

Article

Hydrogel-Based Electrodeposition of Copper Nanoparticles for Selective Detection for Hydrogen Peroxide

Jihun Han ^{1,†}, Jihyeon Kim ^{1,†}, Byung-Kwon Kim ^{2,*} and Kyungsoon Park ^{1,*} 

¹ Department of Chemistry and Cosmetics, Jeju National University, Jeju 63243, Republic of Korea; hanjihun@stu.jejunu.ac.kr (J.H.); harry9626@jejunu.ac.kr (J.K.)

² Department of Chemistry and Nanoscience, Ewha Womans University, Seoul 03760, Republic of Korea

* Correspondence: kimb@ewha.ac.kr (B.-K.K.); kspark895@jejunu.ac.kr (K.P.); Tel.: +82-2-3277-6578 (B.-K.K.); +82-64-754-3545 (K.P.); Fax: +82-2-3277-2385 (B.-K.K.); +82-64-756-3561 (K.P.)

† These authors contributed equally to this work.

Abstract: A copper-modified indium tin oxide (ITO) electrode was utilized as an electrocatalytic sensing platform for hydrogen peroxide (H₂O₂) detection. In this study, instead of conventional solution-phase electrodeposition, electrochemical deposition was used to deposit Cu on an ITO electrode with agarose hydrogel. The hydrogel-based Cu nanoparticles exhibited a more evenly dispersed distribution compared to those in the solution phase. Additionally, by incorporating agarose hydrogel as a solid electrolyte, the overall active surface area of Cu nanoparticles on the electrode surface was increased, mainly resulting from a decrease in the aggregation of Cu nanoparticles. Cyclic voltammetry and chronoamperometry confirmed that the resulting Cu nanoparticles possessed distinct electrocatalytic activity for H₂O₂ reduction and good selectivity for various interfering substances. The chronocoulometry response of the fabricated sensor obtained at −0.6 V (vs. Ag/AgCl) increased linearly with a dynamic range of 1–500 μM, and the limit of detection for H₂O₂ was 1.73 μM. Our research provides new possibilities for the electrochemical synthesis of metal nanoparticles for non-enzymatic sensing applications.

Keywords: nanoparticles; agarose; hydrogel; electrodeposition; electrocatalyst; H₂O₂ sensing



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1. Introduction

Metal nanoparticle-based electrocatalysts belong to a class of catalysts that enhance electrochemical reactions on electrodes by changing the reaction pathways and reducing the activation barrier. Recently, nanocomposites, especially metal nanoparticles, have drawn attention for their numerous advantages in catalysis, such as high surface area, improved mass transport, structural stability, and enhanced optical and electronic properties [1]. The fabrication of electrocatalysts with high selectivity, sensitivity, and stability remains a major research area in microelectronics [2,3], energy conversion [4,5], electroplating [6,7], and electrochemical sensing [8–11]. Among the various strategies, two classical methods can be applied to modify the electrode surface with metal nanoparticles: gaseous [12,13] or solution phase methods [14,15]. However, gaseous-phase deposition under vacuum, such as sputtering, vapor deposition, and chemical vapor deposition, may cause issues such as time consumption, expensive equipment, and trouble in controlling the properties of the synthesized nanoparticles. It is important to note that the electrocatalytic behavior of metal nanoparticles strongly depends on their intrinsic characteristics, such as size, morphology, density, and crystal structure [16,17]. Nanoparticles are generally formed in the solution phase by chemical synthesis. However, the harsh reaction conditions combined with toxic reducing agents and volatile organic solvents present major obstacles to their application [18–20]. To overcome these limitations, electrodeposition of metal nanoparticles on various electrode surfaces is a convenient and efficient alternative strategy [21,22]. Its major advantage is that the deposition process can be conveniently controlled using

electrodeposition parameters such as potential, electrolysis time, chemical composition, and electrolyte solution [23–25].

In our research, we utilized an ITO electrode as the primary electrode. The ITO electrode possesses high electrical conductivity, transparency, and excellent chemical stability, making it a commonly employed basic electrode in electrochemical sensors. Previous studies have shown that the catalytic ability of the ITO electrode itself is relatively limited, posing challenges in the sensitive detection of target materials using only the ITO electrode [26]. However, by introducing metal nanoparticles into the ITO electrode, we were able to effectively detect the current generated by the metal nanocatalyst on the ITO electrode [27]. Therefore, in our study, where copper nanoparticles were used, the ITO electrode proved to be a suitable sensing platform for achieving enhanced detection capabilities. Recently, our group reported on the use of silver nanoparticles as an electrocatalyst for hydrogen peroxide (H_2O_2) sensing [28].

