

Article

Silver Nanoparticle-Decorated Graphene Oxide Composite as a Non-Enzymatic Electrochemical Urea Sensor

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Abstract

Rapid and accurate urea detection is of considerable importance in environmental monitoring and biomedical analysis, as abnormal urea levels are associated with water contamination and various health conditions. In this study, a silver nanoparticle-decorated graphene oxide (Ag/GO) composite was synthesized via a simple chemical reduction method. The characterization results confirmed the successful formation of well-crystalline Ag nanoparticles (7.44 ± 1.46 nm) with uniform dispersion on GO, with a Ag loading of 39.1 wt%. The electrochemical performance for urea detection was evaluated in an alkaline medium (0.1 M NaOH) using cyclic voltammetry and chronoamperometry in a three-electrode system. The Ag/GO-modified glassy carbon electrode exhibited a strong electrocatalytic response toward urea oxidation, with a linear detection range of 1–10 mM. The sensitivity and limit of detection (LOD) were $36.8 \mu\text{A mM}^{-1}$ and 0.11 mM, respectively. The sensor also demonstrated excellent selectivity in the presence of common interfering species, including uric acid, ascorbic acid, and glucose, along with good reproducibility, repeatability, and stability. Furthermore, the practical applicability of the sensor was assessed in real samples, where satisfactory recovery was achieved in tap water, while reduced performance was observed in milk due to matrix effects. These findings indicate that the Ag/GO composite can serve as an effective alternative electrode material for non-enzymatic electrochemical detection of urea, particularly in wastewater and biological systems.

Keywords: silver nanoparticles; graphene oxide; non-enzymatic electrochemical sensor; urea detection



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1. Introduction

Urea detection plays a crucial role in both environmental monitoring and biomedical analysis, as abnormal urea levels can indicate water contamination and various health-related conditions [1]. For example, elevated urea concentrations in water may result from agricultural runoff or wastewater discharge [2], while abnormal levels in biological fluids are associated with renal dysfunction and metabolic disorders [3]. These implications highlight the critical need for analytical methods that are rapid, reliable, and highly sensitive

for accurate and timely detection [4,5]. Although conventional analytical techniques for detection, such as spectrophotometry [6], chromatography [7], and colorimetry [8], provide high accuracy, they are not suitable for real-time and on-site analysis because they require complex sample preparation, expensive instrumentation, and skilled personnel [9].

Electrochemical sensors have recently attracted considerable attention as an alternative approach for urea detection owing to their high sensitivity, rapid response, low cost, potential for miniaturization, and suitability for on-site application [10,11]. Most electrochemical sensors are based on enzymatic systems, in which urease catalyzes the hydrolysis of urea into ammonium and bicarbonate [12]. In such systems, detection is indirect and relies on monitoring the changes in the reaction products generated during enzymatic hydrolysis [13]. Although enzymatic sensors exhibit excellent selectivity, they suffer from two major limitations: (1) the immobilization process of urease on the electrode surface is complex and may reduce enzyme activity, and (2) enzymes are highly sensitive to environmental conditions, such as temperature, pH, and humidity, which restrict their practical applicability and long-term stability. To address these limitations, increasing attention has been directed toward non-enzymatic electrochemical sensors [14]. Unlike enzymatic systems, non-enzymatic sensors enable direct detection of urea through electrocatalytic oxidation at the electrode surface [15]. Therefore, the selection of electrode materials plays a crucial role in determining the electrocatalytic performance of non-enzymatic urea sensors.

Over the past decade, nickel-based materials (e.g., Ni [16], NiO [17], NiOOH [18], NiS [19], Ni-MOF [20], NiCo₂O₄ [21], etc.) have been extensively investigated as electrode materials for non-enzymatic urea detection due to their robust electrocatalytic activity in alkaline media. These materials readily form active redox couples (e.g., Ni²⁺/Ni³⁺), which are crucial for urea oxidation. In many studies, they have been combined with carbon-based supports, such as graphene [17], graphene oxide [19], and carbon nanotubes [20], to form composite systems. These composites enhance electrical conductivity, structural stability, and the availability of active sites for urea oxidation. In contrast, other classes of materials have received comparatively less attention, and the diversity of explored sensing materials remains limited. Therefore, the development of alternative electrode materials is highly desirable.

Among the various candidates, silver (Ag)-based catalysts have attracted growing interest for non-enzymatic urea oxidation [22] due to their favorable interactions with nitrogen-containing molecules and high electrical conductivity. In addition, Ag can undergo surface oxidation to form high-valence species that actively participate in the electrocatalytic process, thereby enhancing its electrocatalytic performance [23,24]. However, studies on Ag-based catalysts for urea detection have been less explored, particularly in real-sample applications, warranting further investigation.

In this study, we report a facile synthesis of the Ag nanoparticle-decorated GO (Ag/GO) composite via a chemical reduction method and its application as an electrode material for non-enzymatic electrochemical detection of urea. The GO was selected as the support material due to its large surface area, excellent dispersibility, and abundance of oxygen-containing functional groups that facilitate the anchoring of metal nanoparticles [25,26]. The morphology and microstructure of the composite were examined by field-emission scanning electron microscopy (FESEM) and field-emission transmission electron microscopy (FETEM), respectively. The Ag content was evaluated by energy-dispersive X-ray spectroscopy and thermal gravimetric analysis (TGA). Structural and chemical properties were analyzed by X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR), respectively. The electrocatalytic performance of the Ag/GO-modified glassy carbon electrode for urea detection was evaluated by electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), and chronoamperometry (CA) using a three-electrode system in 0.1 M sodium hydroxide

(NaOH). Furthermore, the practical applicability of the sensor was demonstrated through the detection of urea in real-world samples, including tap water and milk.

