	96.17
		200	681	97.28
Adol tablet	500	-	532.47	106.49
		100	580	96.67
		200	661.11	94.44

4. CONCLUSION

In this paper, a GCE modified with ZnO nanoparticales and a polymer film of 1, 2-naphthaquinone-4-sulphonic acid has been successfully developed to investigate PA in its commercial tablets at pH 7.6. The modified electrode has great capability in determine PA relative to bare GCE. Each anodic and cathodic current peaks increase with increasing in scan rate and concentration of PA with detection and quantification limits 30.03×10^{-6} and 10.01×10^{-5} M respectively. The values of diffusion coefficient (D), electron transfer rate constant (k_s) and electron transfer coefficient (α) were calculated and found for the PA oxidation to be $5.998 \times 10^{-6} \text{ cm}^2 \text{ s}^{-1}$, 11.279 s^{-1} and 0.721 respectively. The high stability good selectivity and reproducibility makes the prepared modified electrode very useful in the detection of PA in commercial drugs samples.

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