In this study, agarose hydrogel was selected as the solid electrolyte for the electrodeposition of Ag nanoparticles instead of conventional solution media. Hydrogels, as three-dimensional polymers that absorb large amounts of water, have gained attention in the fields of drug delivery [29,30], tissue engineering [31], biomedical engineering [32,33], and other conventional biological applications owing to their superior biocompatibility [34]. In recent years, hydrogels have emerged as fascinating materials for energy and electrochemical applications owing to the employment of functional elements, including building block tailoring [35], functional additive integration [36], and cross-linker modification [37]. Hwang et al. have demonstrated an agarose hydrogel for a nanometer-scale electrochemical reaction by controlled contact area between a pyramidal-shaped hydrogel pen (HYPER) and an electrode surface. They introduce that the mass transport properties of redox molecules in agarose gel were similar to those in solution media, but only the diffusion coefficient was slightly decreased [38].

H_2O_2 is a strong reactive oxygen species (ROS) produced in living organisms during various metabolic processes. It plays a crucial role in cellular processes at low concentrations, but high concentrations can lead to oxidative damage and cell death. In addition, it also serves important functions in cell signaling and defense mechanisms. H_2O_2 is involved in the immune response against pathogens [39]. Consequently, rapid and selective monitoring of H_2O_2 may be of great interest in many research fields, including food safety [40], clinical [41], cosmetic [42], and biomedical analysis [43]. Many analytical methods for the determination of H_2O_2 have been developed, including titrimetry [44], spectrometry [45], fluorescence [46], and electrochemistry [47]. Among the wide range of analytical techniques available, electrochemical analysis proves to be a versatile tool for H_2O_2 detection owing to its speed, affordability, simple instrumentation, and high sensitivity [48]. Recently, various metal composites for H_2O_2 detection with high performance have been introduced [49–51]. Furthermore, metal nanoparticles such as electrodeposited Cu exhibit remarkable catalytic activity in the electrochemical determination of H_2O_2 [52].

In the first phase of this study, we present a method for the electrodeposition of Cu nanoparticles on ITO using agarose hydrogel as a solid electrolyte. In an earlier investigation, we presented several advantageous characteristics of agarose-based metal deposition, including the even and uniformly distributed particles and customizable attributes, when compared to traditional solution media [28]. Furthermore, this research confirmed that the electrochemical deposition of Cu using agarose hydrogel shows a significant increase in the active surface area owing to the well-distributed Cu particles. Based on this research, the electrocatalytic performance towards H_2O_2 reduction was evaluated by cyclic voltammetry (CV), chronoamperometry, and chronocoulometry (CC), whereas the electrodeposited Cu on ITO was examined using field-emission scanning electron microscopy (FE-SEM). Although aqueous electrolytes are commonly used media in electrodeposition, studies on hydrogel-based electrodeposition of metal nanoparticles are limited. Consequently, our research presents new possibilities for the electrochemical synthesis of metal nanoparticles in non-enzymatic sensing applications.

2. Materials and Methods

2.1. Chemicals and Materials

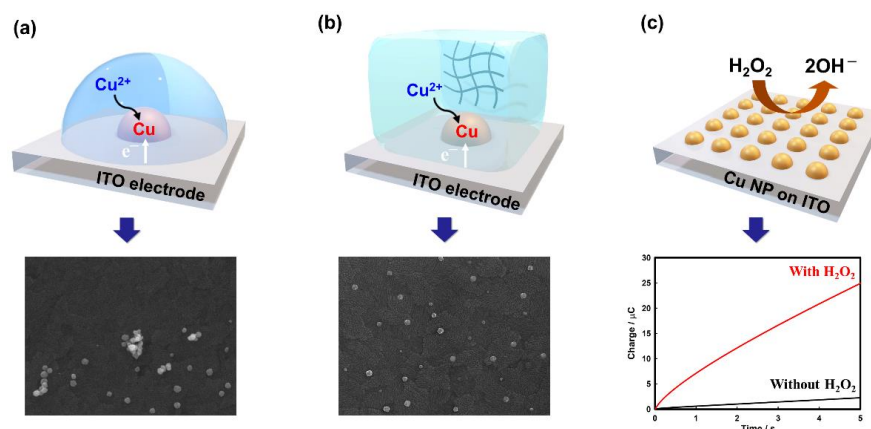
Agarose (Low EEO, purity $\geq 99\%$), CuSO_4 , K_2SO_4 , and H_2SO_4 were purchased from Sigma–Aldrich (St. Louis, MO, USA). H_2O_2 with purity $\geq 30\%$ was obtained from Fisher Chemical. All the chemicals and reagents were used as received, and ultra-pure water (resistivity $\geq 18.2 \text{ M}\Omega\cdot\text{cm}$) was prepared using a Millipore Milli-Q purification system. ITO-coated glass was obtained from Omniscience (Yongin-si, Republic of Korea, $10 \Omega \text{ cm}^{-2}$ sheet resistance). The ITO sheet was divided into $2.5 \times 2.5 \text{ cm}$ pieces for mounting in the electrochemical cell. Before use, the ITO underwent a thorough cleansing process involving a 5 min treatment in an ultrasonic bath with acetone and isopropanol, followed by drying under a stream of N_2 .