2. Results

2.1. Morphology and Microstructure

The morphology and microstructure of the Ag/GO composite were investigated using FESEM and FETEM analyses, respectively. The FESEM image of Ag/GO (Figure 1a) exhibits a wrinkled and stacked sheet-like morphology, which is characteristic of GO materials [27]. The GO surface appears relatively rough, with a uniform distribution of Ag nanoparticles (bright spots). The FETEM image (Figure 1b) further confirms the uniform dispersion of Ag nanoparticles (dark contrast) across the GO surface (bright contrast) without noticeable agglomeration. This suggests that the chemical reduction process effectively controls the nucleation and growth of Ag nanoparticles, thereby preventing abnormal growth. The inset image shows a magnified view of an individual Ag nanoparticle with clearly visible lattice fringes, confirming its high crystallinity. The particle size distribution (Figure 1c), as determined from the FETEM image, shows that the Ag nanoparticles have an average diameter of 7.44 ± 1.46 nm, indicating a relatively narrow size distribution. To further confirm the elemental composition and spatial distribution, EDS analysis was performed, as shown in Figure 1d. The EDS spectrum confirms the presence of carbon (C), oxygen (O), and Ag without detectable impurities, supporting the compositional purity of the Ag/GO composite. The EDS mapping analysis (Figure 1e) shows an even distribution of C, O, and Ag signals across the GO surface. Quantitative EDS analysis reveals that the composite consists of 58.9 at% (36.6 wt%) C, 34.6 at% (28.3 wt%) O, and 6.5 at% (35.1 wt%) Ag.

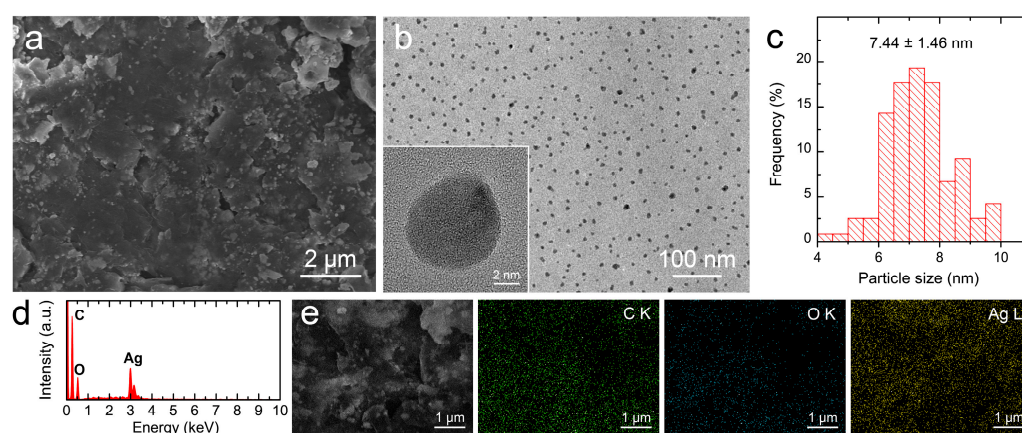


Figure 1. (a) FESEM and (b) FETEM images of the Ag/GO composite. The inset shows a magnified view of an individual Ag nanoparticle. (c) Particle size distribution histogram of Ag nanoparticles with an average diameter of 7.44 ± 1.46 nm. (d) EDS spectrum and (e) the elemental mapping images showing the distribution of C (green), O (blue), and Ag (yellow) in the Ag/GO composite.

2.2. Thermal Properties

TGA was used to evaluate the Ag loading in the Ag/GO composite. Figure 2 shows the TGA curves of bare GO and Ag/GO measured under an oxidative atmosphere from room temperature to 800 °C. Bare GO exhibits a three-step weight loss. The initial weight loss below 150 °C is attributed to the removal of physically adsorbed water. The second stage shows a significant weight loss between 150 and 300 °C, corresponding to the decomposition of oxygen-containing functional groups, such as hydroxyl, epoxy, and carboxyl groups. In the final stage, a further substantial weight loss occurs in the range of 400–500 °C, which is associated with the combustion of the carbon framework. Above 500 °C, GO is completely decomposed, leaving a negligible and constant residue (~ 2.2 wt% at 800 °C). This decomposition behavior shows a

trend similar to that reported in previous works [28,29]. In contrast, Ag/GO exhibits a similar decomposition profile but with a significantly higher residual weight. The residual weight at 800 °C remains approximately 40.4 wt%. This residual weight is attributed to thermally stable metallic Ag nanoparticles in the composite that do not decompose at high temperatures [30]. The Ag loading estimated from TGA (~39.1 wt% after subtracting the GO residue) is in good agreement with the EDS results.

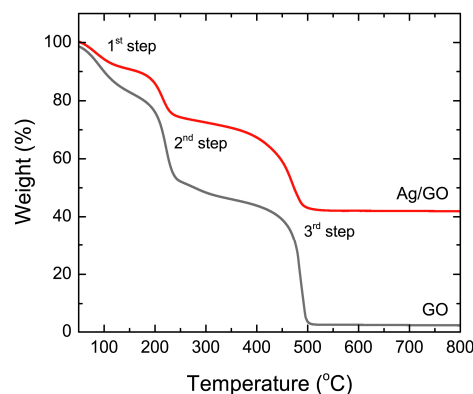


Figure 2. TGA profiles of GO and Ag/GO measured under an O₂ atmosphere.

