

Electrochemical Oxidation of Ranitidine using a Boron-Doped Diamond Electrode in the Presence of Anionic Surfactant: A Comprehensive Investigation

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ABSTRACT

Ranitidine (RAN) is a drug from the histamine H₂ receptor antagonist class and is used to prevent excessive production of stomach acid. An electrochemical investigation of the RAN in pharmaceutical preparation and spiked human urine was performed for the using a boron-doped diamond electrode (BDDE). Voltammetric measurements were performed in a pH 11 BR solution supplemented with the anionic surfactant, sodium dodecyl sulfate (SDS). In the proposed method using optimized experimental conditions, linearity was obtained for RAN in the concentration range of 0.8-50.0 μ M. The LOD value obtained is 0.22 μ M. Good selectivity, accuracy, precision, and acceptable repeatability were also achieved in this proposed electrochemical sensor. Finally, this electrochemical sensor was successfully used for RAN detection in pharmaceutical samples.

Keywords: Ranitidine, Electrochemical detection, Boron-doped diamond electrode, Surfactant, Pharmaceutical samples.

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Introduction

RAN, (N-(2-((5-((dimethylamino) methyl) furan-2-yl) methylthio) ethyl)-N'-methyl-2-nitroethene-1,1-diamine) is an H₂ receptor antagonist and is often used to treat peptic ulcers by reducing stomach acid production. It is used in the treatment of RAN, duodenal and gastric ulceration, gastro-esophageal reflux disease, Zollinger-Ellison syndrome diseases [1,2]. The majority of the oral or intravenous RAN dose excreted in urine is in the unchanged drug form, but only a small fraction of the administered dose is excreted as metabolites (RAN-N-oxide, RAN-S-oxide, and desmethyl-RAN) [3,4]

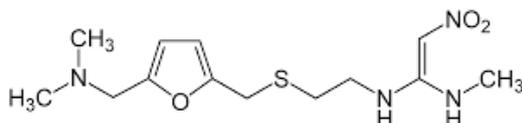


Figure 1. Chemical structure of RAN.

In the literature survey, there are various analytical methods developed for the quantitative analysis of RAN, such as high-performance liquid chromatography (HPLC), liquid chromatography (LC)-mass spectroscopy (MS), fluorescence technique, colorimetric detection, and electrophoresis [5-15]. Although these methods are effective and sensitive, they are known to be quite expensive and not environmentally friendly due to the excessive use of organic solvents. In addition, it creates disadvantages such as long analysis times and the need for experts to use analytical devices. Electrochemical methods, on the other hand, have a more

environmentally friendly feature than other methods and are preferred in the analysis of electro-active compounds used in many areas (such as agriculture, food, plants, health, and drugs) [16-20]. Voltammetry, one of the most used electroanalytical techniques, is widely used especially in drug analysis. These methods have many advantages such as short analysis time, low cost, low use of organic solvents, high sensitivity, precise analytical capabilities, and analysis in low volumes.

Boron-doped diamond electrode (BDD) is a special electrode used in chemical analysis and electrochemical applications. Boron-doped diamond electrodes are widely used in electrochemical analysis, sensor applications, bioanalytical measurements, and various electrochemical research. The properties of boron-doped diamond electrodes combine high mechanical strength, chemical inertness, low surface roughness, and conductivity [21-22]. These electrodes have the potential to provide high sensitivity and durability, especially in harsh environmental conditions and when working with biological samples.

The combination of carbon-based electrodes with surfactants is a widely used strategy in electrochemical studies to improve surface properties and increase measurement sensitivity. As is known, carbon-based electrodes generally have a high surface area, and their surface properties can be further improved with the use of surfactants. This allows reactants on the electrode surface to be adsorbed more effectively and electrochemical reactions to be monitored more precisely. Surfactants have the ability to control molecular adsorption on the electrode surface. This can particularly

increase the selectivity of certain analytes and reduce undesirable interactions on the electrode [23-25].

In this study, it was aimed to obtain a more stable sensor in the anionic surfactant environment with a boron-doped diamond electrode for sensitive voltammetric detection of RAN and to examine the possible electrochemical oxidation mechanism.

