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Research Article

Composite Metal Oxide Modified–Graphene Oxide Electrode for Gallic Acid Determination

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Abstract

The development of selective and sensitive electrochemical sensors for the determination of phenolic compounds remains an important issue due to the limited stability and sensitivity of electrode materials. In this study, composite metal oxide modified graphene oxides were prepared on the surface of a glassy carbon electrode. For this aim, a graphene oxide-modified/glassy carbon electrode (GO/GCE) was initially prepared using the drop-dry technique. Subsequently, tellurium oxide- zinc oxide nanoparticles (TeOx-ZnOx) were modified onto the GO/GCE surface using an electrochemical method (TeOx-ZnOx/GO/GCE). The prepared modified surface was compared with other electrodes using electrochemical impedance spectroscopy and cyclic voltammetry. The electrochemical behavior of gallic acid was investigated with the TeOx-ZnOx/GO/GCE in a pH 2.3 Britton-Robinson buffer. Under optimized conditions, the sensor exhibited a linear range of 0.5 μmol L^{-1} -1000 μmol L^{-1} for gallic acid detection using the amperometry technique, with a limit of detection (LOD) of 0.2 μmol L^{-1}. Real sample analysis was successfully performed with tea samples. In conclusion, the proposed composite modified electrode provides a stable surface for accurate and sensitive analysis of gallic acid determination. As a result, the prepared TeOx–ZnOx composite structure contributes to the literature by providing a stable and sensitive platform as a new electrode surface for the determination of gallic acid.

Keywords: Gallic acid, graphene oxide, tellurium oxide, zinc oxide, modified electrode

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Gallik Asit Tayinine Yönelik Kompozit Metal Oksit ile Modifiye Edilmiş Grafen Oksit Elektrot

Öz

Fenolik bileşiklerin tayini için seçimli ve duyarlı elektrokimyasal sensörlerin geliştirilmesi, elektrot malzemelerinin sınırlı kararlılığı ve hassasiyeti nedeniyle önemli bir konu olmaya devam etmektedir. Bu çalışmada kompozit metal oksit modifiye grafen oksitler camımsı karbon elektrot yüzeyinde hazırlanmıştır. Bu amaçla, öncelikle drop-dry tekniğiyle grafen oksit modifiye/camımsı karbon elektrot (GO/GCE) hazırlanmıştır. Ardından GO/GCE yüzeyine elektrokimyasal yöntemle tellür oksit-çinko oksit nanoparçacıklar (TeOx-ZnOx) modifiye edilmiştir (TeOx-ZnOx/GO/GCE). Hazırlanan modifiye yüzey elektrokimyasal impedans spektroskopisi tekniği ve döngüsel voltammetri kullanılarak diğer elektrotlar ile karşılaştırılmıştır. TeOx-ZnOx/GO/GCE ile gallik asidin elektrokimyasal davranışı pH 2.3 Britton-Robinson tamponu içerisinde incelenmiştir. Optimize edilmiş koşullar altında, sensörün gallik asidi tespit etmek için amperometri tekniği ile elde edilen lineer ölçüm aralığı 0.5 µmol L^{-1} - 1000 µmol L^{-1} olup, LOD (tayin sınırı) 0.2 µmol L^{-1} olarak belirlenmiştir. Gerçek örnek analizi çay örneğinde başarıyla çalışılmıştır. Sonuç olarak, önerilen kompozit modifiye elektrodun, gallic acid tayinine yönelik kararlı bir yüzey olarak doğru ve duyarlı bir analize imkan sağladığı görülmektedir. Sonuç olarak, hazırlanan novel TeOx–ZnOx kompozit yapısı, gallic acid tayini için yeni bir elektrot yüzeyi olarak kararlı ve duyarlı bir platform sunarak literatüre katkı sunmuştur.

Anahtar Kelimeler: Gallik Asit, grafen oksit, tellür oksit, çinko oksit, modifiye elektrot

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1. Introduction

Gallic acid is a phenolic compound naturally found in various plants. Known primarily for its antioxidant properties, it also exhibits antimicrobial, anti-inflammatory, and potential anticancer activities [1–3]. Thanks to these beneficial properties, it is widely used in the pharmaceutical, food and cosmetic industries [4–6]. Accurate determination of the amount of gallic acid in various samples is of great importance for both monitoring product quality and evaluating health-related effects [7].