2.3. Structural Properties

The crystalline structure and phase composition of GO and Ag/GO were examined by XRD, as shown in Figure 3a. The XRD pattern of GO exhibits a prominent diffraction peak at $2\theta \approx 10.5^\circ$, corresponding to the (001) plane of GO [31]. This feature is characteristic of oxidized graphite and indicates an expanded interlayer spacing (~0.84 nm), which is much higher than that of pristine graphite (~0.34 nm). The expansion is attributed to the introduction of oxygen-containing functional groups and intercalated water molecules during the oxidation process [32]. The absence of the graphite (002) peak ($2\theta \approx 26^\circ$) further confirms the successful oxidation of graphite into GO. For Ag/GO, several sharp diffraction peaks appear at 38.1° , 44.3° , 64.4° , and 77.4° , corresponding to the (111), (200), (220), and (311) planes of the face-centered cubic (fcc) structure of metallic Ag (ICSD card no. 01-071-6549), respectively. This result confirms the successful formation of crystalline Ag nanoparticles on the GO surface. Meanwhile, the GO (001) peak is significantly weakened or nearly disappears, likely due to the dominance of the strong Ag diffraction peaks [33,34]. No additional diffraction peaks are observed, indicating the absence of impurity phases and confirming high phase purity.

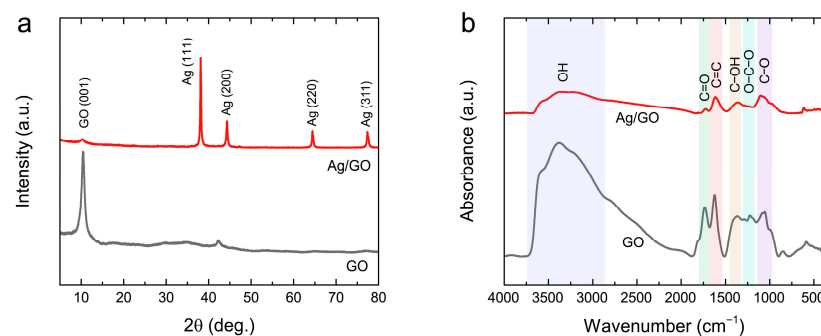


Figure 3. (a) XRD patterns and (b) FTIR spectra of GO and Ag/GO.

2.4. Chemical Functional Groups

The chemical structure and functional groups of GO and Ag/GO were investigated by FTIR (Figure 3b). For bare GO, several characteristic absorption bands are observed.

A broad peak centered around $\sim 3400\text{ cm}^{-1}$ is attributed to the stretching vibration of hydroxyl groups ($-\text{OH}$). The strong band at $\sim 1720\text{ cm}^{-1}$ corresponds to the $\text{C}=\text{O}$ stretching vibration of carboxyl groups, while the peak near $\sim 1620\text{ cm}^{-1}$ is assigned to the skeletal vibration of $sp^2\text{ C}=\text{C}$ aromatic bonds. Additionally, the peaks at 1367 , 1223 , and 1025 cm^{-1} are assigned to $\text{C}-\text{OH}$ stretching, $\text{C}-\text{O}-\text{C}$ stretching, and $\text{C}-\text{O}$ stretching (alkoxy/epoxy), respectively [35]. These results confirm the presence of abundant oxygen-containing functional groups and the successful oxidation of graphite. After the deposition of Ag nanoparticles, noticeable changes are observed in the FTIR spectrum of Ag/GO. The intensities of oxygen-containing functional groups are significantly reduced. This decrease can be attributed to the partial reduction of GO during the chemical reduction process using NaBH_4 [36,37], as well as the involvement of oxygen-containing functional groups in anchoring Ag nanoparticles onto the GO surface [38,39]. The remaining oxygen functional groups on the GO surface of the composite are beneficial for maintaining good dispersion and colloidal stability in aqueous solution, which is advantageous for electrode fabrication in sensor applications.

2.5. Electrochemical Analysis of Electrodes

The GO and Ag/GO were modified on glassy carbon electrodes (GCEs) for electrochemical measurements, which were denoted as GO/GCE and Ag/GO/GCE, respectively. The electrochemical performance of the GO/GCE and Ag/GO/GCE was first investigated by EIS in 0.1 M KCl containing $5\text{ mM } [\text{Fe}(\text{CN})_6]^{3-}/4-.$ Figure 4a presents the Nyquist plots of bare GCE, GO/GCE, and Ag/GO/GCE. The semicircle at high frequencies is associated with the charge transfer process at the electrode–electrolyte interface [40]. The fitted equivalent circuit is shown in the inset of Figure 4a. In this model, the diameter of the semicircle represents the charge transfer resistance (R_{ct}), while the intercept on the real impedance axis (Z_{real}) corresponds to the solution resistance (R_s) [41]. The bare GCE exhibits a relatively small semicircle with an R_{ct} value of $29\text{ k}\Omega$ (Figure 4b), indicating efficient electron transfer. In contrast, the GO/GCE shows a significantly enlarged semicircle, with a much higher R_{ct} value of $993\text{ k}\Omega$. This increase is attributed to the poor electrical conductivity of GO, which hinders electron transfer at the electrode surface [42,43]. Upon the incorporation of Ag nanoparticles, the R_{ct} of Ag/GO/GCE decreases markedly to $130\text{ k}\Omega$, demonstrating a substantial improvement in charge transfer kinetics. This enhancement can be ascribed to the high electrical conductivity of Ag nanoparticles and their role in providing conductive pathways and active sites for electron transfer [44,45]. These results clearly indicate that the introduction of Ag nanoparticles effectively improves the electrochemical properties of the GO-based electrode.