2.2. Preparation of Agarose Hydrogel for Electrochemistry

The agarose solution (3.2 wt%) was prepared by heating it in a heat-resistant container using a microwave (700 W power) until the agarose powder was homogeneously dissolved in water. Subsequently, the solution in its dissolved state was transferred into a vacuum desiccator in order to eliminate any trapped air bubbles within the hydrogel solution that had not undergone gelation. The final viscous and transparent agarose solution was poured into a flat glass mold and cooled gradually in a humidity-controlled chamber, allowing it to solidify. The agarose that had solidified was meticulously separated from the mold and subsequently divided into the desired dimensions with the use of a razor blade. The hydrogel was stored in distilled water to prevent it from drying. Before conducting electrochemical measurements, the separated agarose gels were immersed in a solution containing a supporting electrolyte and redox molecules for 12 h to maintain complete equilibrium. For electrochemical measurement, a redox molecule containing agarose gel was mounted in an electrochemical cell.

2.3. Electrochemical and FE-SEM Measurement

The electrochemical cell configuration for Cu deposition using agarose hydrogel and H_2O_2 sensing is illustrated in Scheme 1. A conventional three-electrode cell consisted of an ITO ($2.5 \times 2.5 \text{ cm}$) as the working electrode, an Ag/AgCl quasi-reference electrode (QRE), and a Pt wire counter electrode. Electrochemical analysis was performed using a potentiostat (CHI 601e, CH Instruments, Austin, TX, USA). To ensure conformal contact between the agarose hydrogel and the ITO electrode, a homemade cell equipped with a vertical press was employed. Field emission scanning electron microscopy (FE-SEM, TESCAN, MIRA III, 15 kV) measurements were performed to image the surface-modified Cu on the ITO substrate.



Scheme 1. Schematic representation and scanning electron microscopy (SEM) images of the Cu electrodeposition on ITO electrode (a) in solution, and (b) in agarose hydrogel. Schematic illustrations of electrochemical H_2O_2 sensing and measured signals are depicted in (c).

2.4. Electrodeposition of Cu on ITO and Electrochemical H_2O_2 Sensing

For the electrodeposition process, agarose gel containing 0.1 mM CuSO_4 , 0.1 M K_2SO_4 , and 0.5 M H_2SO_4 solution was introduced into the aforementioned cell configuration. To ensure clear contact between the ITO electrode and the agarose hydrogel, a press machine was used to apply constant pressure. A CV scan was performed in the potential range from 0.1 to -0.9 V (vs. Ag/AgCl QRE) at a scan rate of 50 mV/s. For the Cu deposition, a potential of -0.8 V was applied for 30 s by the amperometric method. The electrochemical sensing of H_2O_2 was obtained by performing CV and CC in an electrochemical buffer solution (EB; pH 7.4; 0.7 M NaCl and 0.1 M phosphate buffer) with various concentrations of H_2O_2 .

3. Results and Discussion

The electrodeposition of Cu on the ITO electrode was characterized using CV in solution and agarose hydrogel within the potential range from 0.1 to -0.9 V (Figure 1). In both solution and hydrogel, a pair of well-defined redox peaks related to the reduction of Cu ions and subsequent anodic stripping of Cu metal on the ITO substrate were observed. It can be seen that the onset potential of Cu deposition is observed around -500 mV, indicating an initial dominance of kinetic control. Furthermore, the onset potential of metallic ion reduction on a different substrate is usually higher than that between the same metal because of crystallographic substrate–metal misfit, except in the event of underpotential deposition (UPD) [19]. Consequently, a crossover between the cathodic and anodic current waves owing to the difference in the deposition and dissolution potential is observed in both electrodeposition processes. In the reverse scan, the anodic peak potential is observed at around a potential of -140 mV due to the oxidative dissolution of metallic Cu at the electrode surface.

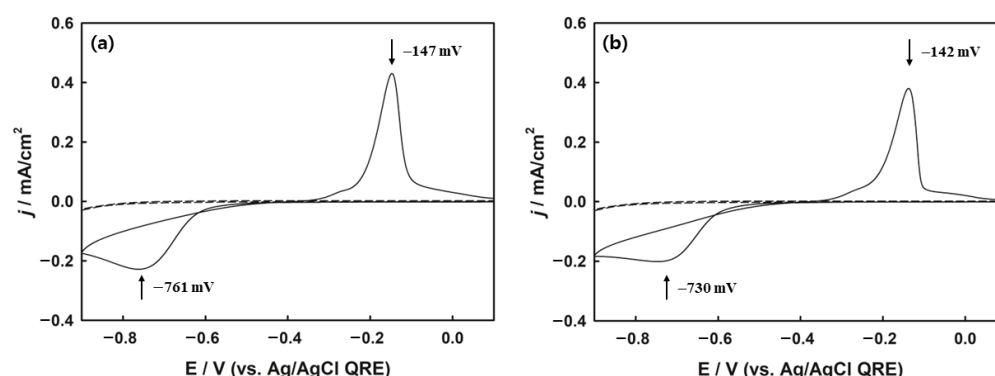


Figure 1. Cyclic voltammetry (CV) of Cu electrodeposition on indium tin oxide (ITO) electrode (a) in EB solution and (b) in hydrogel, both containing 1.0 mM CuSO_4 , 0.1 M K_2SO_4 , and 0.5 M H_2SO_4 at the scan rate of 50 mV/s. The dotted line indicates the background signal without CuSO_4 .