Various analytical techniques such as spectrophotometry, high-performance liquid chromatography (HPLC) [8,9], capillary electrophoresis [10], and electrochemical techniques [11,12] have been employed for the determination of gallic acid. However, many of these methods have certain drawbacks, including the need for expensive instrumentation, complex sample preparation procedures, and time-consuming processes. Due to these limitations, electroanalytical methods—particularly voltammetric techniques—have gained increasing attention in recent years for the analysis of phenolic compounds such as gallic acid [13,14]. Electrochemical sensors are of particular interest owing to their high sensitivity, rapid detection capabilities, and versatility in surface modification with different materials [15,16].

Graphene oxide (GO) is one of the frequently preferred materials as electrode material in sensor applications. These structures obtained by oxidation of graphite are a nanomaterial that attracts attention thanks to its large surface area and functional surface groups. The fact that it can be easily modified with many materials makes it preferred for a wide variety of applications [17,18]. Metal oxides are compounds with a wide range of electronic and structural diversity [19]. In recent years, composite metal oxide structures formed by combining different metal oxides have attracted increasing interest in electrochemical applications. Such composites offer higher surface activity, improved conductivity and enhanced electrocatalytic performance by combining the individual properties of the components. In particular, combinations of semiconducting metal oxides such as tellurium oxide, manganese oxide, zinc oxide, nickel oxide and cobalt oxide accelerate charge transfer on the electrode surface and significantly increase the sensitivity of sensors [20–23]. Thus, it becomes possible to develop both low cost and high performance electrochemical sensors.

Although various metal oxides have been used in many different applications, TeOx has been studied to a limited extent despite its redox-active properties that facilitate electron transfer. ZnOx, on the other hand, is a widely used material due to its high stability and catalytic activity. Combining these two oxides is expected to produce a synergistic effect, leading to increased conductivity, improved electrocatalytic response, and a more stable electrode surface. No studies have been reported in the literature on the use of a TeOx–ZnOx composite structure integrated with graphene oxide for gallic acid determination. This represents the originality of the study. In this study, a new electrode developed by combining composite metal oxides with graphene oxide structure is presented. It was prepared by modification of graphene oxide on the glassy carbon electrode surface followed by electrochemical deposition of TeOx-ZnOx nanoparticles. The electrochemical performance of the prepared electrode surface on the determination of gallic acid was evaluated. The results obtained from CV and amperometry studies under optimum conditions show that the developed electrode surface is a practical and effective alternative for gallic acid analysis.

2. Material and Methods

Gallic acid, tellurium dioxide (TeO_2), zinc sulfate ($ZnSO_4$), and graphene were purchased from Sigma-Aldrich. Britton-Robinson (B-R) buffer solutions in the pH range of 2.0–6 was prepared using a mixture of acetic acid, phosphoric acid, and boric acid, and the desired pH was adjusted with 0.2 M NaOH. All solutions were prepared with ultrapure deionized water.

Electrochemical measurements were carried out using an Autolab PGSTAT 101 voltammetric device in a standard three-electrode system (modified glassy carbon electrode (GCE) as the working electrode, a platinum wire as the counter electrode, and an Ag/AgCl (sat. KCl) electrode as the reference). Cyclic voltammetry (CV) and amperometry were used for analytical studies under optimized conditions in 0.1 M B-R buffer solution (pH 2.3). Electrochemical impedance spectroscopy (EIS) was employed to characterize the modified electrode surfaces.

Fabrication of the TeOx-ZnOx/GO Modified Electrode

Initially, the bare GCE was polished with alumina slurry, rinsed with distilled water, and sonicated in ethanol and water sequentially. To prepare the GO-modified electrode, a homogeneous dispersion of GO (10 µL) was drop-cast onto the clean GCE surface and dried at room temperature. The composite metallic oxide layer was subsequently deposited by electrochemical codeposition. For this, the GO-modified electrode was immersed in a solution containing 3.0 mM TeO_2 and 1.0 mM $ZnSO_4$ dissolved in 0.1 M HCl, and CV was applied in the potential range of –1.3 V to +1.0 V for 10 cycles at 100 mV s^{-1}. The obtained TeOx-ZnOx/GO/GCE was rinsed and dried before use.

