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A Novel Nonenzymatic Hydrogen Peroxide Sensor Based on Electrocodeposition of Copper and Silicomolybdate on Multi-Walled Carbon Nanotubes

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Novel copper and silicomolybdate (SiMO) decorated multi-walled carbon nanotubes (Cu/SiMO/MWCNT) has been fabricated for the detection of hydrogen peroxide (H_2O_2). The Cu/SiMO/MWCNT can be prepared by the electrocodeposition of copper and silicomolybdate on a MWCNT modified electrode. As prepared hybrid composite was characterized by X-ray diffraction and scanning electron microscopy. The composite modified electrode shows good catalytic activity towards reduction of H_2O_2 with low overpotential (-0.16 V) and high sensitivity than that obtained using Cu, MWCNT, Cu/MWCNT, and Cu/SiMO modified electrodes. The maximum current response of the hybrid composite for H_2O_2 reduction was found at pH 1 than that of other pH. Amperometric response shows that the catalytic response of the composite was in the linear range of 6.53×10^{-6}–5.2×10^{-3} M with high sensitivity of 650 μA mM^{-1} cm^{-2} and response time of 5 s for the detection of H_2O_2. The detection limit was estimated as 6.53×10^{-6} M (S/N = 3). The Cu/SiMO/MWCNT composite electrode is not influenced by potential interferents and can be a good electrocatalyst for a nonenzymatic H_2O_2 sensor due to its low overpotential, high sensitivity, low detection limit, fast response, good stability, good selectivity, and simple method.

Keywords: Hydrogen peroxide, Sensor, Copper, Silicomolybdate, Carbon nanotubes

1. INTRODUCTION

H_2O_2 is one of the most important analytes and has been used in various fields including paper bleaching, food processing, textile industry, cleaning products and fuel cells [1–3]. Furthermore, H_2O_2 is well-known by product which generated from many enzyme-involved reactions, while it also plays an important role as a signalling molecule in regulating various biological processes [2]. Hence, accurate determination of H_2O_2 is more important and can received considerable attention to the research community. So far, electrochemical methods are widely used for the detection of H_2O_2 due to its convenience, sensitivity, selectivity, and immediate response.

Hemeproteins such as horseradish peroxidase, hemoglobin, myoglobin, cytochrome c and catalase are commonly utilized to fabricate H_2O_2 electrochemical biosensors, because these enzymes containing iron centered porphyrin can easily undergo the redox reaction, thus catalyzing the electrochemical oxidation and reduction of H_2O_2 [4, 5]. However, the instability of enzymes results in great difficulties for the assembly, storage and use of enzyme-based biosensors, especially in a complicated real environment. Therefore, increasing efforts are being made on the development of nonenzymatic H_2O_2 electrochemical sensors without standing properties [6–13].

Noble metals show noticeable catalytic activity to H_2O_2 reduction but their high price encourages developing low-cost and efficient electrocatalysts such as electrodeposited-copper [14]. Another approach is using the copper complex compounds [15–20] which are potential electrocatalysts for preparing carbon paste electrodes. A simple procedure is also proposed for preparation of copper complex $[Cu(bpy)_2]Br_2$ with silicomolybdate and single-walled carbon nanotubes [21], presenting a very low detection limit for H_2O_2.

In the present study, an effective nonenzymatic H_2O_2 sensor was developed for electrocatalytic reduction of H_2O_2. Electrocodeposition of copper and silicomolybdate was carried out using a bare and MWCNT-modified electrode. The Cu/SiMO/MWCNT hybrid composite was further for amperometric detection of H_2O_2. The sensitivity and selectivity of the sensor was further investigated in detail.

2. MATERIALS AND METHODS

2.1. Reagents and materials

Hydrogen peroxide (H_2O_2), silicomolybdate (SiMO), and multi-walled carbon nanotubes (MWCNT) were purchased from Sigma-Aldrich (USA) and used as received. All other chemicals (Merck) used were of analytical grade (99%). Double-distilled deionized water (> 18.1 MΩ cm^{-1}) was used to prepare all the solutions. All other reagents were of analytical grade and used without further purification.

2.2. Apparatus and measurements

The electrochemical experiments were conducted using a CHI 1205a electrochemical workstation (CH Instruments, USA) with a conventional three-electrode setup using the Cu/SiMO/MWCNT/GCE as the working electrode, an Ag/AgCl (3 M KCl) reference electrode, and a platinum wire counter electrode. Glassy carbon electrode (GCE) with a diameter of 0.3 cm was used for all electrochemical experiments. All electrochemical experiments were performed in an inert atmosphere.