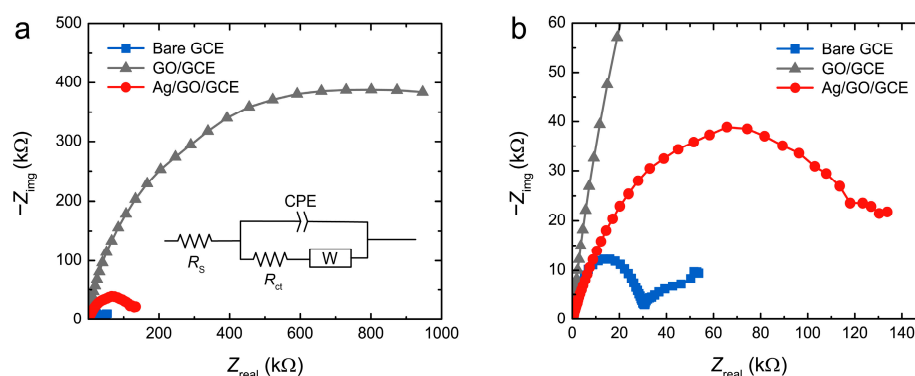


Figure 4. (a) Nyquist plots of bare GCE, GO/GCE, and Ag/GO/GCE measured in 0.1 M KCl containing $5\text{ mM } [\text{Fe}(\text{CN})_6]^{3-}/4-.$ The inset shows the fitted equivalent circuit used to determine the R_{ct} values. (b) Enlarged view of the Nyquist plots for bare GCE and Ag/GO/GCE.

2.6. Electrochemical Detection of Urea

The electrochemical performance of the Ag/GO/GCE toward urea detection was evaluated using CV in 0.1 M NaOH containing 10 mM urea, as shown in Figure 5a. During the forward scan, multiple anodic peaks are observed, corresponding to the stepwise oxidation of the metallic Ag surface. Initially, the surface of Ag is oxidized in an alkaline environment to form a silver hydroxide (AgOH) layer, followed by the formation of more stable monovalent silver oxide (Ag₂O) (Equations (1) and (2)) and subsequently divalent silver oxide (AgO) in the potential range of ~0.1–0.6 V (Equation (3)). At more positive potentials, further oxidation at ~0.8 V leads to the formation of silver oxyhydroxide species (AgOOH) (Equation (4)). These oxidized silver species collectively form an electrochemically active layer that significantly enhances catalytic activity. Among them, AgOOH-like species are generally considered the key active sites for urea oxidation, acting as redox mediators during the electrochemical process. The overall electrochemical reactions during the forward scan can be summarized as follows [46]:

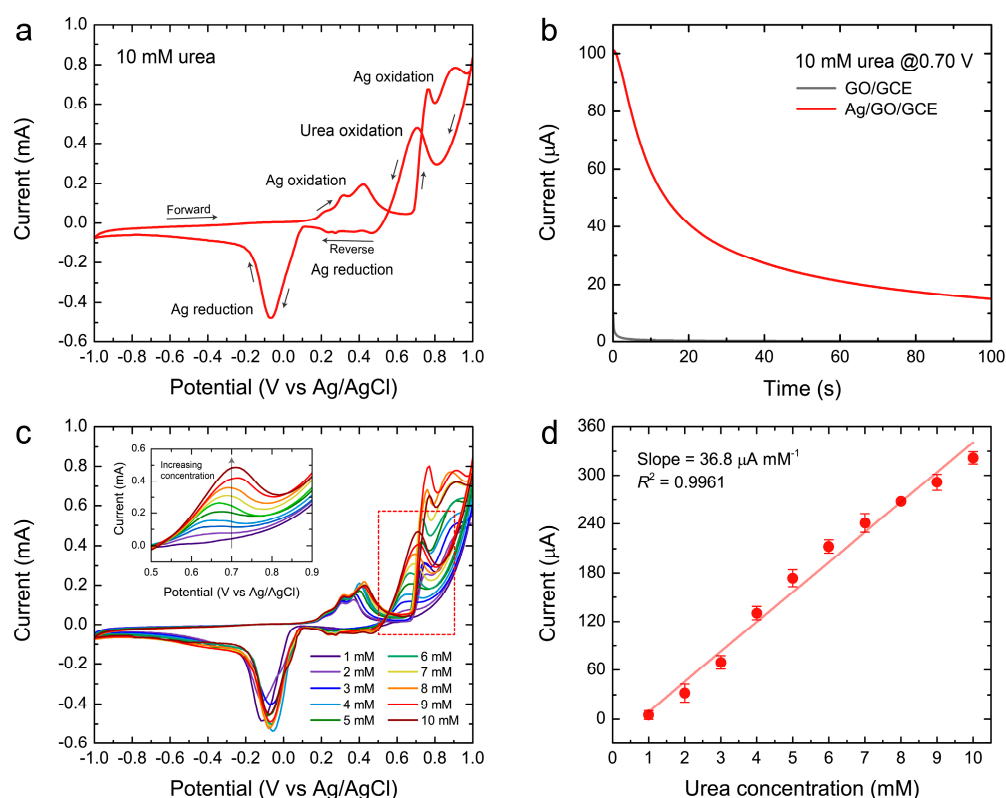
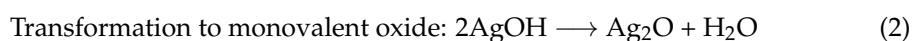
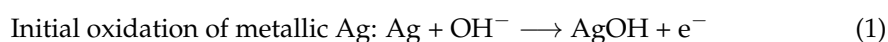
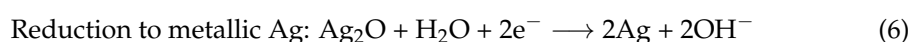
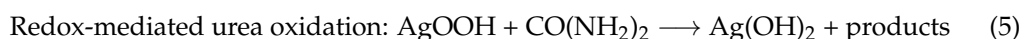


Figure 5. (a) CV curve of Ag/GO/GCE in 0.1 M NaOH containing 10 mM urea. The arrows indicate the potential scanning direction of the CV curve: forward scan (left to right) and reverse scan (right to left). (b) CA responses of GO/GCE and Ag/GO/GCE at an applied potential of 0.70 V in 0.1 M NaOH containing 10 mM urea. (c) CV curves of Ag/GO/GCE in 0.1 M NaOH with different urea concentrations (1–10 mM), with the inset highlighting the increase in urea oxidation current with increasing urea concentration. (d) Calibration curve of oxidation peak current as a function of urea concentration.