Materials and Methods

RAN ($C_{13}H_{22}N_4O_3S \cdot HCl$) standard pharmaceutical active ingredient was purchased from Sigma-Aldrich (Türkiye, CAS: 66357-59-3). 1.0×10^{-3} M stock solution was prepared by dissolving the appropriate amount of RAN in salt form in water. A stock solution of the surfactant tested in this study, sodium dodecyl sulfate, SDS (anionic) (1×10^{-2} M), was prepared by dissolving it in water. 0.1 M Acetate buffer (pH 4.8), 0.1 M phosphate buffer (pH 2.5, 7.4), 0.1 M Britton-Robinson buffer (pH 2.0 – 12.0), and H_2SO_4 (96%) (0.1 and 0.2 M) were used as supporting electrolytes. Additionally, dopamine, ascorbic acid, uric acid, lactose, glucose, potassium chloride, magnesium chloride, sodium sulfate, and potassium nitrate were obtained from Sigma-Aldrich to perform interference studies. The chemicals H_3PO_4 (85%), $NaH_2PO_4 \cdot H_2O$, CH_3COOH (100%), HCl (37%), H_3BO_3 (99.5%), and Na_2HPO_4 used in the preparation of supporting electrolyte solutions were obtained from Sigma-Aldrich.

Electrochemical studies were carried out with cyclic voltammetry (CV), and square wave voltammetry (SWV) techniques using Autolab PGSTAT 101 (Metrohm Autolab B.V., Netherlands) electrochemical analyzer with NOVA 2.1.6 electrochemical software.

Electrochemical studies were carried out using a triple electrochemical cell system containing a working electrode, counter electrode, and reference electrode. The BDD electrode as the working electrode (Windsor Scientific Ltd., UK, declared boron doping level of 1000 ppm, disk diameter of 3 mm); the platinum wire (MW-1032, USA) obtained from BASi was used as the counter electrode and $Ag/AgCl$ electrode (3 M NaCl; BASi, MF 2056, USA) was used as the reference electrode. In addition, solid chemical substances were weighed with a Vibra brand electronic scale with a sensitivity of 0.01 mg. ISOLAB model ultrasonic bath was used to clean the working electrode and dissolve some substances. WTW inoLab pH7110 digital pH meter was used to adjust the pH of the solutions.

Before starting the voltammetric experiments, BDD was pretreated cathodic and anodic in 0.5 M H_2SO_4 by applying -1.8 V, then +1.8 V for 120 s, respectively, to activate the electrode. Between individual measurements, the BDD electrode was gently cleaned with a damp velvet cloth and then rinsed with water.

Zantac® (contains 50mg 10mL-1 RAN HCl) brand syrup was used for RAN syrup formulations. The contents of the bottle were mixed thoroughly to ensure homogeneity. The required volume for voltammetric analysis was taken

and diluted with buffer containing anionic surfactant (SDS). The prepared solutions were analyzed directly voltammetrically without being subjected to any further processing.

Results and Discussion

Electrochemical Behavior of RAN

First, the CV technique was used to investigate the electrochemical response of 1×10^{-4} M RAN on BDD in pH 11 BR buffer solution (the best medium for RAN analysis has been selected and will be mentioned later) is presented in Fig. 2. As seen in the voltammogram, oxidation peaks of RAN were obtained at 0.75 μA at 1.28 V in pH 11 BR buffer solution, and no reduction peak was observed in the cathodic scan. This result shows that the oxidation of the RAN molecule under all applied conditions is completely irreversible.

