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Detection of Quinine in Phospate Buffered 0,1 M by Using Boron-Doped Diamond Electrodes

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Abstract. This study investigates the electrochemical reduction of quinine (QN) detection using boron-doped diamond electrodes (BDD). Different pulse voltammetry (DPV) of QN in a 0.1 M PB solution exhibits reduction peaks at -0.86 V (vs. Ag/AgCl). Additionally, the effects of pH and scan rate were explored to investigate the reduction mechanism within a potential range of -1.4 V to -0.4 V (vs. Ag/AgCl). Furthermore, a linear calibration curve was observed in the concentration range of 2 μM to 25 μM (R^2=0.99) with a detection limit of 0.62 μM (S/B=3).

Introduction

Quinine (QN) is an alkaloid plant with effective medicinal use as an adjunctive therapy for immunodeficiency syndrome and neurodermatitis and has antipyretic, anti-inflammatory, analgesic, muscle relaxant and antimalarial properties. In excess, QN can cause gastrointestinal problems, cinchonism, visual disturbances, damage central nervous system, and cardiac dysrhythmias [1–3]. To prevent overdose, the level of QN in the body must be maintained. The therapeutic plasma concentration range of QN is 3–7 g mL^{-1}, and blood concentrations greater than 10 g mL-1 [4,5]. Between 1965 and 2006, FDA documented 665 cases of serious adverse reactions associated with the use of QN, including 93 fatalities. [6].

Due to its side effects and potential toxicity, several analytical methods have been reported to determine QN detection use in biological, pharmaceutical, and food samples. Methods include high-performance liquid chromatography [6,7], mass [8], chemiluminescence [9,10], gas chromatography mass spectrometry [11,12], and capillary electrophoresis [13]. However, these methods are costly, time-intensive, and utilize hazardous reagents. Moreover, some recent reports have indicated electrochemical methods for determining QN; which have proved to be more beneficial in terms of simplicity, rapid response, high sensitivity, low detection limits, the absence of expensive and complex devices, and the ease of integration with other systems, for example. The choice of working electrode materials for electrochemical sensors is crucial as it affects the cost, sensitivity, selectivity, and stability of these devices [14–16].

Based on the advantages of electrochemical analytical techniques described above, previous research has been conducted using pretreated graphite pencils [8], bentonite modified electrodes [17], and modified glassy carbon electrodes [2] [18]. It is proven that QN detection determination achieves good accuracy and precision compared with other detection methods[14], [19–21]. However, the use of modified working electrodes will increase cost and time in the modification process. Recently, the QN detection was carried out using Boron Doped Diamond (BDD) electrodes with Britton-Robinson buffer (BRB) as the supporting electrolyte and this electrode offers simple preparation and good sensitivity. Thus, BDD is one of the working electrodes widely used today for detection in electrochemical methods.

BDD is an effective working electrode used in electrochemical sensors and is suitable for use in dilute and solid electrolytes [22]. BDD offers several advantages in electrochemical sensing applications, such as a broad potential window, low background current, excellent chemical and mechanical stability, and strong resistance to impurities [23,24]. The use of BDD in electrochemical sensors includes as a method of detecting pollutants in water [25],[26], as a gas detection method [22,27], as a drug detection method [21] and for medical purposes. Therefore, based on the advantages of the present study, research will be carried out by replacing the working electrode using a BDD with 1% boron content.

In this study, QN detection was performed using BDD and phosphate buffer solution (PB) as support electrolytes. The pH of the electrolyte and the scan rate of the CV technique were systematically varied to explore the electrochemical behavior of QN. Then, the performance of the BDD is investigated by calculating the detection limit (LOD) on the basis of changes in the concentration of the analyte.

Experimental

Preparation of BDD Electrodes

The BDD is deposited on a thin BDD polycrystalline layer on a Si (100) wafer substrate through the microwave plasma-assisted chemical vapor deposition (MP-CVD) method for the MP-CVD method. Boron base material is trimethyl borate and carbon base material is acetone. Boron-carbon ratio was 1% with deposition carried out for 7 h at 5 kW [24,28].