During the reverse scan, a distinct oxidation peak appears at 0.70 V, which is associated with the electrocatalytic oxidation of urea into N_2 and CO_2 , mediated by the surface AgOOH species (Equation (5)). Simultaneously, the oxidized silver species are reduced back to lower oxidation states (e.g., $\text{Ag}(\text{OH})_2$ or Ag_2O). This behavior is consistent with previous reports in which Ag-based catalysts facilitate urea oxidation in alkaline media through redox transitions of surface silver species [23,24]. In addition, the weak cathodic peaks appearing at ~ 0.1 – 0.6 V are attributed to the stepwise reduction of oxidized silver species, while a pronounced cathodic peak at -0.08 V corresponds to the Ag_2O reduction to metallic Ag (Equation (6)).



The CA measurements were performed at a constant potential of 0.70 V in 0.1 M NaOH containing 10 mM urea, as shown in Figure 5b. The Ag/GO/GCE exhibits a significantly higher current response than the GO/GCE, indicating the electrocatalytic activity toward urea oxidation imparted by the presence of Ag nanoparticles. A gradual decrease in current is observed over time, which is attributed to the depletion of urea molecules near the electrode surface during the electrochemical reaction [47].

The concentration-dependent electrochemical response was further investigated by recording CV curves at different urea concentrations ranging from 1 to 10 mM (Figure 5c). The oxidation peak observed at 0.70 V during the reverse scan was selected for quantitative analysis, as it corresponds to the electrocatalytic oxidation of urea mediated by AgOOH. This peak is well-defined and highly sensitive to urea concentration, making it suitable for calibration purposes. As the urea concentration increases, the oxidation peak current increases. This behavior can be attributed to the greater availability of urea molecules at the electrode surface, which enhances the interaction with the active AgOOH species and accelerates the catalytic reaction. The quantitative relationship between the peak current and urea concentration is presented in Figure 5d. A linear calibration curve was obtained over the concentration range of 1–10 mM. The sensitivity, determined from the slope of the calibration curve, was $36.8 \mu\text{A mM}^{-1}$, with a high correlation coefficient ($R^2 = 0.9961$). The good linearity indicates that the electrocatalytic process remains efficient and that the active sites are not saturated within this concentration range.

The limit of detection (LOD) of the Ag/GO/GCE for urea detection was calculated using the following equation [48]:

$$\text{LOD} = 3\sigma/S \quad (7)$$

where σ is the standard deviation of the blank signal ($1.39 \mu\text{A}$), and S is the sensitivity ($36.8 \mu\text{A mM}^{-1}$). The LOD was calculated to be 0.11 mM. The observed sensing performance of the Ag/GO/GCE can be attributed to several factors: (i) the high electrocatalytic activity of AgOOH species, which facilitates urea oxidation; (ii) the large surface area and good electrical conductivity of the GO support, which enhance electron transfer; (iii) the uniform dispersion of small-sized Ag nanoparticles, which provides a high density of accessible active sites; and (iv) the strong interaction between Ag nanoparticles and the GO matrix, which contributes to improved catalytic efficiency.

The sensing performance of the Ag/GO/GCE was compared with previously reported Ag-based sensors for non-enzymatic electrochemical detection of urea, as summarized in Table 1. The Ag/GO/GCE exhibits moderate LOD and sensitivity compared to some high-performance systems. However, it demonstrates a relatively wide linear detection range (1–10 mM), which is comparable to or broader than several previously reported sensors. This detection range is particularly relevant for practical applications, as it corresponds to

typical urea concentrations in wastewater and biological systems. In contrast, some sensors with low LODs operated within narrow or sub-micromolar ranges, which may be less suitable for direct analysis of real samples without dilution. Therefore, the Ag/GO/GCE provides a good balance between sensitivity and practical applications.

Table 1. Comparison of the electrochemical performance of the Ag/GO/GCE and Ag-based sensors reported in the literature for non-enzymatic electrochemical detection of urea.

Sensors	Technique	LOD	Sensitivity	Linear Detection Range	Reference
AgNP-coated test strip ^a	CV	0.14 mM	9.212 $\mu\text{A mM}^{-1}$	1–8 mM	[23]
EG-Ag-Z-Epoxy ^b	CV, CA, SWV	0.05 mM	30–65 $\mu\text{A mM}^{-1}$	0.2–1.4 mM	[24]
Ag-N-SWCNTs/GCE ^c	CV	4.7 nM	141 $\mu\text{A mM}^{-1} \text{ cm}^{-2}$	66 nM–20.6 mM	[49]
Ag/ZnO NR/carbon ^d	CA	13.98 μM	0.1622 $\mu\text{A } \mu\text{M}^{-1} \text{ cm}^{-2}$	26.3–427 μM	[50]
Ag/NiOOH/carbon paper ^e	CA	5 μM	233.7 $\mu\text{A mM}^{-1} \text{ cm}^{-2}$	0.2–26.0 mM	[51]
PANI-AgNP/SPE ^f	CV	0.021 μM	4.526 $\mu\text{A mM}^{-1}$	5–9 mM	[52]
Ag/GO/GCE	CV	0.11 mM	36.8 $\mu\text{A mM}^{-1}$	1–10 mM	This work

^a Silver nanoparticle-coated test strip. ^b Expanded graphite–silver-modified zeolite-epoxy composite. ^c Silver nanoparticles functionalized nitrogen-doped single-walled carbon nanotubes on a glassy carbon electrode. ^d Sputtered silver on zinc oxide nanorods grown on carbon paper. ^e Silver–nickel oxide hydroxide nanorods grown on carbon paper. ^f Polyaniline–silver nanoparticle nanocomposite on screen-printed electrode.

