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Voltammetric Determination of Gallic Acid and its Content in Tea Samples Using Modified Iodine-Coated Platinum Electrode

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ABSTRACT

While platinum electrode shows high reactivity towards adsorption processes, iodine-coated platinum electrode offers remarkable inertness toward these processes. Therefore, iodine-coated platinum electrodes have become very significant in voltammetric applications. This study reports the use of the modified iodine-coated polycrystalline platinum electrode as a voltammetric sensor for gallic acid determination in tea samples. The optimized experimental parameters for determining gallic acid used 0.5M H_2SO_4 as a supporting electrolyte with a scan rate of 50 mV/s. The anodic peak related to gallic acid oxidation was centered at approximately 0.60V. The extended detected linear range for the developed method was between 0.025 and 2.0 mM. The anodic current showed excellent linearity with R^2 =0.9975. The limit of detection (LOD) and limit of quantitation (LOQ) were 0.022mM and 0.0669 mM, respectively, which confirmed the method's sensitivity. The investigation for the effect of potential interferences from tea components indicated a specific selectivity toward gallic acid with only slight interference due to the presence of ascorbic acid and the absence of any electrochemical response toward glucose, caffeine, Ni^{2+}, K^+, Na^+, and Zn^{2+}. The proposed method was successfully applied in the analysis of gallic acid in tea samples. The recovery values (90.0 - 103.25%) obtained in this study support the use of the developed method to analyze gallic acid in tea samples.

Keywords : Gallic acid, Tea, Voltammetric analysis, Iodine-coated platinum electrode, Modified platinum electrode.

Introduction

In recent times, the analysis of gallic acid ($C_7H_6O_5$, Figure 1) has been a subject of great interest because it is one of the main phenolic components with important properties due to its significant antioxidant activities,[1] pharmacological activity as radical scavengers,[2] besides its anticancer properties.[3]Gallic acid is found in black and green tea,[4] mango,[5] and several other plants.[6] Several methods have been reported for the determination of gallic acid in various matrices. These methods include flow injection analysis,[7] resonance light scattering,[8] high-performance liquid chromatography (HPLC),[9,10] and electrochemical methods.[11-15] All the methods mentioned above are expensive, possess low reproducibility and required highly trained personnel. It, therefore, necessitates the need to develop an accurate, simple, reliable, and fast method for gallic acid analysis in matrices. However, electrochemical methods have attracted much attention due to their low sensitivity toward the matrix effects compared to other analytical techniques. The direct use of conventional electrodes like platinum, graphite, and glassy carbon electrodes is challenging due to their high overpotential, leading to electrode fouling, poor selectivity, and low reproducibility.[16] These have opened a research interest towards the modification of solid electrodes.[17] Such change of solid electrode surfaces is intended to improve the electrochemical methodology. For instance, adsorbed iodine on platinum electrode surface enhances voltammetry's reproducibility and simplifies background behavior.[18] Also, coating of solid electrodes surface alters the kinetics and mechanisms of reactions run at the electrode surface. Iodine is one of the anions adsorbed to an electrode surface. The chemisorption process is achieved in two ways; from solution or vacuum to form stable chemisorbed monolayers; subsequently, the iodine coated electrode is rinsed or evacuated.[19] The iodine is adsorbed at potential 0.2 V vs Ag/AgCl or SCE reference electrodes,[20] which is the double layer potential. At the surface of the polycrystalline platinum electrode, the reaction of the iodine anions from the solution leads to the spontaneous chemisorption of iodide anion to form stable neutral iodine atoms accompanied by an evolution of hydrogen gas. The adsorbed iodine is less reactive toward electrochemical oxidation than the free iodine anion in solution [21] and depends on the electrode potential .[22] The chemisorbed iodine could be desorbed from the platinum electrode surface if the potential scanned is lower than -0.2 V, leading to a reduction of hydrogen ions and hydrogen gas generation.[21] Also, the rate of iodine desorption from the electrode surface increases as the potential become more negative .[22] In the positive direction, the chemisorbed iodine begins to desorbed at a potential of 1.0 V .[20] Studies have shown that carbon monoxide (CO) completely desorbed iodine from platinum electrode surface at potentials lower than 0.35 V, while at higher potentials, the desorption is incomplete.[23]

Iodine-coated platinum electrode has been applied for the electrochemical determination of organic and non-organic species in many studies.[24-32] Iodine-coated platinum electrode is characterized by the simplicity in preparation, application, and the use of environmentally friendly chemical reagents. The simplicity of the method's instrumentation stimulates our interest in the present

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research. This work aims to develop a simple method for gallic acid determination in black and green tea samples.