Preparation of Reagents

Reagents solution was derived from 10 mM QN dissolved in 0.5% DMSO stock solution. First, the PB was prepared by mixing $NaH_2PO_4.2H_2O$ and $Na_2HPO_4.12H_2O$ in distilled water with 0.1 M concentration. Afterward, the stocks solution was diluted in PB 0.1 M at pH 7.4.

Electrochemical Measurement

The electrochemical cell was configured using a three-electrode system consisting of Ag/AgCl (3M KCl) electrode as the reference, BDD electrode as the counter, and another BDD electrode as the working electrode, with 0.025 mM QN in 0.1 M PB serving as the electrolyte. Initially, the electrochemical circuit is connected to the PGSTAT204, and the potential window is set at potential -1.4 V to -0.4 V (vs. Ag/AgCl). The BDD working electrode previously was washed in aqua regia solution containing a 3:1 combination of HCl and HNO 3 for 30 min. BDD soaking procedure cleans the surface of the BDD of metallic species contamination while improving its electrochemical performance [29], [30]. BDD electrode was thoroughly rinsed with distilled water for 15 min and subsequently dried using nitrogen gas. Then, BDD was chronoamperometry in 0.1M H_2SO_4 solution with a potential of +3 V (vs. Ag/AgCl) for 5 min to produce oxygen-terminated boron-doped diamond (O-BDD) and -3 V (vs. Ag/AgCl) for 10 min to form hydrogen-terminated boron-doped diamond (H-BDD) [31]. Data were then collected through Autolab/PGSTAT101 using cyclic voltammetry and differential pulse voltammetry.

Results and Discussion

Effect of Surface Termination on Electrochemical Properties

Surface termination type plays a crucial role in influencing the electrical and electrochemical properties of the BDD surface. Furthermore, the surface termination of BDD can affect significantly its electrochemical properties. [32,33]. The results show that the reduction peak current value at both terminations is different, where the reduction peak current at H-BDD (blue) is higher than in O-BDD (red) (see Fig. 3). This is influenced by the surface conductivity of H-BDD and increased sensitivity, while O-BDD shows lower electrochemical activity [28,34,35]. Based on these results, the surface termination of H-BDD is used in this study for the detection of QN.