2.7. Reproducibility, Repeatability, Selectivity, and Stability Tests

Reproducibility, repeatability, selectivity, and stability are key parameters for evaluating the performance of electrochemical sensors. To assess reproducibility, CV measurements were conducted using five independently prepared Ag/GO/GCEs in 0.1 M NaOH containing 10 mM urea under identical conditions. The electrodes showed highly consistent current responses, with a relative standard deviation (RSD) of 2.71% (Figure 6a), indicating excellent reproducibility and precision. The repeatability of the Ag/GO/GCE was further evaluated by performing five consecutive CV measurements using the same electrode under identical conditions. The peak current remained highly stable throughout the measurements with an RSD of 1.84% (Figure 6b), demonstrating excellent repeatability. The selectivity of the Ag/GO/GCE was evaluated using CA measurements at a constant potential of 0.70 V (Figure 6c). Upon successive addition of urea, a sharp increase in current response was observed. In contrast, the successive introduction of common interfering species, including uric acid, ascorbic acid, and glucose, resulted in only a minor current change. The subsequent addition of urea again produced a significant increase in current, confirming the high selectivity and strong anti-interference capability of the sensor. The stability of the developed sensor was further evaluated by storing the Ag/GO/GCE under ambient conditions and measuring its current response toward 10 mM urea over time, as shown in Figure 6d. The sensor retained 91.3% of its initial current response after storage for 5 days, indicating good storage stability. Overall, these results confirm that the Ag/GO/GCE exhibits reliable electrochemical performance, including excellent reproducibility, repeatability, selectivity, and stability, highlighting its potential for practical non-enzymatic electrochemical urea sensing applications.

2.8. Urea Detection in Real Samples

To further assess the practical applicability of the Ag/GO/GCE, tap water and milk were selected as representative matrices for detecting 10 mM urea using CV measurements under identical conditions. As summarized in Table 2, the Ag/GO/GCE exhibited a recovery rate of 92.19% with a small RSD of 1.89% in tap water. This result falls within the generally accepted range for electrochemical quantification at this concentration [53], indicating satisfactory analytical performance. In contrast, a significantly lower recovery of 24.9% with a high RSD of 19.4% was observed in the milk sample, reflecting poor accuracy in a complex matrix. This behavior can be attributed to matrix effects arising from

coexisting ions, proteins, and organic components that interfere with the electrochemical response. Although the Ag/GO/GCE shows limited performance in milk, it demonstrates strong potential for urea detection in simple aqueous systems.

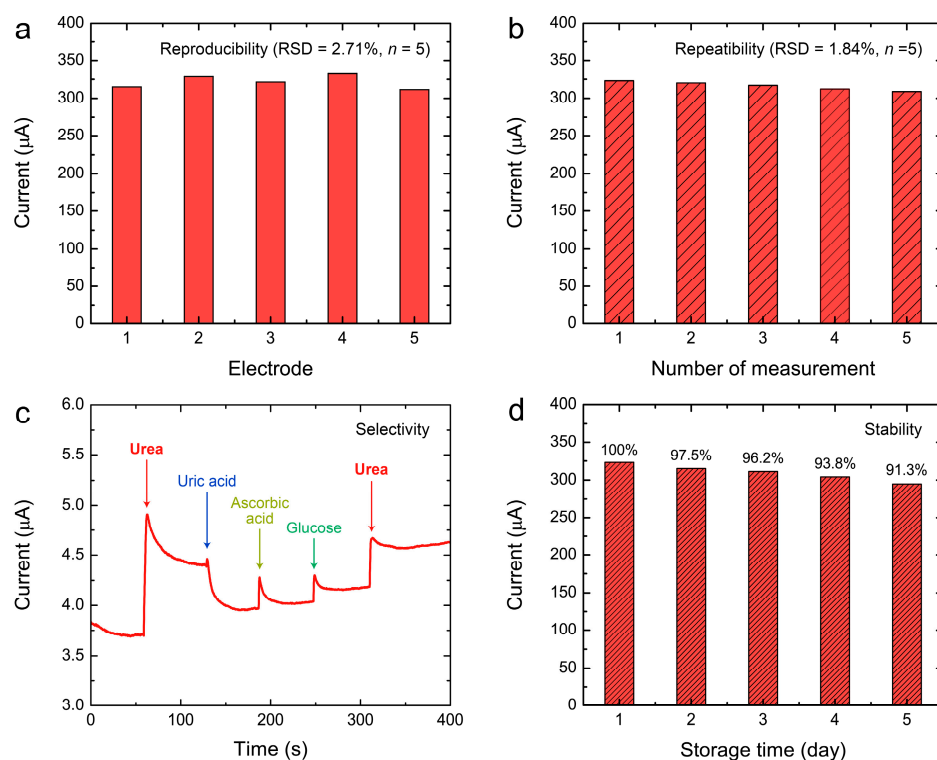


Figure 6. (a) Reproducibility, (b) repeatability, (c) selectivity, and (d) stability of the Ag/GO/GCE evaluated in 0.1 M NaOH containing 10 mM urea.

Table 2. Determination of urea in real samples (tap water and milk) using the Ag/GO/GCE.

Sample	Added (mM)	Found (mM)	RSD (% , n = 4)	Recovery (%)
Tap water	10	9.21	1.89	92.1
Milk	10	2.49	19.4	24.9

To broaden the applicable scenarios of this sensor, the Ag/GO/GCE can potentially be applied to millimolar-level urea detection in wastewater and simple biological systems. Urea concentrations in wastewater systems have been reported in the range of hundreds to thousands of mg L^{-1} , corresponding to the millimolar level. For example, an average urea concentration of 130 mg L^{-1} (2.2 mM) has been reported in dairy processing water [54], while values around 209 mg L^{-1} (3.5 mM) are commonly observed in municipal wastewater [55]. In addition, the typical urea concentration ranges in biological fluids are 2.5–7.5 mM, 1.2–3.3 mM, and 1.2–7.8 mM for human blood, urine, and saliva, respectively [10].