Real Sample Preparation

A black tea sample was purchased from a local market. The preparation procedure was followed according to our previous publication [24], ensuring consistency with earlier studies. Each real tea sample analysis was performed in triplicate to ensure reproducibility. 2 g of dried tea leaves were infused in 50 mL of hot distilled water for 15 minutes. After cooling to room temperature, the solution was filtered to obtain a clear extract. An appropriate volume of the filtrate was diluted with supporting electrolyte and used directly for voltammetric measurements. Known concentrations of standard gallic acid solution were added to the tea sample for recovery studies.

3. Results and Discussion

Fig. 1A shows the cyclic voltammetry (CV) curves used to compare the electrochemical behavior of bare GCE, GO/GCE, TeOx-ZnOx/GCE, and TeOx-ZnOx/GO/GCE electrodes in 0.1 M B-R buffer solution (pH 2.3) containing 0.1 mmol L^{-1} gallic acid. The bare GCE showed a weak and broad oxidation peak, indicating sluggish electron transfer and limited surface activity. Upon modification with GOs, a clear enhancement in peak current was observed, which is mainly attributed to the large surface area of GO. When the GCE was modified with TeOx-ZnOx nanoparticles, a current increase was detected. This can be explained by the redox behavior of TeOx, which plays a key role in promoting electron exchange at the electrode surface. ZnOx likely contributes to a more stable structure and additional active sites. The most significant enhancement in electrochemical response was obtained at the TeOx-ZnOx/GO/GCE electrode. The remarkable increase in oxidation peak current indicates a synergistic effect among TeOx, ZnOx, and GO. While GO provides a large and conductive surface, TeOx improves electron transfer kinetics, and ZnOx offers structural support and catalytic contribution. The combination of these materials results in a composite surface with enhanced electron transfer capability and improved interaction with gallic acid molecules. These findings indicate that the proposed TeOx-ZnOx/GO/GCE electrode offers a promising surface for the electrochemical determination of gallic acid.

Moreover, the interfacial properties of all electrodes were studied via EIS measurements were carried out in a solution containing 5.0 mmol L^{-1} $[Fe(CN)_6]^{3-/4-}$ and 0.1 M KCl. The Nyquist plots (Fig. 1B) clearly reveal that the charge transfer resistance (Rct) values vary significantly among the different electrode surfaces. Bare GCE exhibited the largest semicircle diameter, corresponding to the highest Rct (698 Ω), indicating poor electron transfer kinetics. Modification with GOs significantly reduced Rct (387 Ω), reflecting the improved conductivity and enhanced charge transport pathways offered by the graphene network. The TeOx-ZnOx/GCE electrode also showed a smaller semicircle than bare GCE, due to the redox-active behavior of TeOx and the catalytic contribution of ZnOx. The TeOx-ZnOx/GO/GCE electrode exhibited the smallest semicircle among all, indicating the lowest Rct (72 Ω) and the increased charge transfer characteristics. This confirms the synergistic effect between the conductive GOs and the electrocatalytically active TeOx-ZnOx nanoparticles. The combined structure promotes efficient electron transfer at the electrode–electrolyte interface, which agrees with the CV results and supports the good performance of the composite electrode for gallic acid detection.