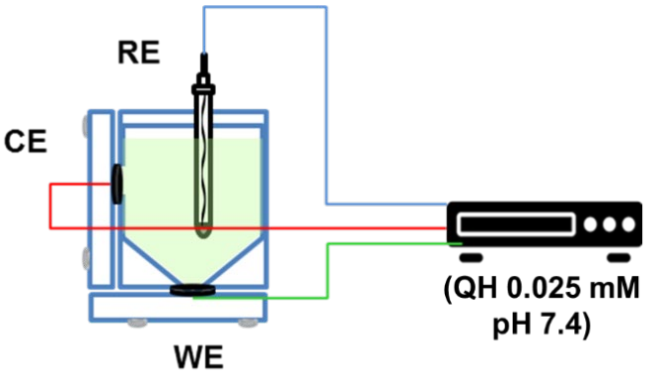

Fig. 1. Electrochemical Measurement Setup Scheme

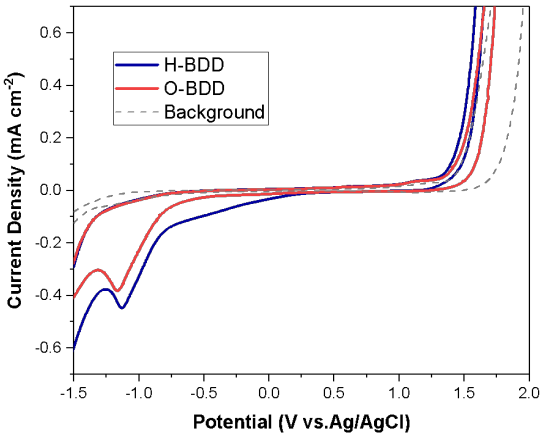

Fig. 2. Voltammogram of 25 µM QN in 0.1 M PB

Electrochemical Reduction of QN

CVs were recorded at 100 mV s^{-1}, in the potential range -0.4 V to -1.5 V (vs. Ag/AgCl). Based on the observation of QN electrochemical reduction by CV technique from -0.4 V to -1.5 V (vs Ag / AgCl) there is a peak current in the voltammogram in the range of -1.14 V (vs Ag / AgCl). This indicates a reduction reaction that occurs during the measurement by the CV method without any oxidation reaction seen throughout the potential range (Fig. 4A). It was also observed using the DPV method from the range of -1.00 V to -0.70 V (vs. Ag/AgCl), where it can be confirmed that there is a peak reduction current in the potential of -0.86 V (vs. Ag/AgCl). The reaction on the electrode surface can be analyzed from the relationship between current and potential on the electrode, where the DPV potential has shifted compared to CV (see Fig. 4B). This shift is due to the difference in concentration of QN, scan rate, and pH electrolyte [36].

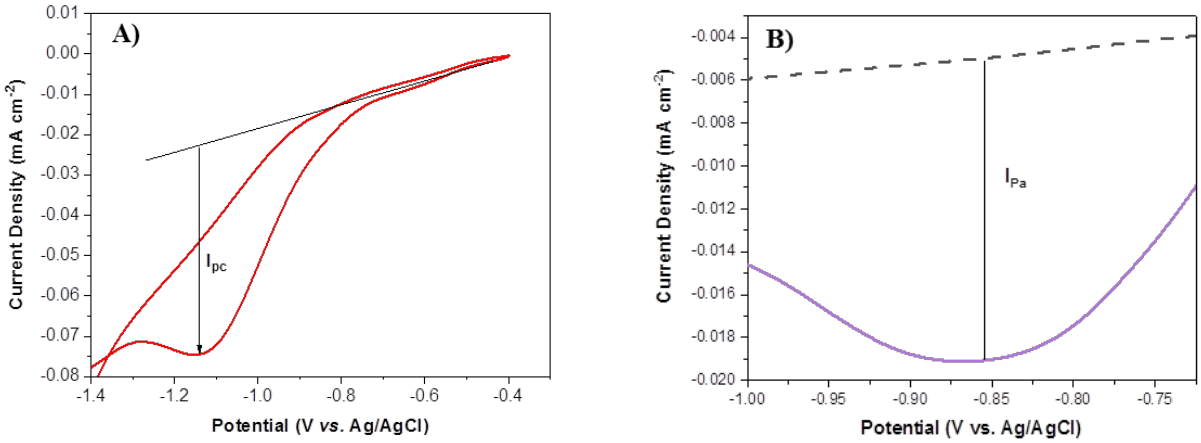

Fig. 3 Voltammogram QN **(A)** CV 25 µM QN at pH 7 in 0.1 M PB
(B) DPV 25 µM QN at pH 7 in 0.1 M PB

To study the reduction mechanism, CVs were conducted at various scan rates. The scan rate range of 40 to 100 mV s1 was employed within the potential window of 0.4 V to 1.4 V (vs. Ag/AgCl) using a QN concentration of 25 M (Fig. 4). The higher the scan rate, the higher the peak current appearing. This is due to the fact that as the scan rate increases, the thickness of the diffusion layer decreases and the concentration of electroactive substances on the electrode surface changes faster, which can result in an increase in current and a shift in peak potential. Thus, the maximum potential may shift to a more positive or negative value, depending on the electrochemical reaction and experimental conditions [37], [38]. This also indicates that the QN reaction occurs irreversibly on the surface of the BDD [18]. This phenomenon can also be described by the Randles-Sevcik equation [39]. The number of electrons involved can be calculated using the Randles-Sevcik equation for irreversible processes (1). The diffusion coefficient (D) is the slope value of the calibration curve of Fig. 5. D value of reduction reaction 4×10^{-5} derived by slope. The diffusion coefficient obtained from the results of the calculation findings was ($\mathbf{R^2}$ = 0.99). At the same time, the predicted value for the number of electrons involved (n) was about 2 electrons for the reduction.