It is interesting to note that the cathodic peak potential (E_{pc}) of -730 mV in agarose hydrogel decreased compared to that of -761 mV in solution, whereas there was a negligible difference in the anodic peak potential in both cases. This suggests that Cu ions in agarose are more stable than in solution because of the higher interaction and stabilization between charged Cu ions and the hydrophilic functional group of the agarose polymer. By comparing the cathodic peak current (I_{pc}), it was found that the solution exhibited a higher I_{pc} of 0.227 mA/cm² compared to the agarose hydrogel media, which had an I_{pc} of 0.198 mA/cm². It is assumed that the hindered mass transport of Cu ions in agarose hydrogel is owing to steric hindrance and interaction between the redox probe and agarose polymer network [53].

The CVs of the Cu redox process with the variation of scan rate were obtained to understand the mass transport properties of Cu ions in the agarose hydrogel in more detail. Figure 2a shows an increase in both the cathodic and anodic currents as the scan

rate increased. Based on these results, as shown in Figure 2b, the cathodic peak current (I_{pc}) changed as a function of the square root of the scan rate (v) for Cu electrodeposition. The Randles–Sevcik equation is derived by assuming that the electroactive species (in the bulk solution) are only transported to the electrode by the diffusion process. According to these equations, a linear relationship between the cathodic peak current (I_{pc}) and the square root of the scan rate (v) revealed that the mass transport of Cu ions in agarose hydrogel is mainly controlled by the diffusion process without migration or convection [54]. Furthermore, a negative shift of the Cu cathodic peak potential was observed as the scan rate increased from 10 to 200 mV/s owing to the uncompensated iR drop (Figure 2c). Figure 2d shows the change in the cathodic peak potential (E_p) according to the log (scan rate, v) for Cu deposition onto ITO with agarose hydrogel. The electrodeposition of Cu onto ITO is an electrochemically irreversible reaction, as confirmed by the linear relationship between E_p and $\log v$. To determine the kinetics of the Cu deposition process on ITO, the coefficient of charge transfer (α) was calculated to be 0.411 at room temperature using the following equation [55,56].

$$|E_p - E_{p/2}| = 1.857RT/\alpha nF \quad (1)$$

where R is the ideal gas constant (8.314 J/mol·K), T is the temperature (K), n is the number of electrons in the rate-determining step, α is the charge-transfer coefficient, and F is the Faraday constant (96,485 C/mol).

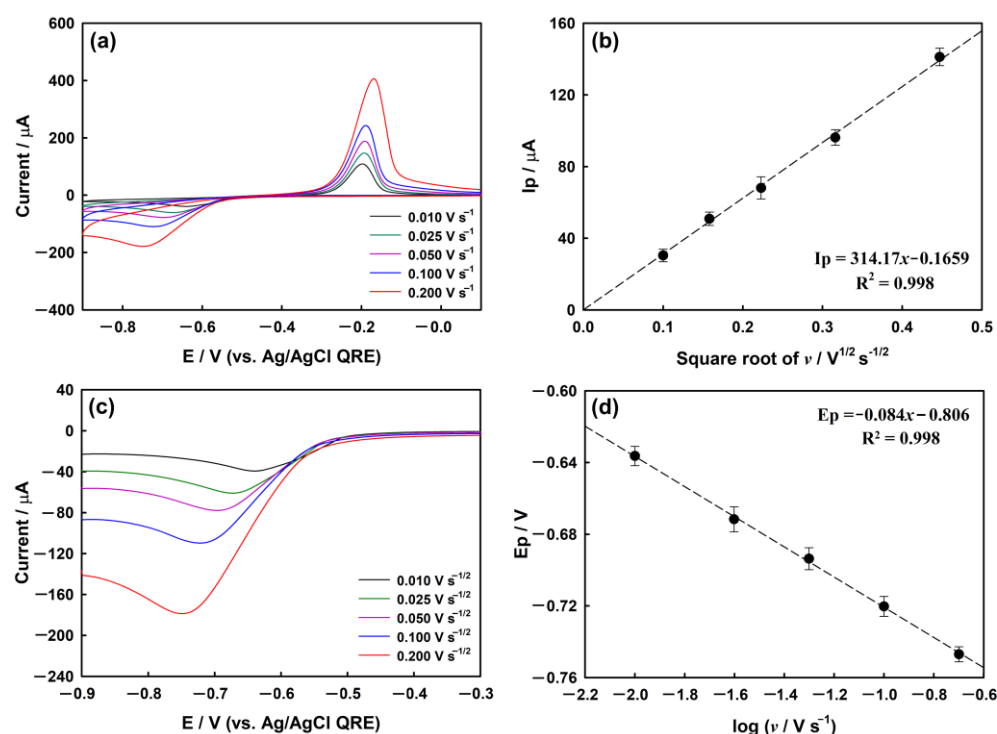


Figure 2. (a) CV and (c) LSV of Cu electrodeposition on ITO using agarose hydrogel containing 1.0 mM $CuSO_4$, 0.1 M K_2SO_4 , and 0.5 M H_2SO_4 depending on a scan rate. (b) Plots of cathodic peak current (I_{pc}) vs. square root of the scan rate (v) and (d) plots of cathodic peak potential (E_{pc}) vs. $\log v$ of (a). Error bars are obtained based on three independent measurements.