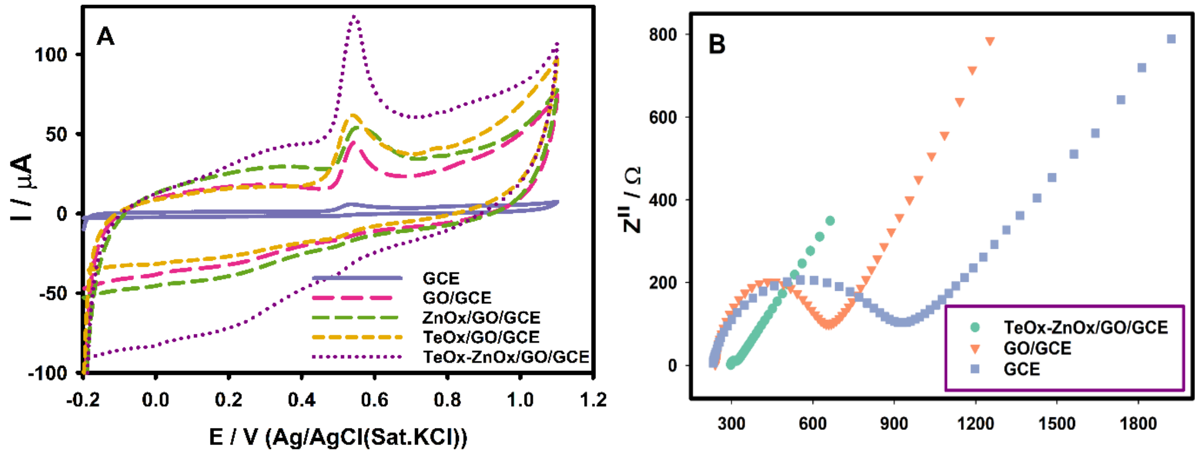

Figure 1. A) CVs of GCE, GO/GCE, TeOx/GO/GCE, ZnOx/GO/GCE, TeOx-ZnOx/GO/GCE electrodes recorded in 0.1 mmol L^{-1} gallic acid in pH 2.3 B-R buffer solution. **B)** Nyquist plots of the modified electrodes in a solution containing 5 mmol L^{-1} $[Fe(CN)_6]^{3-/4-}$ and 0.1 M KCl.

The electrochemical behavior of gallic acid at the TeOx-ZnOx/GO/GCE electrode was further investigated by cyclic voltammetry at varying scan rates between 5 and 200 mV s^{-1} (Figure 2). As the scan rate increased, an enhancement in the anodic peak current was observed. In addition, the anodic peak potential showed a slight positive shift with increasing scan rate, which is characteristic of a quasi-reversible electron transfer. The inset of Figure 2 demonstrates a good linear relationship between the anodic peak current (Ipa) and the square root of scan rate ($v^{1/2}$), indicating that the oxidation of gallic acid occurs through a diffusion-controlled process on the modified electrode surface. The corresponding linear regression was obtained as Ipa= 6.974 $v^{1/2}$ -4.51169 ($R^2 = 0.9992$).

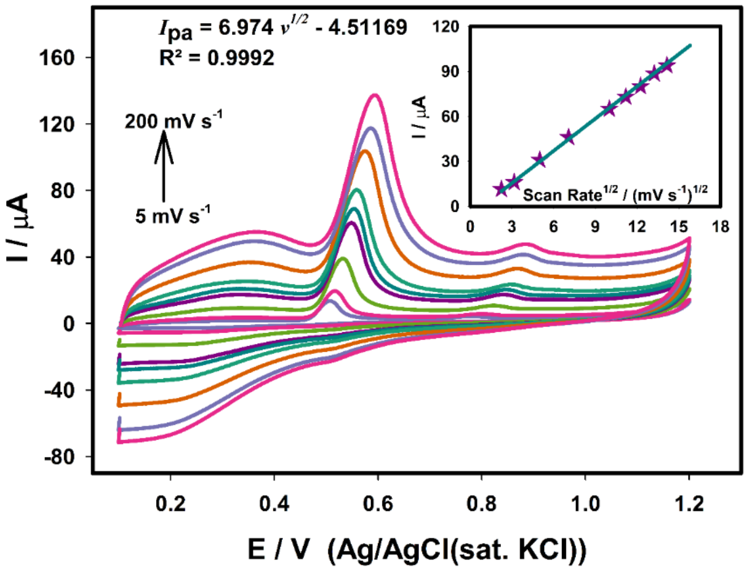

Figure 2. CVs of gallic acid (10 µM in pH 2.3 B-R buffer) at the TeOx-ZnOx/GO/GCE electrode recorded at different scan rates (5–200 mV s^{-1}). (Inset: The linear relationship between the anodic peak current and the square root of scan rate).