2.3. Preparation of the Cu, SiMO, MWCNT, Cu/MWCNT, SiMO/MWCNT, Cu/SiMO and Cu/SiMO/MWCNT modified electrodes

The Cu and Cu/MWCNT modified electrodes were individually prepared in a nitric solution (pH 1.5) containing 0.5 M $Cu(NO_3)_2$ using a bare electrode and a MWCNT-modified electrode. By consecutive cyclic voltammetry, they were controlled in the potential range of -0.6–0.3 V with a scan rate of 0.1 Vs^{-1} and 30 scan cycles. The SiMO and SiMO/MWCNT modified electrodes were prepared by drop-casting of 10 μl of a nitric solution (pH 1.5) containing 1×10^{-3} M silicomolybdate was directly dropped on a bare electrode and MWCNT modified electrode. The resulting electrodes were further dried out in an oven at 40 ºC.

The electrocodeposition of copper and silicomolybdate was easily carried out in a nitric solution (pH 1.5) containing 0.5 M $Cu(NO_3)_2$ and 1×10^{-3} M silicomolybdate using a bare electrode and a MWCNT-modified electrode to prepare the Cu/SiMO and Cu/SiMO/MWCNT modified electrodes. By consecutive cyclic voltammetry, they were controlled in the potential range of -0.6–0.3 V with a scan rate of 0.1 Vs^{-1} and 30 scan cycles. Prior to the preparation of MWCNT-modified electrodes, all MWCNT were functionalized with carboxylic groups to confer good dispersion in the prepared solution. This MWCNT solution was drop-casted on the electrode surface to form a MWCNT-modified electrode. 10 μL of the MWCNT solution was used in this work to ensure coverage of the entire electrode surface. Next, the effluent from the effective surface area was carefully removed. The electrodes were cleaned and dried in an oven at 40 ºC. The MWCNT-modified electrodes (MWCNT/GCE and MWCNT/ITO) were easily prepared using this method. The Cu, SiMO, MWCNT, Cu/MWCNT, SiMO/MWCNT, Cu/SiMO and Cu/SiMO/MWCNT modified electrodes were individually prepared as the aforementioned processes and they were stored at room temperature before use.

3. RESULTS AND DISCUSSION

3.1. Characterisation of the Cu/SiMO/MWCNT hybrid composite

Fig. 1 shows the voltammograms of the Cu, SiMO and Cu/SiMO composites examined in pH 7 PBS. Curve (a) exhibits significant redox peaks at E_{pa} = -0.171 V and E_{pc} = -0.348 V, revealing to Cu(I)/Cu(II) redox process. Curve (b) exhibits redox peaks at E_{pa1} = 0.338 V and E_{pc1} = 0.058 V; E_{pa2} = 0.035 V and E_{pc2} = -0.096 V; E_{pa3} = -0.175 V and E_{pc3} = -0.360 V; revealing to $H_4SiMo_{12}O_{40}/H_6SiMo_{12}O_{40}$, $H_6SiMo_{12}O_{40}/H_8SiMo_{12}O_{40}$, and $H_8SiMo_{12}O_{40}$/H10SiMo$_{12}$O$_{40}$ redox processes [22]. However, the hybrid composite do not maintain the electrochemical characteristic peaks of SiMO. Curve (c) exhibits specific redox peaks at E_{pa} = -0.017 V and E_{pc} = -0.193 V which are different from curve (a) and (b). This result indicates that the Cu/SiMO composite shows more positive potential for the Cu(I)/Cu(II) redox process. This phenomenon might be caused by that the spherical silicomolybdate as a high dimensional template to load more copper resulted in compact Cu/SiMO composite.

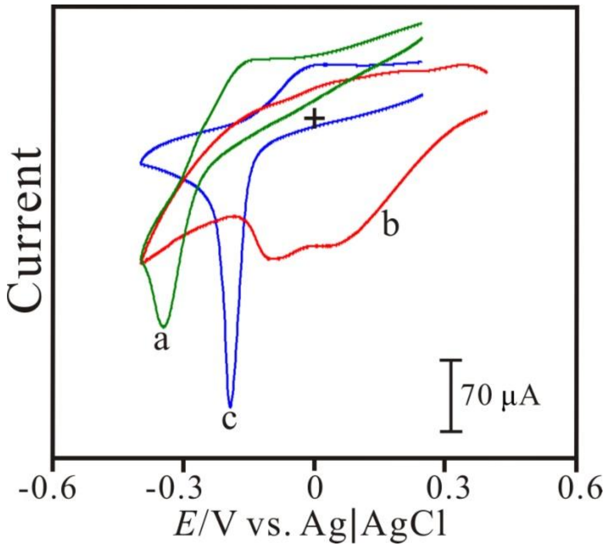

Figure 1. Cyclic voltammograms of (a) Cu, (b)SiMO, and (c) Cu/SiMO modified GCEs examined in pH 7 PBS solution. Scan rate = 0.1 Vs^{-1}.