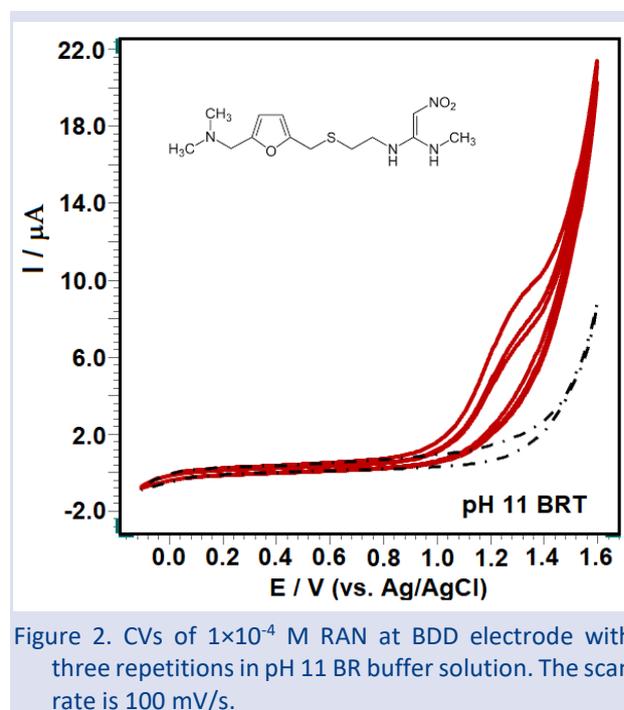


Figure 2. CVs of 1×10^{-4} M RAN at BDD electrode with three repetitions in pH 11 BR buffer solution. The scan rate is 100 mV/s.

Valuable information regarding the electrode reaction mechanism (rate determination step) can be obtained from the relationship between oxidation peak current and scan rate. The effect of scan rate on the electrochemical oxidation of 1×10^{-4} M RAN was studied using different scan rates (75-500 mV/s; n:6) with BDD electrode in pH 11 BR buffer solution and the corresponding voltammograms are shown in Fig. 3. $v - I_p$ (Eq.1), $\log v - \log I_p$ (Eq.2) graphs were drawn using the findings obtained from these voltammograms, and the linearity equations of these graphs are presented below.

$$\log I_p = 0.7809 \pm 0.16 \log v - 1.6105 \pm 0.562; r = 0.999 \quad (1)$$

$$\log I_p = 0.7809 \pm 0.16 \log v - 1.6105 \pm 0.562; r = 0.999 \quad (2)$$

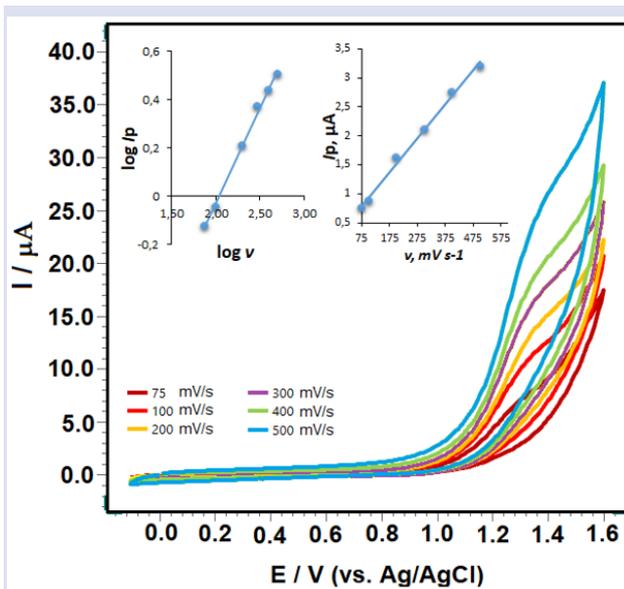


Figure 3. Cyclic voltammograms of 1×10^{-4} M RAN recorded using a BDD electrode in the scan rate range of $75\text{--}500\text{ mV s}^{-1}$ in pH 11 BR buffer solution. Inset: Linearity graphs of $v\nu - I_p$ and $\log v - \log I_p$.