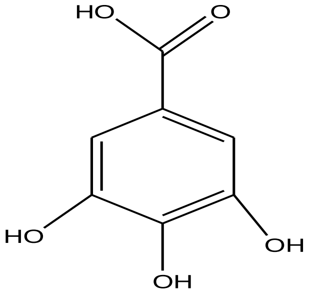

Figure 1: The chemical structure of gallic acid

Materials and Methods

Materials

A potentiostat (PAR model 362, EG & G) connected to a computer via GPIB interface (IEEE) data procuration was used. Locally modified Labview® (IEEE) software was used for data procuration. The used electrochemical cell comprises one compartment with one inlet/outlet for gas purging and blanketing with nitrogen gas. A 0.5 mm polycrystalline platinum wire purchased from Aldrich (99.99% minimum purity certified reagent) was used as a working electrode. The electrode surface area was determined by folding the immersed end to a U-shape to mark a constant surface area of the immersed part of the electrode. Ag/AgCl was used as a quasi-reference electrode (QRE). The auxiliary electrode used was a 0.5 mm polycrystalline platinum wire (Aldrich, certified 99.99% minimum purity). All chemicals used were analytical grade and used as obtained without further purification. Sulfuric acid (95-97%) was supplied from Merck, Gallic acid (97.5-102.5%) was purchased from Sigma-Aldrich, potassium iodide was purchased from Sigma-Aldrich. A high purity water system (Millipore-MilliQ) was used for the preparations of all solutions. The N_2 gas was a five grade, 99.999% minimum purity supplied from the International Jordanian Gases Company (Amman, Jordan).

Preparation of iodine-coated platinum electrode

Preparation of iodine-coated platinum electrode was described in one of our previous publications[24]. The preparation steps include cyclization of a platinum electrode immersed in 0.5M H_2SO_4 between -0.25V and 1.3V until a reproducible cyclic voltammogram of the platinum electrode is obtained, with a defined oxygen and hydrogen adsorption/desorption peaks, indicative of the complete cleanliness of the electrode surface and electrochemical cell contents (Figure 2). After that, the clean electrode was placed in a 0.5M H_2SO_4+10.2M KI solution for five minutes under a potentiostatic condition of closed-circuit in the double layer region (~ 0.2V) dosing of the platinum electrode surface with iodine. After that, the electrode was subjected to multiple rinses with water and a 0.5M H_2SO_4 solution. The electrode potential was cycled in a 0.5M H_2SO_4 solution between -0.2V and +0.8V at 50 mv/s to verify the completeness of the coating process (Figure 2). The absence of oxygen and hydrogen adsorption-desorption peaks from the recorded cyclic voltammograms of an iodine-coated platinum electrode is indicative of complete coverage of platinum electrode surface with a monolayer of iodine.

Sample preparation

The tea samples were purchased from the local Jordanian market. Three brands were analyzed for their gallic acid content. The tea samples were prepared by adding 10 mL of Millipore-MilliQ water to 100 mg of dry tea in a 50 mL conical flask and stirred over a hot plate at 80ºC for 10 min. Then, the tea solution was filtered off through a Whatman paper (No. 1). The tea filtrate was left to cooled to room temperature and then diluted to 50 mL with 0.5M H_2SO_4 solution.

Statistical Analysis

All the experiments were conducted in triplicate. Statistical analyses were carried out using Microsoft Excel 2007. The oxidation peak current was extracted for each cyclic voltammogram and plotted against concentration to generate the standard curves using linear regressions. Data were expressed as means ± S.D.