Figure 3 shows the surface morphology of Cu deposits on ITO under two different electrodeposition conditions: solution and agarose hydrogel. Our previous work showed that silver deposition in agarose hydrogel exhibited regular and well-dispersed nanoparticles compared to those obtained through solution-phase electrodeposition. It is assumed that the irregular transport of silver ions to the electrode can be suppressed by the polymeric network of the agarose hydrogel [53]. As depicted in Figure 2b, the Cu electrodeposition

through agarose hydrogel is also conducted via a diffusion process similar to Ag deposition. Therefore, it is crucial to prevent interference from factors other than diffusion, such as irregular and uncontrollable convection, in order to obtain well-dispersed Cu nanoparticles on the electrode. When metal nanoparticles are used as electrocatalysts, they serve as sites for electrochemical reactions involving electron transfer between the metal nanoparticles and the reactants in the solution. Generally, as the number of available active sites of metal nanoparticles increases, more reactants can be converted into products, leading to higher catalytic activity. Therefore, controlling the active area of nanoparticles, as well as their size, shape, and composition, is crucial to enhance their electrocatalytic ability. Figure 3a shows the SEM images of Cu nanoparticles obtained at -0.8 V (vs. Ag/AgCl QRE) for 30 s in solution, indicating an uneven density and agglomeration of particles. In contrast, Cu deposition in the agarose is relatively well distributed between each individual Cu particle, with a more regular shape (Figure 3c).

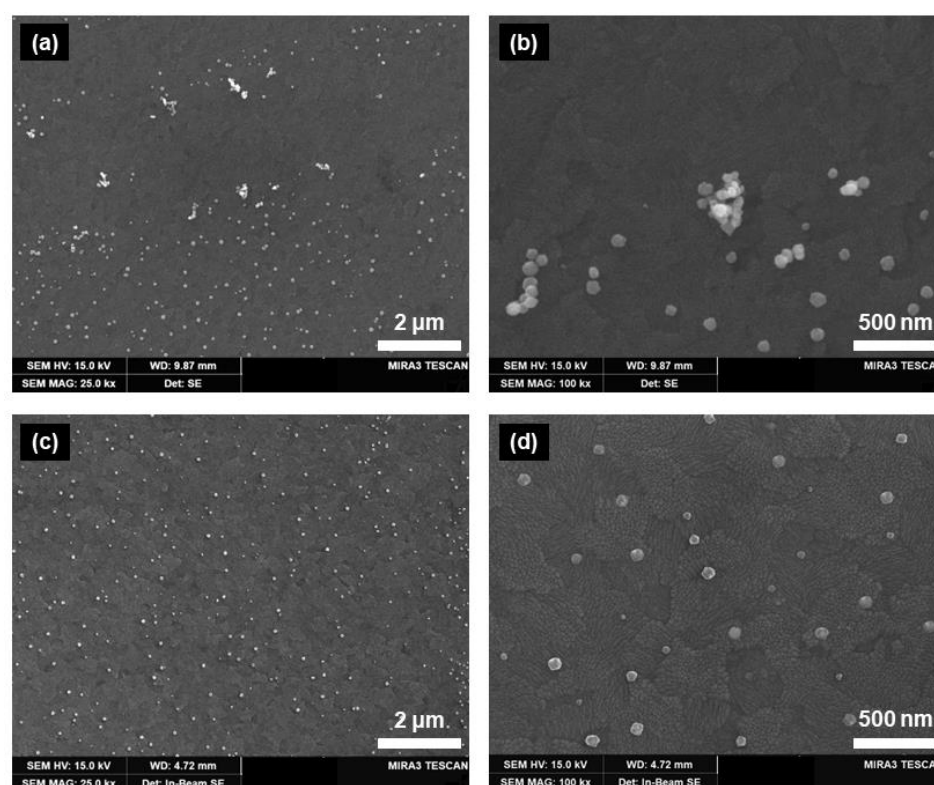


Figure 3. Scanning electron microscopy (SEM) images of Cu nanoparticles on ITO electrode (a) in solution, (c) in agarose hydrogel, both containing 1.0 mM CuSO_4 , 0.1 M K_2SO_4 , and 0.5 M H_2SO_4 at a potential of -0.8 V (vs. Ag/AgCl QRE) and deposition time of 30 s. (b,d) shows a high magnification of Cu particles of (a,c), respectively.