The linearity obtained from the I_p/v relationship shows that the electrochemical oxidation of the RAN molecule is adsorption-controlled under the experimental conditions studied. When the logarithmic equations of current and scan rate are examined, the slope obtained (0.78) is between the theoretical values of 0.5 and 1.0, indicating that this reaction is adsorption-controlled in the irreversible electro-oxidation process of RAN on the BDD electrode. On the other hand, anodic peak potentials (E_p) were observed to slowly shift linearly to more positive values from 75 to 500 mV s^{-1} . This phenomenon, as is known, is characteristic of the irreversible electrochemical reaction. The plot of the $E_p/\log v$ (Eq.3) between $75\text{--}500\text{ mV s}^{-1}$ can be expressed as follows:

$$E_p = 0.054 \pm 0.085 \log v + 1.1978 \pm 1.238; r = 0.9949 \quad (3)$$

The number of electrons (n) transferred in the oxidation of the RAN molecule on the BDD electrode was calculated by taking measurements on CVs and using the ‘Laviron’s equation (Eq. 4) given below, which is valid in the adsorption-controlled irreversible electrode process [26].

$$E_p = E^\circ + (2.303RT / \alpha nF) \log (RTk^0 / \alpha nF) + (2.303RT / \alpha nF) \log v \quad (4)$$

The constants T , R , F , E° , v , n , k^0 and α in the formula are expressed as absolute temperature, universal gas constant, Faraday constant, formal redox potential, scanning speed, number of electrons transferred, heterogeneous transfer constant, respectively. Considering the slope of the graph of E_p (V) versus $\log v$ (mV s^{-1}), the resulting αn is 1.0. In irreversible systems, the

α value is accepted as 0.5. Thus, the value of n is calculated as 2. This result shows that 2 electrons are transferred per molecule in the redox reaction of RAN [27].

As it is known, the pH of the analytical solution is an important step that determines whether protons will take part in electrode reaction mechanisms. In studies carried out to develop a sensitive and selective voltammetric method for the determination of RAN, sharper and well-defined peaks were obtained with the SWV technique. The effect of pH value on the anodic potential and current responses at the BDD electrode was investigated in 1×10^{-4} M RAN. In order to examine the effect of supporting electrolyte and pH on the voltammetric behavior of RAN, SW voltammograms were recorded in the (0.0) - (+1.6) V potential scan range of RAN solutions prepared in the appropriate support electrolyte. For this purpose, supporting electrolyte solutions of 0.1 M Britton-Robinson buffer (pH 2.0-12.0) were used. Figure 4 shows the effect of pH on square-wave voltammograms recorded in 1×10^{-4} M RAN solutions.

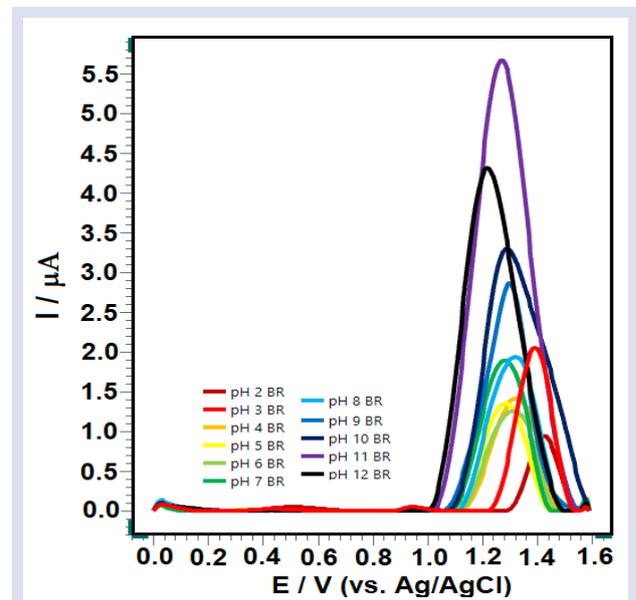


Figure 4. SWVs of 1×10^{-4} M RAN in BR buffer (pH 2.0–12.0) at a range of pH values. Electrode, BDDE; SWV parameters: 50 Hz frequency, 8 mV scan increment, and 30 mV pulse amplitude.