Results and Discussion

The study revealed a reproducible cyclic voltammogram for the polycrystalline platinum electrode (Figure 2-a), confirming the cleanness of all components of the electrochemical system. On the other hand, the cyclic voltammogram of the iodine-coated electrode (Figure 2-b) indicates complete oxidation by the absence of any surface activity in the scanned potential range (-0.2–0.80V). The complete absence of hydrogen and oxygen reduction features is the primary evidence for full coverage of the platinum surface. The potential scan was limited to the range mentioned above to avoid desorption of iodine from the platinum surface. Figure 3 shows a representative irreversible cyclic voltammogram for the iodine-coated platinum electrode in 10mM gallic acid+0.5M H_2SO_4. The recorded cyclic voltammogram shows a prominent anodic peak for gallic acid oxidation at a potential of~ 0.60V versus Ag/AgCl at a scan rate of 50 mv/s. Subsequently, the anodic peak was considered as the analytical signal for gallic acid determination.

The effect of pH on the iodine coating on platinum electrode has been reported elsewhere [33] where it was found that iodine coating is unstable upon cyclization in basic solutions.[34] This fact limits the use of an iodine-coated electrode to acidic solutions only. The effect of the pH in the acidic range was investigated by following the effect of pH change on the peak current for gallic acid oxidation. Three representative pH values, 7, 3.5, and 0.3, were selected to demonstrate the effect of solution acidity on the analytical signal (peak current). Gallic acid standard solution was analyzed in 0.5M H_2SO_4 (pH=0.3), phosphate buffer (pH=3.5), and 0.1M KCl (pH=7) media. Figure 4 shows the effect of pH on the gallic acid oxidation current peak. As shown in Figure 4, the value of the oxidation peak current of gallic acid is highest at a pH of 0.3. After that, 0.5M H_2SO_4 was considered a supporting electrolyte to determine gallic acid in this study.

The effect of scan rate on the obtained anodic peak current of gallic acid was also studied. As presented in Figure 5, a linear relationship was obtained between the square root of scan rate and oxidation peak current of gallic acid over the range of 10-100 mV/s, which suggested a diffusion-controlled irreversible oxidation process of gallic acid at the iodine-coated platinum electrode.

The obtained cyclic voltammograms for the iodine-coated platinum electrode in a series of gallic acid standard solutions show that the oxidation current increased steadily with gallic acid concentration (Figure 6). Three voltammograms were recorded for each standard solution. The anodic peak current was extracted for each cyclic voltammogram. Plotting the anodic peak current variation against gallic acid concentration gave a straight and extended dynamic range with concentrations ranging from 0.025 mM to 2.0 mM. The calibration curve displayed in Figure 8 shows remarkable linearity; R^2=0.9975 and the calibration equation is given by: $I\ (\mu A)=76.913C_{Gallic\ acid}+10.592$ Where i indicates the anodic peak current attributed to gallic acid oxidation as presented in Figure 7.

The precision, that is, the repeatability of the method was assessed by extracting the anodic peak current of recorded cyclic voltammograms for a solution containing 1mM of gallic acid. The standard deviation obtained for 20 successive measurements was 2.67% which attests to the high precision of the developed voltammetric method.

The limit of detection based on the formula $LOD=3.3\sigma/S$, and the limit of quantitation based on the formula $LOQ=10\sigma/S$, where σ represents the blank signal (background current), and S represents the sensitivity of calibration curve were calculated. The estimated limits were 0.022 mM and 0.0669 mM, respectively.[35] Acceptable sensitivity of the applied method with high precision was obtained. Higher sensitivity can be achieved by using a more sensitive technique like differential pulse voltammetry. However, differential pulse voltammetry technique was not used because cyclic voltammetry provides satisfactory sensitivity for gallic acid determination in pharmaceutical formulations.

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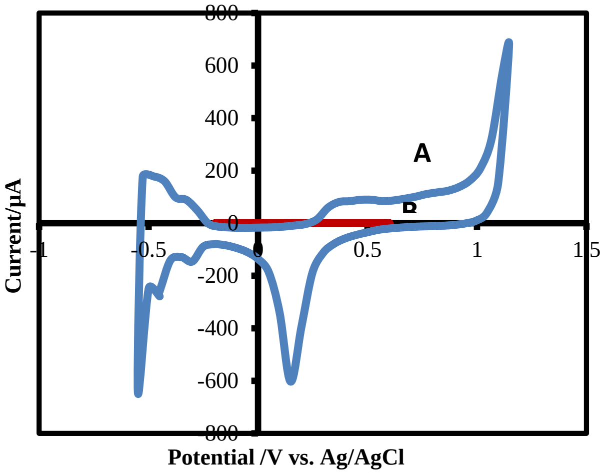

Figure 2: Cyclic voltammogram curves of (a) platinum electrode and (b) the same electrode after adsorption of iodine from 1x10^{-2} MKI in 0.5 M H_2SO_4 solution.