$$n\,(\alpha\, n_\alpha)^{1/2} = (2.99\times10^5)\, Ip\, AD^{1/2}\, v^{1/2}\, CA \qquad (1)[39].$$

Where Ip is the maximum current (A), n is the number of electrons, α is the charge transfer coefficient, n_α is the number of electrons involved in the charge transfer step, (D) is the diffusion coefficient (cm^2s^{-2}) or the slope value at reduction reaction, A is the electrode surface area of the electrode (cm^2), v is the scan rate (Vs^{-1}), and C is the concentration in solution (mol cm^{-3}).

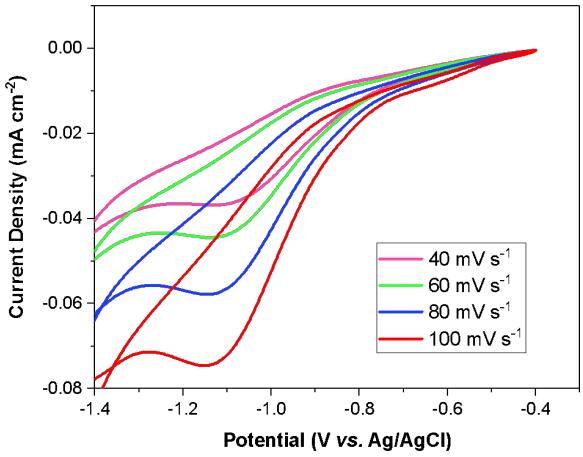

Fig. 4. Cyclic Voltammogram of QN with Variation of Scanning Rate at pH 7 in 0.1 M PB

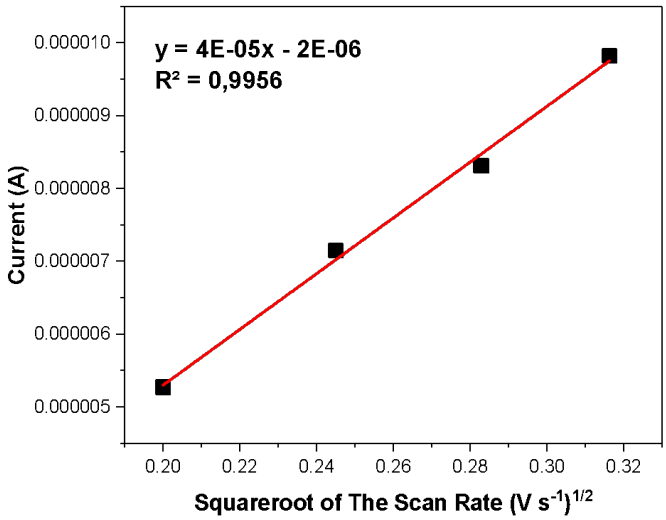

Fig. 5 Linear Curve of Current as a Function of Square Root of Scan Rate

Based on the voltammogram using the scan rate dependence, the plot of the relationship between the maximum current and the square root of the scan rate (Fig. 6) obtained results that are more linear than the maximum current with the scan rate with a slope of Ip (A) = 2 x 10^{-6} (R^2=0.99). It indicates that the reaction process on BDD is influenced by diffusion controlled [17], [19], [40]**.** Moreover, the number of electrons involved (n) in the reduction process is approximately 2 electrons [3]. The scheme of the reduction reaction is shown in Fig. 8.