As seen in Fig. 4 the oxidation peak current of RAN can change with different pH values. The highest oxidation peak current was obtained at the pH 11 BR buffer. The oxidation peak potential of RAN appears to shift towards a more negative potential between pH 2 and 5. In the more basic region, pH again shifts to negative potential between 8 and 11. Therefore, there are two inclined regions of RAN on the BDD electrode surface, one being an acidic region and the other being a basic region. This result shows that protons on the BDD electrode surface are effective on the electrochemical mechanism. As can be seen from the SW voltammograms in Fig. 4, pH 11 BR buffer solution was preferred in terms of both peak morphology and oxidation peak current, and all studies

were continued in this medium. When the relationship between pH and peak potential (E_p) of RAN is examined; It can be seen that there are two slope region. The linear regression equations of RAN are expressed as follows.

$$E_{pa} \text{ (V) (pH 2 - 5)} = -0.047 \text{ pH} + 1.5178 \text{ } r: 0.996$$

$$E_{pa} \text{ (V) (pH 8 - 11)} = -0.016 \text{ pH} + 1.4477 \text{ } r: 0.995$$

When the obtained slopes are examined, it is seen that the proton transfer in acidic and basic environments changes on the BDD electrode surface of RAN. Therefore, since the slope value obtained in a strongly basic solution is closer to the theoretical value of 30 mV/pH, two electrons and one proton transfer are involved in the oxidation of RAN in BR buffer, pH 11 solution. Accordingly, the possible electrochemical oxidation mechanism of RAN has been reported previously [27].

Since the signals occurring in voltammetric techniques may vary with the variables of the device used, it is necessary to optimize these variables. For this purpose, square wave pulse variables were optimized in the range of 15 - 200 Hz frequency (f), 4 - 20 mV step potential (ΔE_s), and 10 - 100 mV pulse amplitude (ΔE_{sw}) of 1×10^{-5} M RAN in pH 11 BR buffer. The optimization process was carried out by changing one parameter at a time and keeping the others constant. By increasing the frequency, the oxidation peak current increased up to 200 Hz, but after 125 Hz, the oxidation peak current increased but the peak morphology deteriorated. For this reason, the most appropriate frequency value was determined as 125 Hz. The best result in terms of peak shape and oxidation peak current value in the potential range of 4–20 mV was obtained at $\Delta E_s = 14$ mV, and the best result was obtained at 40 mV in oxidation peak current in the pulse amplitude range of 10–100 mV.

Finally, the effect of surfactant (SDS; anionic surfactant, CTAB; cationic surfactant, Tween 20; non-ionic surfactant) on the electrochemical oxidation of RAN was examined. As is known, the sensitivity of voltammetric methods can be increased thanks to this electrostatic interaction between the electrode surface - surfactant and analyte. Figure 5 shows the interaction of RAN with cationic, anionic and nonionic surfactants in the same medium (pH 11 BR). As can be seen from the figure 5, the oxidation peak current of RAN increases in the presence of SDS, and decreases significantly in the presence of CTAB and Tween 20.

By keeping the RAN concentration constant at 5×10^{-5} M in the pH 11 BR buffer solution, the surfactant effect was investigated by adding SDS in different concentration ranges (1×10^{-5} to 1×10^{-3} M). The maximum value was observed when 8×10^{-4} M SDS (Figure 6) was added, and the 8×10^{-4} M SDS concentration was chosen for the remainder of the present analytical study.

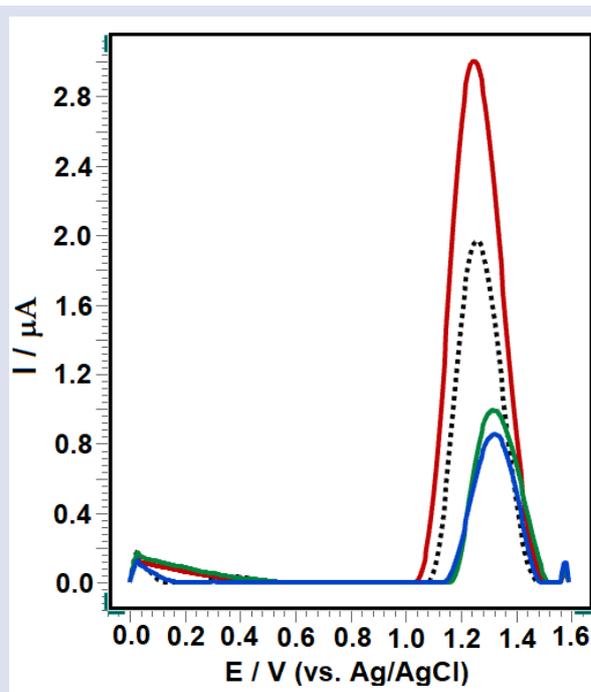


Figure 5. SWVs of 5×10^{-5} M RAN (----) in BR buffer (pH 11.0) with 8×10^{-5} M SDS (Red), CTAB (green), and Tween 20 (blue) surfactant. Electrode, BDDE; SWV parameters: 125 Hz frequency, 14 mV scan increment, and 40 mV pulse amplitude.