3. Materials and Methods

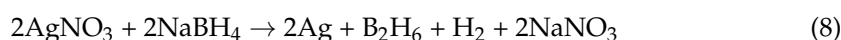
3.1. Materials and Reagents

Graphite powder ($<20 \mu\text{m}$), silver nitrate (AgNO_3 , $\geq 99.0\%$), and NafionTM 1100W were purchased from Sigma-Aldrich (St. Louis, MO, USA). Sodium borohydride (NaBH_4 , 98%) and uric acid ($\text{C}_5\text{H}_4\text{N}_4\text{O}_3$, $\geq 99.0\%$) were purchased from Thermo Fisher Chemicals (Waltham, MA, USA). Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$, $\geq 99\%$) and D-glucose, anhydrous, were purchased from KemAUS (Cherrybrook, NSW, Australia). Ethanol ($\text{C}_2\text{H}_5\text{OH}$, $\geq 99.9\%$), urea ($\text{CO}(\text{NH}_2)_2$, 99%), sodium hydroxide (NaOH , 99%), potassium chloride (KCl , $\geq 99\%$), and

acetonitrile ($\text{C}_2\text{H}_3\text{N}$, $\geq 99.7\%$) were obtained from RCI LabScan Ltd. (Bangkok, Thailand). Potassium ferrocyanide trihydrate ($\text{K}_4[\text{Fe}(\text{CN})_6] \cdot 3\text{H}_2\text{O}$, $\geq 99\%$) was supplied by DAEJUNG Chemicals & Metals Co., Ltd. (Gyeonggi-do, Republic of Korea). Ultrapure water (resistivity: $18.2 \text{ M}\Omega \text{ cm}$ at 25°C) was produced using a Millipore Direct-Q™ 5 UV water purification system (Bedford, MA, USA). Milk (Dutch Mill, Nakhon Pathom, Thailand) was purchased from a local shop. Tap water was supplied by the Metropolitan Waterworks Authority (Bangkok, Thailand). All chemicals and materials were used as received without further purification.

3.2. Synthesis of Ag/GO Composite via Chemical Reduction

GO was synthesized using a modified Hummers' method [56]. Subsequently, a Ag nanoparticle-decorated GO (Ag/GO) composite was prepared via the chemical reduction of AgNO_3 with NaBH_4 in an aqueous GO solution. Initially, 1 g of GO was dispersed in 1 L of AgNO_3 solution (4 mM) and sonicated to obtain a homogeneous suspension. The mixture was then stirred continuously in an ice bath for 1 h to maintain the reaction temperature. After that, a NaBH_4 solution (19 mL, 0.2 M) was added dropwise under vigorous stirring while maintaining the temperature below 5°C . During this process, the solution gradually turned grey, indicating the formation of Ag nanoparticles. The reaction mixture was further stirred overnight at room temperature to ensure complete reduction of Ag^+ ions and uniform deposition of Ag nanoparticles onto the GO sheets. The resulting product consisted of Ag nanoparticles uniformly decorated on GO. The overall reduction reaction can be expressed as follows [57]:



The resulting sample was thoroughly washed with deionized (DI) water to remove unreacted and residual species. The washed sample was then dried in a hot-air oven (Memmert UN55, Schwabach, Germany) at 80°C for 24 h, yielding the Ag/GO composite, which was subsequently used for characterization and electrochemical measurements. The synthesis procedure of the Ag/GO composite is schematically illustrated in Figure 7.

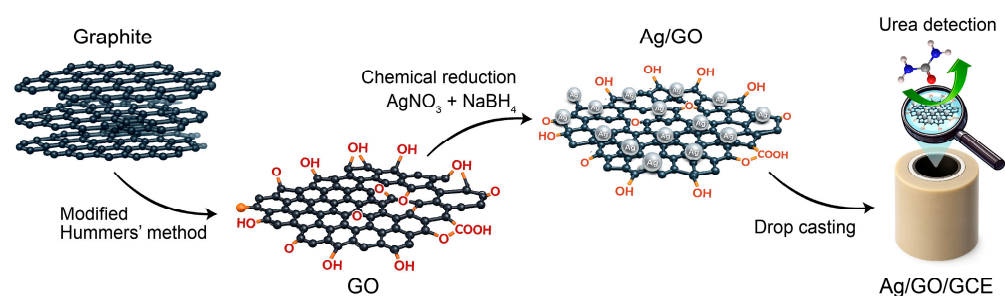


Figure 7. Schematic illustration of the synthesis of the Ag/GO composite as an electrode material for non-enzymatic electrochemical detection of urea.

3.3. Characterization

The surface morphology was examined using a JEOL JSM-7600F field-emission scanning electron microscope (Tokyo, Japan) operated at 5 kV. The dispersion of Ag nanoparticles on GO was analyzed using energy-dispersive X-ray spectroscopy (EDS) analysis using an Oxford Instruments AZtecOne spectrometer (High Wycombe, UK) equipped with a Hitachi SU3500 scanning electron microscope (SEM, Tokyo, Japan). The particle size and distribution of Ag nanoparticles on GO were further investigated using a JEOL JEM-3100F field-emission transmission electron microscope (Tokyo, Japan) operated at 300 kV. The Ag loading in the composite was estimated using a TGA/DSC 3+ Mettler–Toledo instrument

(Zurich, Switzerland). Samples were heated from 30 to 800 °C at a rate of 10 °C min^{−1} under an O₂ atmosphere. The crystalline structure was characterized by XRD using a Malvern Panalytical Empyrean diffractometer (Worcestershire, UK) with Cu K α radiation (λ = 1.5406 Å) over a 2 θ range of 5–80° at a scan rate of 4° min^{−1}. Chemical functional groups were analyzed by FTIR in transmission mode using a PerkinElmer Spectrum 3 Tri-Range spectrometer (Shelton, CT, USA). The spectra were recorded over the range of 4000–400 cm^{−1} with a scanning resolution of 4 cm^{−1} and accumulated over 64 scans.