Then, different pulse voltammetry (DPV) methods were used to determine the effect of the pH of the solution on the reaction mechanism and peak currents during the test. This study was carried out using pH dependence of three conditions including pH 5 (acid), 7 (neutral) and 9 (alkaline). Based on DPV voltammogram, it was obtained that the reduction peak shifted along the pH changing (see Fig. 7). The potential reduction peak is -0.91 V (vs. Ag/AgCl) at pH 9 and the potential reduction peak is -0.81 V (vs. Ag/AgCl) at pH 5. This result can be explained by the Nernst equation, where the lower the pH (the higher the concentration of H^+ ions), the more positive the potential value [41].

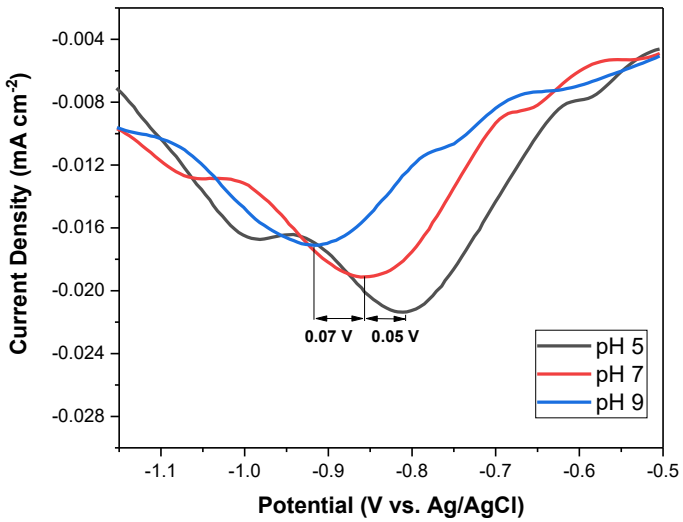

Fig. 6. Different Pulse Voltamogram of Potential Shift on pH Variation

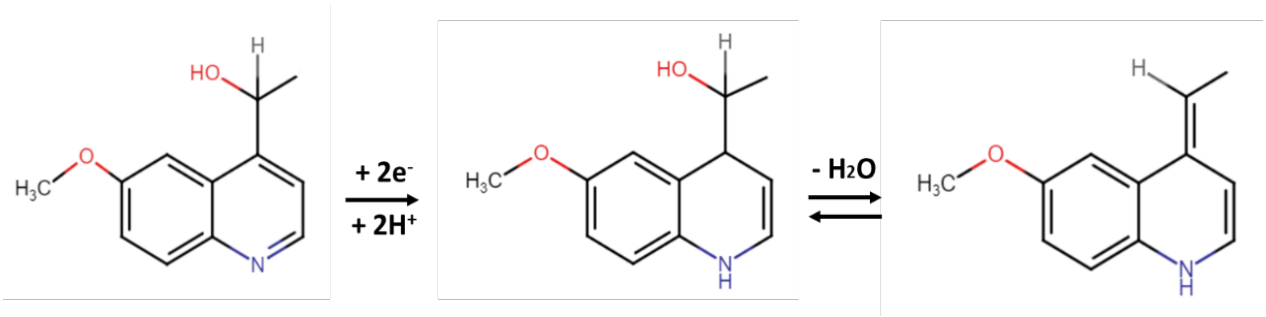

Fig. 7 Schematic of QN reduction reaction [40]

Limit of Detection (LOD) Determination

Determination of the detection limit using a variation of the QN concentration value in PB solution using DPV technique in the potential range of -1.00 V to -0.70 V (vs. Ag/AgCl). Based on Fig. 9A, the higher concentration shows the higher current density. The curves have good linearity (R^2= 0.99) in the concentration range of 2 – 25 µM as shown in Fig. 9B. By calculating using the equation of LOD (2) below:

$$\text{LOD} = \frac{\text{3.3 x STD of three blank samples}}{\text{slope of callibration curve}} \quad (2)[42].$$

The LOD calculation results obtained a detection limit value of 0.62 µM. Therefore, the BDD detection method is used to detect QN, whose maximum concentration in the human blood is 25 µM.