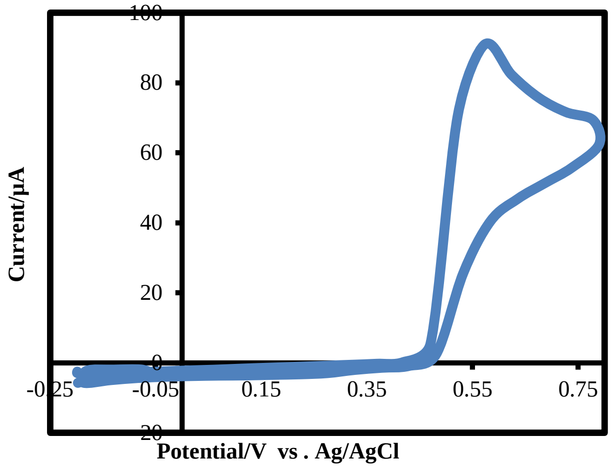

Figure 3: Cyclic voltammogram of 10mM gallic acid in 0.5 M H_2SO_4solution at the iodine-coated electrode and a scan rate of 50 mV/s.

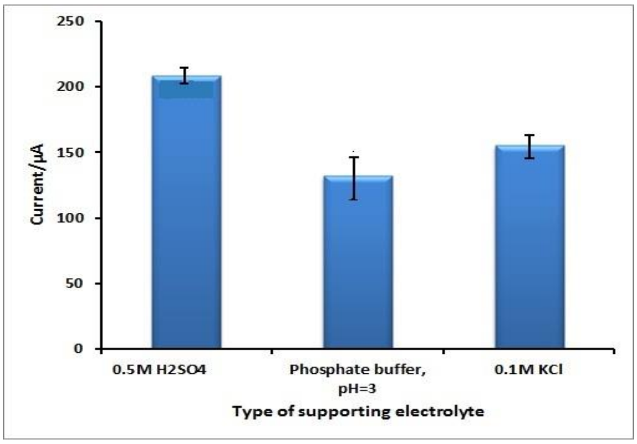

Figure 4: Effect of supporting electrolyte on the electrochemical signal of gallic acid at the iodine-coated platinum electrode, 2.35mM gallic acid; 0.5 M H_2SO_4; phosphate buffer pH = 3.0; 0.1M KCl, n = 3; Scan rate:50 mV/sec.

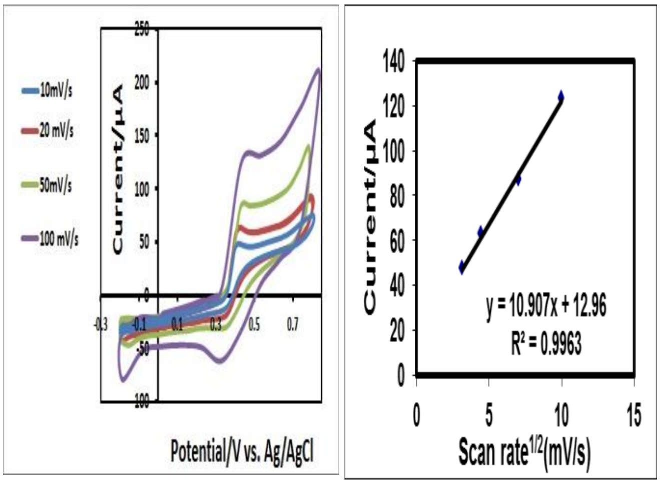

Figure 5: a) Cyclic voltammograms of an iodine-coated platinum electrode in 0.5 M H_2SO_4 and 1mM of gallic acid obtained at scan rates of 10, 20, 50 100 mv/s. b) The least-square line for the gallic acid oxidation peak current vs the square root of scan rate.