To compare the relative surface area of Cu nanoparticles on ITO obtained in a solution and agarose hydrogel, we adopted the method based on measuring the double-layer capacitance (C_{dl}) in a potential range where non-faradaic charge/discharge is the only electrochemical process. According to the literature [57], Cu does not absorb perchlorate anions (ClO_4^-). Therefore, in the presence of HClO_4 , the double-layer charge/discharge of H^+ is the only electrochemical process in the double-layer region. It can be seen in the inset of Figure 4 that the cyclic voltammograms were recorded in 0.1 M HClO_4 with a variable scan rate (0.01–0.08 V/s) within the capacitive region. A linear relationship between capacitive current at -0.1 V and scan rate was shown for both solution (Figure 4a) and agarose hydrogel (Figure 4b).

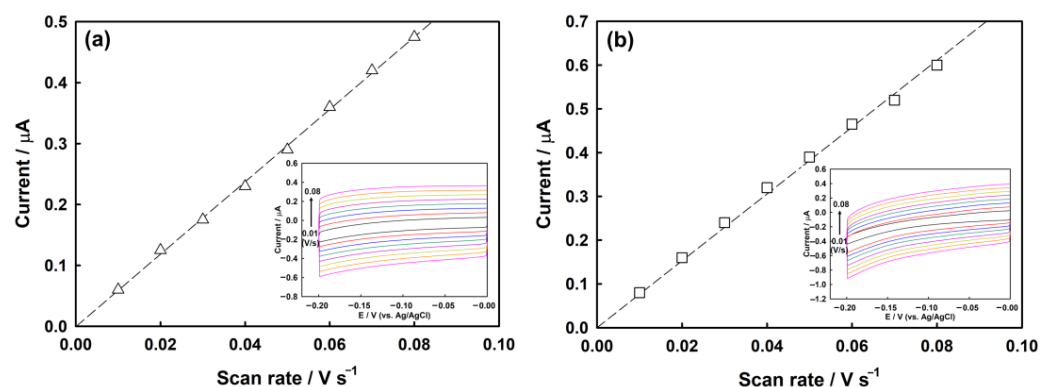


Figure 4. Plots of the capacitive current measured at -0.1 V vs. Ag/AgCl with the scan rate variation at Cu-modified ITO electrode (a) in solution, (b) in agarose hydrogel containing 0.1 M HClO₄. Inset: cyclic voltammograms of each electrode according to the scan rate in the same condition.

Using the following equation, the double-layer capacitance (C_{dl}) corresponding to the slope was calculated as $5.93 \mu\text{F}$ and $7.64 \mu\text{F}$ for the solution and agarose hydrogel, respectively.

$$I = C_{dl} (dV/dt) \quad (2)$$

As ascertained by the literature [58], the surface of a Cu electrode reveals a charge value of $28 \mu\text{F}/\text{cm}^2$ when measured in a 0.1 M HClO₄ solution. As a reference of this value, the active surface areas of the Cu electrodeposited in the solution and agarose hydrogel were calculated to be 0.211 cm^2 and 0.273 cm^2 , respectively.

These results indicate that agarose hydrogel-based electrodeposition may not only decrease the aggregation of Cu nanoparticles but also increase the number of active sites for electrocatalytic reactions compared to conventional solution-based electrodeposition.

To confirm the sensing possibility of Cu nanoparticle as an electrocatalyst, the electrocatalytic reduction of H₂O₂ on the ITO electrode was studied. Figure 5 shows the cyclic voltammograms of the ITO electrode modified with Cu nanoparticles, both before and after the addition of 5.0 mM H₂O₂ in a deaerated 0.1 M phosphate buffer solution (pH 7.4). Cu nanoparticles have been found to possess electrocatalytic ability in the reduction of H₂O₂ through the following two-electron reaction [56]:

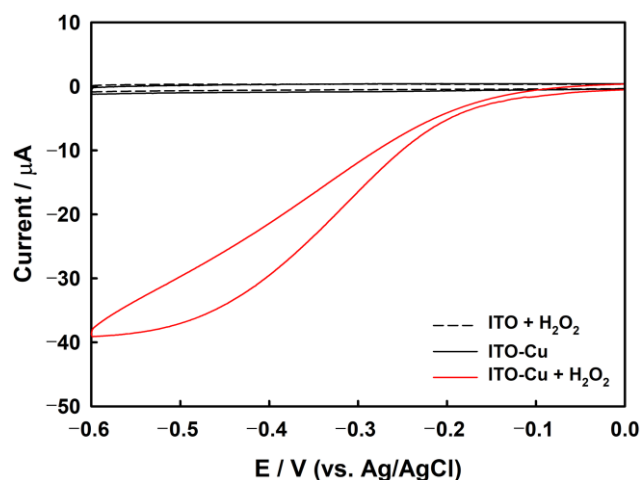
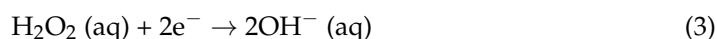


Figure 5. CV curves of the bare ITO (dotted line) and Cu nanoparticle-modified ITO in the presence (red line) or absence of 5.0 mM H₂O₂ in phosphate buffer (pH 7.4). Scan rate: 100 mV/s.