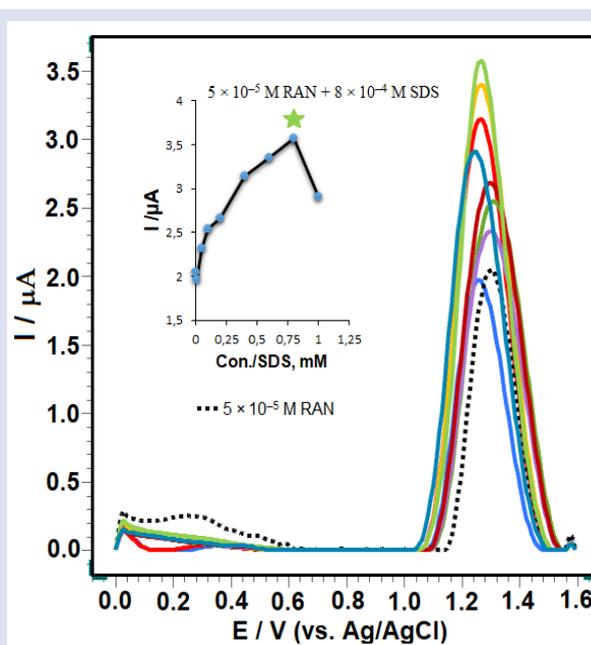


Figure 6. SW voltammograms of 5×10^{-5} M RAN in pH 11 BR buffer solution in the concentration range of 1.0×10^{-5} to 1×10^{-3} M SDS. Dashed lines represent voltammograms without SDS. Inset: i_p plot and SDS concentration. Electrode, BDDE; SWV parameters: 125 Hz frequency, 14 mV scan increment, and 40 mV pulse amplitude.

Analytical Application of RAN

The sensitivity, selectivity, repeatability and linearity range of the studied voltammetric method were investigated in detail. To examine the effect of RAN concentration on oxidation peak current under the best experimental conditions, voltammograms of RAN solutions at different concentrations were evaluated with the BDD electrode in pH 11 BR buffer. The SW voltammograms for calibration were obtained by increasing the concentrations of RAN as shown in Fig. 7. As seen in the figure, the oxidation peak currents of RAN at increase in proportion to their concentrations, and the relevant analytical parameters are summarized in Table 1. Fig. 7 shows that RAN has a linear range between 0.8 and 50 μM , [i_p (μA) = 0.1181 C (μM) + 0.0113], $r=0.999$].