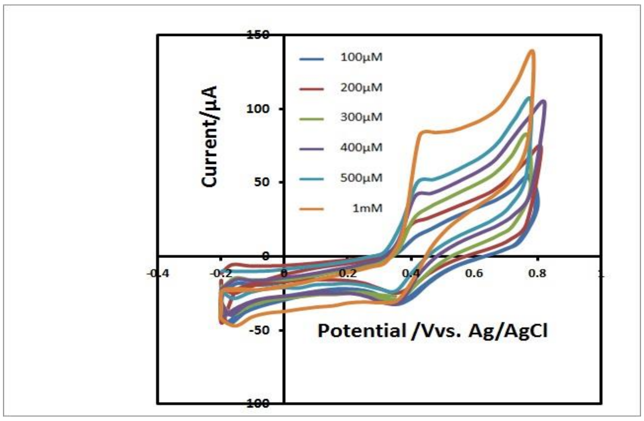

Figure 6: Cyclic voltammograms of iodine-coated platinum electrode in 0.5M H_2SO_4 solution containing 100,200, 300,400,500µMand 1mM of gallic acid. All the scans were recorded at a scan rate of 50 mV/s.

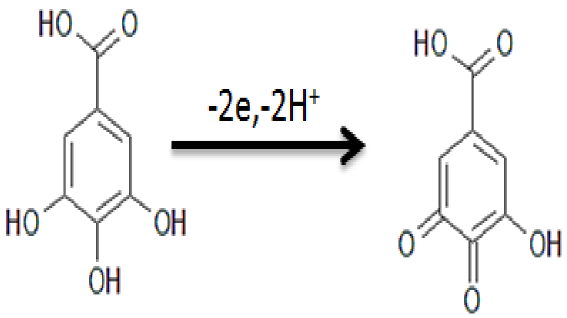

Figure 7: Gallic acid oxidation equation

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Potential interference

Method selectivity is a critical feature in analytical studies; hence, the developed method (the iodine-coated platinum electrode), was examined for possible interference with elements such as ascorbic acid, glucose, caffeine, and some cations as Ni^{2+}, K^{+}, Na^{+}, as well Zn^{2+}.[36] This study showed that glucose, caffeine and the cations did not exhibit any electrochemical response toward the iodine-coated platinum electrode, even at a 30-fold concentration of ascorbic acid compared to gallic acid. However, there was a negligible interference with the peak of gallic acid (Figure 9), observed as a slight increase (31.35%) in gallic acid signal compared with the gallic acid signal in the absence of ascorbic acid. The results demonstrate that despite this common interferent, the iodine-coated platinum electrode can be used to determine gallic acid.

Recovery

The validity of the developed voltammetric method for gallic acid determination was tested on three types of tea samples, Rabee, Alghazaleen, and Lebton. The Gallic acid standard of known concentration, 10 mM was spiked into the samples to evaluate the percentage recovery. The standard addition method was applied to determine the concentration of gallic acid in tea samples. The determined concentration of gallic acid in tea samples was between 0.016 and 0.095 mM (n=3) for both green and black tea sample, corresponding to 0.68 mg/100 mg to 4.04 mg/100 mg tea samples. As listed in Table 1, the recoveries for gallic acid in the tea samples are between 90.0 and 103.25 %, showing the appropriateness of the iodine-coated electrode for the quantitative analysis of gallic acid.

A comparison between the developed voltammetric method and some of the standard analytical and voltammetric methods for gallic acid determination in terms of limit of detection and the linear range is presented in Table 2. As shown, the iodine-coated platinum electrode exhibited a lower concentration detection limit than that of the square wave voltammetric method.[36] Also, a convenient and extended linear range compared to other voltammetric methods was obtained. [36,37] In addition, the developed voltammetric method application has several advantages such as simplicity in sample preparations and analysis, short time of analysis, and the low price of instrumentations compared with other analytical methods.[38-40]