In the presence of H_2O_2 , the Cu-modified ITO electrode (red line) shows the highest catalytic reduction current peak of approximately $38 \mu\text{A}$ at -0.6 V . Meanwhile, both bare ITO with H_2O_2 (dotted line) and Cu-modified ITO without H_2O_2 (black line) demonstrate a weak current similar to the background signal. From the up to 100 cycles of CVs measurement, it was confirmed that the prepared Cu nanoparticles on the ITO electrode represent good stability for long-term electrochemical analysis (see Figure S1 in the Supplementary Material). These results indicate that the Cu-modified ITO electrode has good catalytic activity for H_2O_2 reduction, with the catalytic current mainly originating from the Cu nanoparticles on the electrocatalytic reduction of H_2O_2 .

Selectivity, among other features in sensing, is a crucial parameter for detecting a specific analyte in the presence of other interfering species. An ideal sensing condition is one that can differentiate the analyte from two or more interfering substrates. Recently, cubic silicon carbide (SiC) nanowires for nitrate detection with high sensitivity have been introduced [59]. To confirm the selectivity of our sensing system, the chronoamperometric response was measured in the presence of several possible electroactive interferences, such as nitrate, glucose, and ascorbic acid, as these are commonly known to interfere with the electrochemical detection of H_2O_2 . Figure 6 shows the amperometric results in a continuously stirred and deaerated phosphate buffer (pH 7.4) at -0.6 V vs. Ag/AgCl. A clear current response was observed upon the addition of 0.5 mM H_2O_2 , represented as a square in Figure 6. However, the introduction of various interfering substances resulted in a negligible current response compared with that of H_2O_2 . Subsequently, re-injecting 1.0 mM H_2O_2 into the solution with interfering substances showed a further stepwise increase in the current response. These results demonstrate that the sensing of H_2O_2 remains unaffected by these potential interfering substances, indicating the high selectivity of our sensor system.

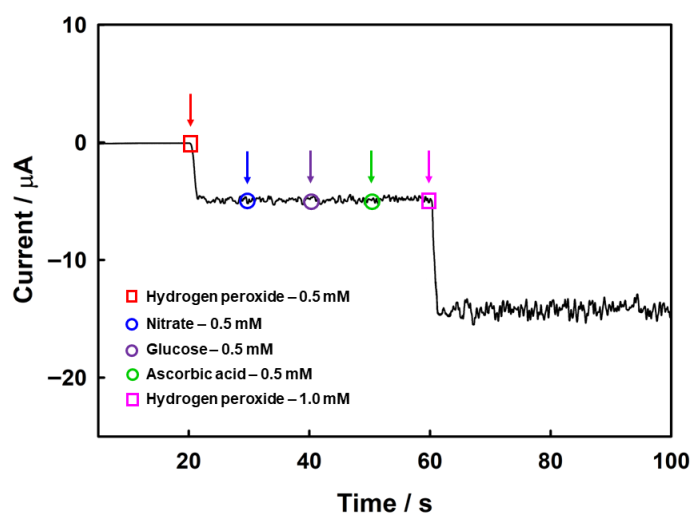


Figure 6. Amperometric behaviors observed from an ITO electrode modified with copper nanoparticles. The measurements were conducted in an Ar-saturated phosphate buffer solution, where the sequential addition of 0.5 mM interference molecules (nitrate, glucose, and ascorbic acid) and $0.5/1.0 \text{ mM}$ H_2O_2 as the target molecule.

CC measurements were performed to clarify the sensing performance of the fabricated Cu-modified ITO electrode in relation to the H_2O_2 concentration ranging between $1\text{--}500 \mu\text{M}$ (Figure 7a). The presence of a capacitive charging current poses a significant challenge in interpreting the sensing signal when employing potential sweep techniques like CV. However, in CC, the effect of the capacitive charging current can be significantly diminished by integrating the data over a specific time period [60]. In addition, the CC technique provides direct quantitative measurements of the charge transfer process, ensuring it is suitable for determining the amount of analyte present in a sample. These features make it particularly useful for electrochemical sensing and quantification applications. The

charge-time response increased with the H_2O_2 concentration owing to the electrocatalytic reduction of H_2O_2 on Cu nanoparticles. The standard deviation (SD) at 5.0 s in the 0 μM of H_2O_2 is 0.025 μC , and the sensitivity is 0.0434 $\text{C } \mu\text{M}^{-1}$. The calibration curve for the charge response at 5.0 s is shown in Figure 7b. The developed sensor system possesses a wide linear range, and the corresponding charge value represents an excellent linear relationship with the concentration of H_2O_2 owing to the high linear correlation coefficient (0.997). The estimated limit of detection (LOD) is ca. 1.73 μM using Equation (4).

$$\text{LOD} = 3S_b/m \quad (4)$$

where S_b is the SD of the background and m is the slope of the calibration curve. Based on these results, the hydrogel-based Cu-ITO electrode shows a relatively remarkable catalytic ability for H_2O_2 sensing in terms of high sensitivity and selectivity and a low LOD. The analytical performance of our sensor system is compared with that of other sensors based on metal nanomaterials in Table 1.