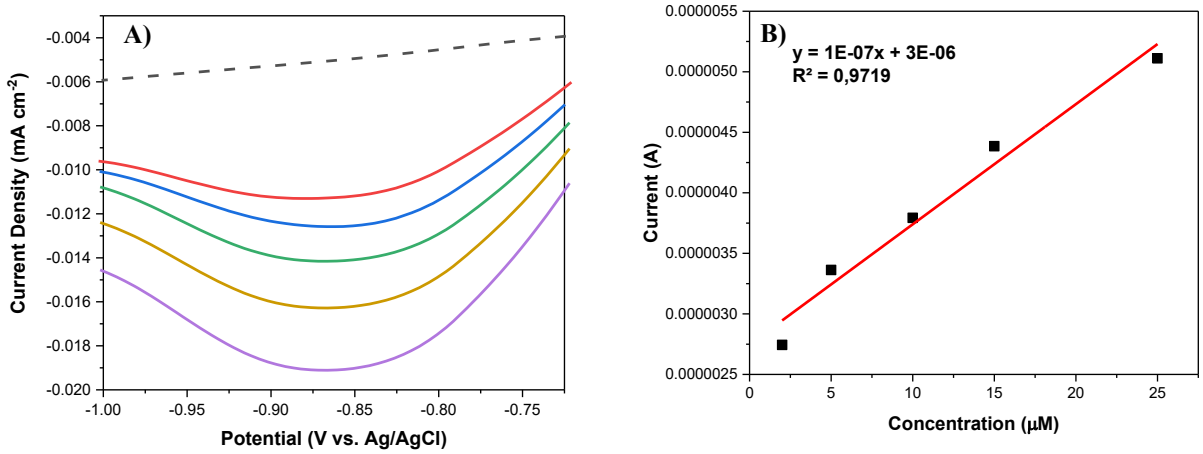

Fig. 8. (**A**) Different Pulse Voltammogram on Concentration Variation (**B**) Linear regression curve plot of peak current against concentration variation

Furthermore, the stability of the BDD was investigated by measuring the peak current 10 times. As shown in Fig. 10, constant currents were obtained on BDD with a 0.000269 deviation standard and an average of 0.86031 $mAcm^{-2}$. The graph indicates that consistent measurement values are acquired.

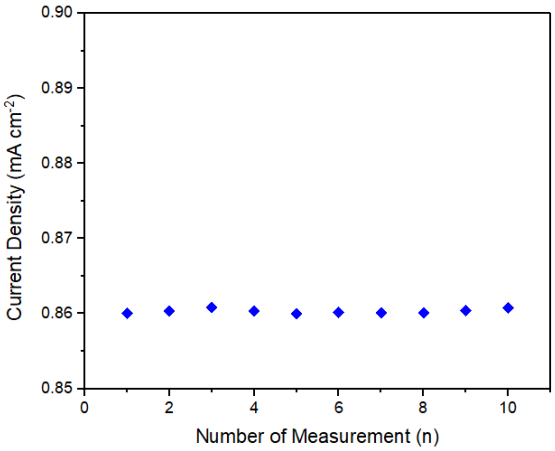

Fig. 9. Peak current of QN 25 µM QN on BDD electrodes versus number of measurements

Conclusion

This work reports the electrochemical behavior of QN detection using BDD. The BDD has been studied for detection of low QN concentrations. The advantages of the BDD electrode are as follows: (1) the detection target of the reduction reaction on the BDD is observed, (2) the reaction mechanism shows that the number of electrons involved in the reduction process (n) is approximately two electrons, and (3) the detection limit is low, and can't be achieved with other electrodes. These results suggest that BDD is potentially can be used as QN sensor.

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