Fig. 3 shows the amperogram of gallic acid (GA) added at increasing concentrations in pH 2.3 BR buffer solution. Increasing peak currents have been observed at the TeOx-ZnOx/GO/GCE electrode with rising GA concentrations. As can be seen from the calibration curve in the inset, a linear relationship has been obtained within the concentration range of 0.5 μmol L^{-1} to 1000 μmol L^{-1}, described by the equations as; Ip (μA)=20032·C_{GA} (mol L^{-1}) + 9.9447 (R^2 = 0.9928), and Ip (μA)=108731·C_{GA} (mol L^{-1}) – 0.1004 (R^2 = 0.9987) with the detection limit has been calculated as 0.2 μmol L^{-1}. These results demonstrate the success of the proposed electrode for GA determination.

The analytical performance of the proposed TeOx–ZnOx/GO/GCE electrode has been compared with previously reported gallic acid sensors. The obtained LOD and wide linear range are comparable to those of carbon paste electrodes modified with carbon nanotubes (LOD: 0.3 μmol L^{-1}, range: 0.5–15 μmol L^{-1} [25]), and gelatin-functionalised reduced graphene oxide (LOD: 0.47 μmol L^{-1}, range: 1–110 μmol L^{-1} [26]), and superior to several previously reported electrodes. Although some advanced nanocomposite-based electrodes, such as Poly(L-Methionine)/Carbon Nanotube Glassy Carbon Electrode (LOD: 3.1 nmol L^{-1} [24]) or ZrO_2/Co_3O_4/ reduced graphene oxide nanocomposite (LOD: 1.56 nmol L^{-1} [27]), exhibit lower detection limits, their linear ranges are relatively narrow. In contrast, the TeOx–ZnOx/GO/GCE electrode offers a balanced performance with both a low detection limit and a broad linear range.

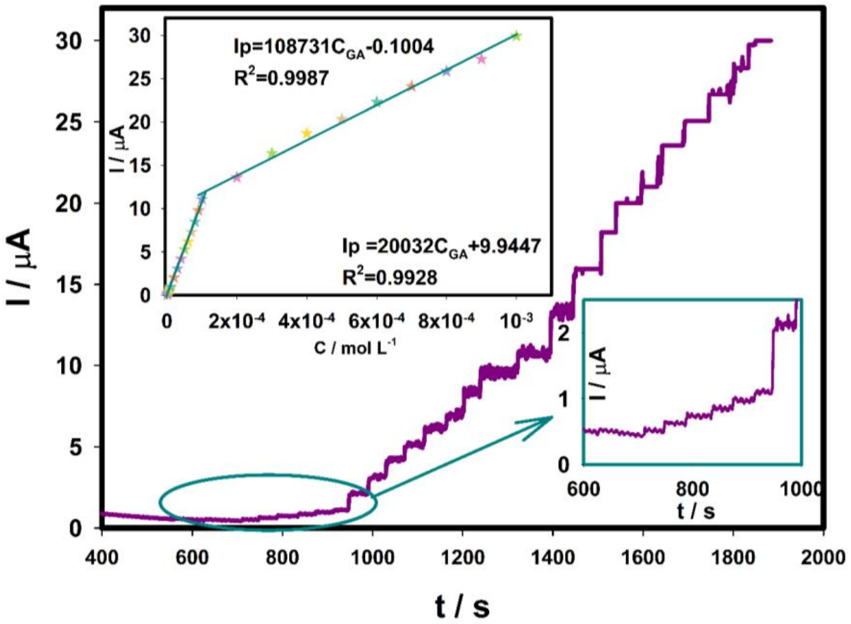

Figure 3. Amperometric measurement results of gallic acid at TeOx-ZnOx/GO/GCE electrode with increasing concentrations (0.5–1000 μmol L^{-1}) in pH 2.3 B-R buffer solution. (Inset: The related calibration curve).