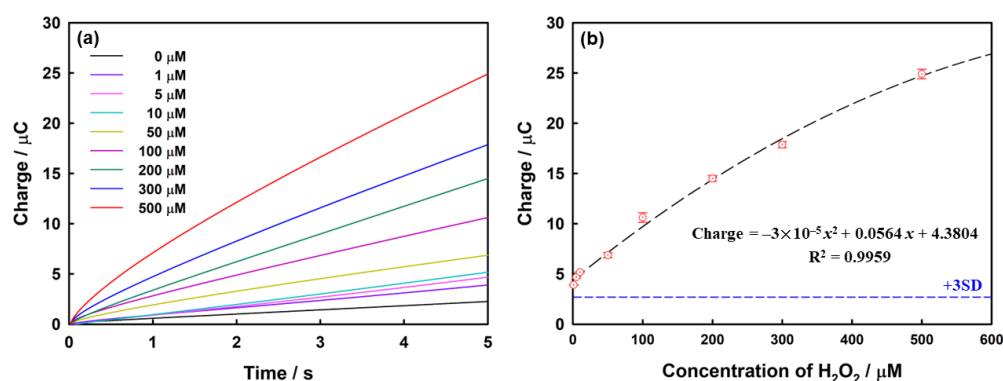


Figure 7. (a) The measured CCs at -0.6 V (vs. Ag/AgCl) with various concentrations of H_2O_2 in phosphate buffer (pH 7.4). (b) Calibration results for the charge values at 5 s in panel (a). The electrodeposition of Cu was conducted at -0.8 V for 30 s. All data were corrected using the average value of the double-layer charging current obtained from five measurements in the absence of H_2O_2 . The dotted line represents the three times SD value of the background current without H_2O_2 .

Table 1. The comparison of various nanomaterial-based sensors for non-enzymatic detection of H_2O_2 .

Sensor Type	Dynamic Range (μM)	LOD (μM)	Ref.
AgNPs@GNR/SPCE	50–5000	20	[61]
CuNPs@GCE	8–70	3.45	[62]
AgNPs@LSG	10–10,000	7.9	[63]
AgNPs/MoS ₂ @GCE	25–135,200	3.5	[64]
CoNPs/CNF@GCE	10–5000	10	[65]
PtNP/rGO-CNT/PtNP@SPCE	25–1000	4.3	[66]
Ag-exGRC-CI@SS	100–8000	5	[67]
Ag/H-ZIF-67@GCE	5–7000	1.1	[68]
CuNp@ITO	1–500	1.73	This work

P—nanoparticle, GNR—graphene nanoribbon, SPCE—screen-printed carbon electrode, GCE—glassy carbon electrode, LSG—laser-scribed graphene electrode, Co-NC—carbon particles containing Co nanoparticles, CNF—carbon nanofiber, rGO—reduced graphene oxide, CNT—carbon nanotube, Ag-exGRC—silver nanoparticle oxidized carbonate green rust, CI—carbon ink, SS—stainless steel electrode, H-ZIF-67—Co-containing zeolite imidazolate framework-67 obtained from pure H_2O .

4. Conclusions

The agarose hydrogel-based electrodeposition of Cu nanoparticles was successfully performed on the ITO electrode, and the fabricated electrode was used for the electrocatalytic determination of H_2O_2 . The Cu nanoparticles were not only more evenly dispersed on the electrode but also exhibited an increased active surface area compared to the nanopar-

tics synthesized in the solution phase. We have shown that these outstanding features can be used for the synthesis of various metal nanoparticles. Furthermore, the fabricated sensing platform showed excellent catalytic response toward H_2O_2 reduction and relatively better LOD ($1.73 \mu\text{M}$) compared to other metal nanoparticle-based electrochemical sensors. The prepared sensor system represents electrocatalytic activity toward H_2O_2 detection, but it did not respond to typical interfering substances such as nitrate, glucose, and ascorbic acid. Metal nanoparticles as electrocatalysts by hydrogel-based electrodeposition might offer new possibilities in the development of electrochemical sensor systems.

Supplementary Materials: The following supplementary material are available online at <https://www.mdpi.com/article/10.3390/chemosensors11070384/s1>. Figure S1: CV curves of Cu nanoparticle on ITO electrode containing $5.0 \text{ mM H}_2\text{O}_2$ in phosphate buffer (pH 7.4) after each of the 25 cycles. Scan rate: 100 mV/s .

Author Contributions: Conceptualization, K.P. and B.-K.K.; methodology, J.K. and J.H.; software, J.K., J.H. and K.P.; validation, J.K., J.H. and K.P.; formal analysis, K.P. and B.-K.K.; investigation, J.H., J.K., B.-K.K. and K.P.; resource, K.P. and B.-K.K.; writing—original draft preparation, K.P.; writing—review and editing, K.P. and B.-K.K.; supervision, K.P.; project administration, K.P. and B.-K.K.; funding acquisition, K.P. All authors have read and agreed to the published version of the manuscript.

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