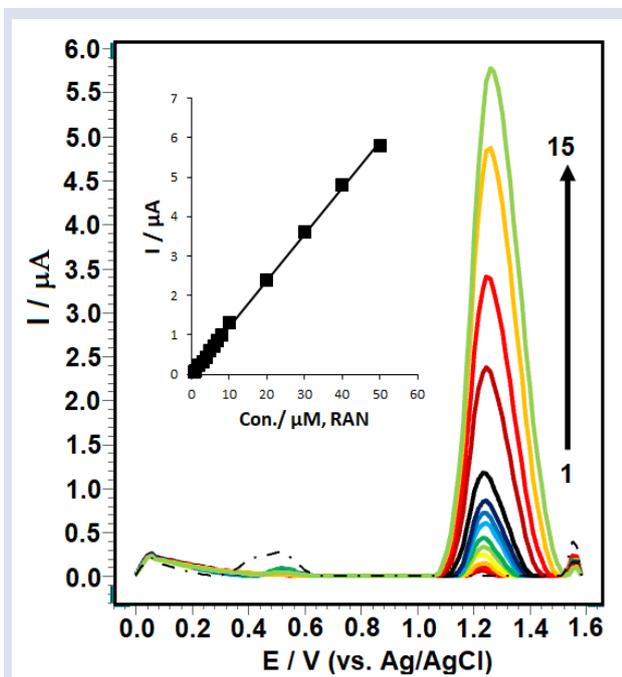


Figure 7. SWVs for RAN levels of (1–15) 0.8, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, 9.0, 10.0, 20.0, 30.0, 40.0, 50.0 μM in pH 11 BR solution with presence 8×10^{-4} M SDS. Inset shows the corresponding calibration plot for the quantitation of RAN. Electrode, BDDE; SWV parameters: 125 Hz frequency, 14 mV scan increment, and 40 mV pulse amplitude.

Table 1. Analytical validation parameters of the proposed SW voltammetric method

Parameters	Oxidation of RAN
Linearity range (μM)	0.8 – 50
Slope ($\mu\text{A}/\mu\text{M}$)	0.1181
Intercept (μA)	0.0113
Correlation coefficient	0.999
LOD (μM)	0.22
LOQ (μM)	0.74

The RSD value of the slope of calibration curve for oxidation of RAN was found to be 4.53%. This result show that the BDD electrode has very high reproducibility for the electrochemical oxidation of RAN. Comparison between previously reported methods for electrochemical oxidation of RAN and the method presented here is given in Table 2.

Table 2. Comparison of the proposed method for determination of RAN with previously reported electrochemical oxidation using different electrodes.

Electrode	LOD (μM)	Sample	Ref.
GCE	0.6	Drug	[28]
Gr-GCE	0.1	Drug, Serum	[29]
PDA-CPE	0.019	Drug, Urine	[27]
Printex 6L-GCE	0.24	Urine, Serum	[30]
BDDE	0.22	Drug	This work

GCE: Glassy carbon electrode, Gr-GCE: Graphene modified glassy carbon electrode, PDA-CPE: poly(dopamine) modified carbon paste electrode.

The precision values of the method were evaluated with the relative standard deviations (RSD) of both intraday and interday repeatability of RAN oxidation peak current values. It was taken again eight times on the same day and in three different solutions on different days, using the square wave voltammetry method. Intraday and interday % RSD values of oxidation peak currents of RSD were determined as 3.55% and 5.23%, respectively. The results show us that the repeatability of oxidation in peak current and peak potential values is quite good.

To examine the selectivity of the proposed voltammetric method, it was tested on substances that could interfere with the 3.0 μM RAN solution. In the selectivity study, no change in the peak potential of RAN was observed in the presence of Ca^{2+} , Cu^{2+} , Zn^{2+} , Ag^+ , Na^+ , NO_3^- , Cl^- , uric acid and dopamine compounds. The results obtained from these data show that; It can be said that the method designed with BDD electrode is selective. In the next step, the amount of RAN was determined using the BDD electrode at presence 8×10^{-4} M SDS. Finally, to check the practical applicability of the determined method, the analysis of syrup containing RAN was tested using the calibration method. The assay results with recoveries for the pharmaceutical formulation examined are summarized in Table 3.

Table 3. The results for quantification and recovery of RAN from the syrup sample (Zantac[®], mg/10 mL)

Declared amount	Determined amount	Recovery (%)	Bias (%)
150	148	98.66	1.34

Conclusions

Electrochemical studies of RAN in the literature are mainly related to electrochemical reduction. There are a limited number of studies on electrochemical oxidation. Here, a new electroanalytical method is proposed for the determination of RAN with BDD electrode for the first

time in the presence of SDS, an anionic surfactant. The results obtained show the sensitivity of the proposed electrochemical method and its low cost since it does not require expensive apparatus. The main advantage of the proposed method is that it is directly applicable to the analysis of biological samples without the need for separation or complex sample preparation since there are no problems caused by endogenous substances compared to other techniques. The recovery results show that the proposed procedures are sufficiently accurate and precise. Therefore, this proposed technique is quite fast, simple to implement, and most importantly, inexpensive. It can be considered as a suitable alternative to other existing analytical methods.

Conflicts of interest

There are no conflicts of interest in this work.

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