3.4. Electrochemical Measurements

The catalyst ink was prepared by dispersing 1 mg of Ag/GO in a mixture of 475 μ L ultrapure water, 475 μ L ethanol, and 50 μ L Nafion, followed by sonication for 1 h to obtain a homogeneous suspension. The ink (5 μ L) was drop-cast onto a freshly prepared glassy carbon electrode (GCE, 3 mm diameter, ALS Co., Ltd., Tokyo, Japan). Prior to modification, the GCE was sequentially polished with 0.1 μ m diamond slurry and 0.05 μ m alumina slurry, rinsed with ultrapure water, and air-dried for 1 h. The Ag/GO-modified GCE is denoted as Ag/GO/GCE. The GO/GCE was also prepared using the identical process for comparison.

Electrochemical measurements were performed using a three-electrode system connected to a PalmSens4 potentiostat (PalmSens BV, Houten, The Netherlands) controlled by PSTrace 5.9 software. The Ag/GO/GCE served as the working electrode, while a platinum coil (ALS Co., Ltd., Tokyo, Japan) and Ag/AgCl (in saturated KCl) (ALS Co., Ltd., Tokyo, Japan) were used as the counter and reference electrodes, respectively. EIS measurements were carried out in a 0.1 M KCl solution containing 5 mM [Fe(CN)₆]^{3−/4−} as a redox probe at room temperature. The impedance spectra were recorded at the open-circuit potential (OCP) in the frequency range of 100 kHz to 10 mHz with a sinusoidal AC amplitude of 10 mV.

For the electrochemical detection of urea using the Ag/GO/GCE, CV was conducted over a potential range of −1.0 to 1.0 V at a scan rate of 100 mV s^{−1} in 0.1 M NaOH solution with urea concentrations ranging from 1 to 10 mM. The reproducibility of the Ag/GO/GCE was evaluated using CV measurements performed with five independently prepared electrodes under identical conditions. Each electrode was tested in a 0.1 M NaOH solution containing 10 mM urea. The peak current values obtained from the five electrodes were used to calculate the RSD. The repeatability of the Ag/GO/GCE was examined by conducting five successive CV measurements using a single Ag/GO/GCE in 0.1 M NaOH containing 10 mM urea. All measurements were carried out under identical conditions on the same day to minimize external variations. The RSD of the peak current responses was calculated to evaluate repeatability. The selectivity of the Ag/GO/GCE was investigated by CA measurements at a constant applied potential of 0.70 V in a 0.1 M NaOH solution. After achieving a stable baseline current, successive additions of 0.5 mM urea and potential interfering species, including uric acid, ascorbic acid, and glucose, were sequentially introduced into the electrolyte. The corresponding current responses were recorded to evaluate the sensor's anti-interference capability. In addition, the stability of the Ag/GO/GCE was assessed by storing the modified electrode under ambient conditions for 5 days. The electrochemical response toward 10 mM urea in 0.1 M NaOH was measured by CV each day, and the retained current response relative to the initial value was calculated.

For real sample analysis, tap water and milk were selected as representative matrices. Prior to analysis, tap water was mixed with 0.1 M NaOH at a ratio of 1:4 (*v/v*). Milk was mixed with acetonitrile at a ratio of 1:2 (*v/v*), followed by centrifugation at 10,000 rpm for 3 min to remove precipitated proteins. The resulting supernatant was collected and further mixed with 0.1 M NaOH at a ratio of 1:4 (*v/v*). The prepared tap water and milk samples were subsequently spiked with 10 mM urea prior to measurements. CV measurements

were then performed under the same conditions described above to evaluate the sensor performance in real samples.

4. Conclusions

In this work, a Ag/GO composite was successfully synthesized via a simple chemical reduction method. The characterization results confirmed the formation of well-dispersed, highly crystalline Ag nanoparticles on the GO surface, with an average particle size of 7.4 ± 1.46 nm and a loading of 39.1 wt%. The oxygen-containing functional groups on GO served as anchoring sites, facilitating the uniform dispersion and stabilization of Ag nanoparticles. The Ag/GO/GCE exhibited excellent electrocatalytic performance for the detection of urea in alkaline media. The sensing mechanism was governed by the redox cycling of Ag species, in which high-valence Ag species acted as the active catalytic centers for urea oxidation. The sensor demonstrated a high sensitivity of $36.8 \mu\text{A mM}^{-1}$ with a low detection limit of 0.11 mM, over a linear range of 1–10 mM. In addition, it exhibited excellent selectivity toward urea detection in the presence of common interfering species, along with good reproducibility, repeatability, and stability. This urea-detection performance is attributed to the synergistic interaction between Ag nanoparticles and the GO support, which increases the availability of active sites and facilitates efficient electron transfer. Furthermore, the uniform dispersion of small Ag nanoparticles and the GO network contribute to the overall catalytic efficiency. For the detection in real samples, although reduced performance was observed in complex matrices such as milk due to matrix effects, the sensor demonstrated satisfactory performance in water-based samples, highlighting its practical applicability. These findings demonstrate that Ag/GO/GCE is a simple and effective platform for non-enzymatic electrochemical urea sensing, with potential applications in wastewater and biological analysis.

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