Stability is an extremely important parameter, especially for electrochemical sensors that require long-term and repetitive use. As shown in Figure 4, the stability of the TeOx-ZnOx/GO/GCE electrode was investigated over a period of 7 days by recording the GA oxidation peak current values. During this time, the electrode retained 96.6% of its initial peak current, demonstrating high stability with no significant signal loss. These results indicate that the developed electrode has excellent operational stability and confirm its potential as a promising candidate for sensor applications. Moreover, five independently prepared TeOx–ZnOx/GO/GCE electrodes were tested under same experimental conditions. The relative standard deviation values were calculated as 3.9% for intra-day (n = 5) and 4.3% for inter-day (n = 5) measurements, confirming good repeatability and reproducibility of the electrode response.

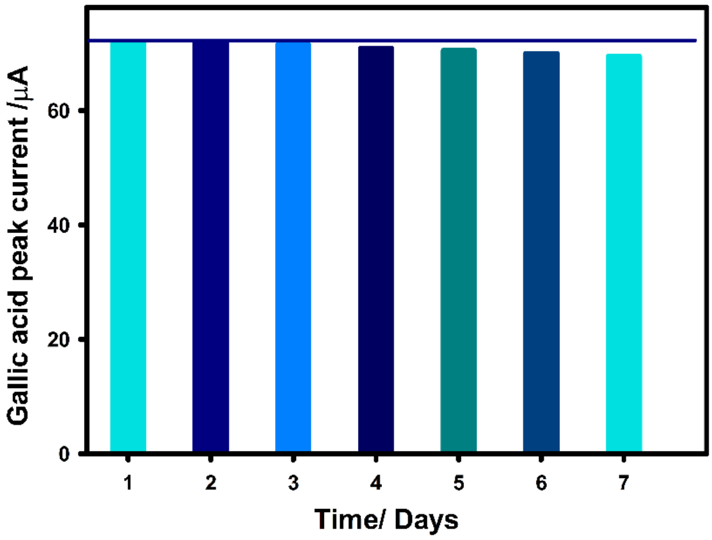

Figure 4. Bar graph for the stability of the TeOx-ZnOx/GO/GCE electrode over 7 days.

The determination of gallic acid in black tea was successfully carried out method and results were represented in Table 1. The recovery percentages obtained for the spiked samples clearly show the accuracy of the method. Each recovery experiment was performed in triplicate (n = 3), and the mean values are presented. For added concentrations of 10, 20, and 30 μmol L^{-1}, the recoveries were 104.6%, 101.2%, and 98.4%, respectively. These results confirm that the method is reliable. In addition, the low RSD (%) values indicate good repeatability of the measurements. Overall, the developed TeOx-ZnOx/GO/GCE electrode appears to be an applicable surface for gallic acid detection in real samples.

Table 1. Gallic acid determination in tea samples using the TeOx-ZnOx/GO/GCE electrode (n = 3).

Sample	Added (μmol L^{-1})	Found (μmol L^{-1})	Recovery (%)	RSD (%)
Black Tea	0	20.7	-	3.4
	10	32.12	104.6	3.2
	20	41.2	101.2	2.6
	30	49.9	98.4	2.4

4. Conclusion

In this study, a composite metal oxide modified–graphene oxide electrode (TeOx-ZnOx/GO/GCE) was successfully developed for the electrochemical determination of gallic acid. The combination of tellurium oxide and zinc oxide enhanced the electrocatalytic activity. TeOx facilitates charge transfer processes due to its semiconducting nature and favorable surface characteristic. ZnOx contributes to the structural stability of the electrode and provides additional active sites. GO supports the formation of active regions on the electrode surface through its large surface area and functional surface groups. As a result, the developed sensor exhibited high sensitivity, a low detection limit, and good stability. Its successful performance in real sample analysis using tea further demonstrated its practical applicability. Overall, the TeOx-ZnOx/GO modified electrode highlights the potential of composite metal oxide–graphene structures in the development of effective electrochemical sensors. Since the performance of the electrode has been evaluated only for gallic acid in tea matrices, this indicates a limitation in scope. Future studies focusing on different phenolic compounds and more complex real samples could further confirm the robustness of the sensor, keeping the platform promising for practical industrial applications

Ethics in Publishing

There are no ethical issues regarding the publication of this study

Author Contributions

The author was responsible for the investigation, conception, design, data collection, analysis, and interpretation of the research, as well as writing and editing the manuscript.

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