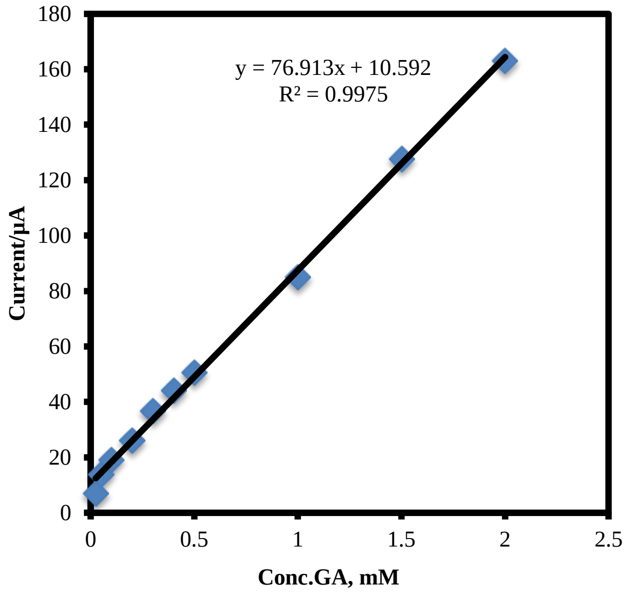

Figure 8: An extended calibration curve shows the relationship between gallic acid concentration in mM and the oxidation peak current measured from cyclic voltammograms for gallic acid in 0.5 M H_2SO_4 solution at the iodine-coated platinum electrode. Scan rate = 50 mv/s.

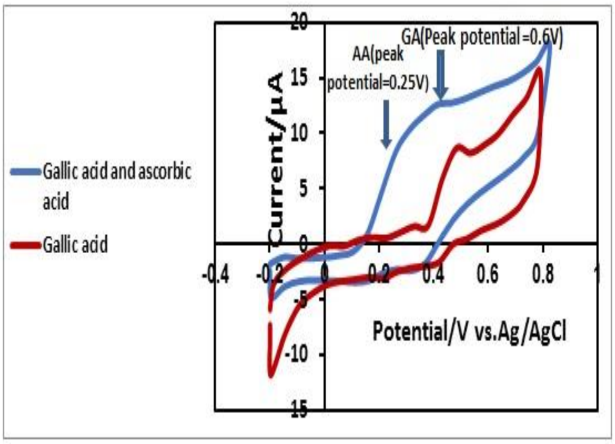

Figure 9: The cyclic voltammograms of **(a)** 0.05mM gallic acid (GA) in 0.5M H_2SO_4, **(b)** 0.05mM of gallic acid (GA) +1.5 mM of ascorbic acid (AA) in 0.5 M H_2SO_4.scan rate:50 mv/sec.

Table 1: Recovery test for gallic acid in tea samples using cyclic voltammetry technique at the iodine-coated platinum electrode (n = 3).

Tea sample	Added (mM)	Detected (mM)	Recovery (%)	RSD (%)
Rabee®	0	0.0504	-	-
(green tea)	0.2	0.229	90.0	10.92
	0.4	0.448	99.50	4.46
Alghazaleen®	0	0.016	-	-
(black tea)	0.2	0.195	92.025	5.12
	0.4	0.429	103.25	7.23
Lebton®	0	0.095	-	-
(black tea)	0.2	0.289	97.00	2.214
	0.4	0.5045	102.37	6.34

Table 2: A comparison of the analytical performance of the developed voltammetric method using the iodine-coated platinum electrode with other electroanalytical methods reported in the literature.

Method	Linear range (µM)	LOD (µM)	Ref
Calorimetry	0.01-15	0.105	38
Mass Spectrometry	$2.936x10^{-4}$-$2.939x10^{-3}$	$8.876x10^{-5}$	39
HPLC	$1.469x10^{-2}$-0.176	$2.35x10^{-3}$	40
Square Wave voltametry	20-166	30.8	36
Cyclic Voltammetry	200-980	-	37
Cyclic Voltammetry	25-2000	22	This work

Conclusion

An electrochemical analytical method for gallic acid determination using iodine-coated platinum electrode was demonstrated in this work. The developed method is simple, fast with reproducible results. It could be considered a green method due to its simplicity in analytical procedures. The developed method reported an extended dynamic range (0.025-2.0) mM, high sensitivity, and low detection limit for gallic acid determination. It was successfully applied in the quantification of gallic acid in three tea samples. The results were reproducible with % RSD less than 11% and excellent percentage recovery, making the developed method suitable for real-life applications.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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