Fig. 2 shows the SEM images for (A) Cu/ITO, (B) SiMO/ITO, (C) MWCNT/ITO, and (D) Cu/SiMO/MWCNT, respectively. Fig. 2A-D exhibits globular clusters, flat sheets, and fiber-like structures for (A) Cu, (B) SiMO, and (C) MWCNT, respectively. Particularly, Fig. 2D exhibits significant cocoon-like image with more uniform and smaller globular clusters and fiber-like structure. This result indicates that the Cu/SiMO can be successfully electrosynthesized on MWCNT due to the high conductive and steric MWCNT and static interaction between Cu and SiMO.

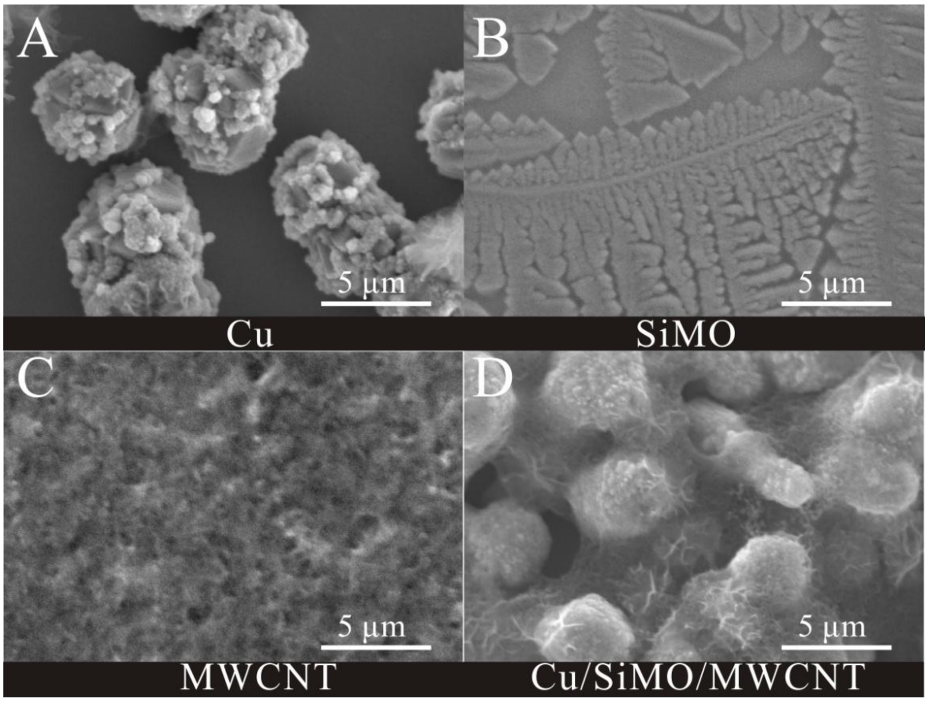

Figure 2. SEM images of (A) Cu, (B) SiMO, (C) MWCNT, and (D) Cu/SiMO/MWCNT modified ITO electrodes.

Fig. 3 shows the XRD patterns for (a) Cu/SiMO/MWCNT/ITO, (b) Cu/SiMO/ITO, (c) Cu/ITO, (d) SiMO/ITO, and (e) MWCNT/ITO. When the MWCNT was immobilised on ITO, it exhibited patterns (curve (e)) almost identical to that of ITO. This phenomenon might indicate the MWCNT is well dispersion and very thin on ITO surface. So that, the Cu/SiMO and Cu/SiMO/MWCNT coated on the ITO (patterns (a) and (b) of Fig. 3A) exhibit almost the same patterns with three characteristic peaks for Cu at $2\theta = 43.4°$, 50.6°, and 74.3°, corresponding to Miller indices (1 1 1), (2 0 0), and (2 2 0), respectively. There are also four characteristic peaks observed for Cu_2O at $2\theta = 36.7°$, 42.6°, 61.4°, and 73.9°, corresponding to Miller indices (1 1 1), (2 0 0), (2 2 0), and (3 1 1), respectively. Considering the SiMO/ITO patterns, which exhibits two characteristic peaks for SiO_x at $2\theta = 10.7°$ and 21.4°; and two characteristic peaks for MoOx at $2\theta = 25.8°$ and 39.0°, corresponding to Miller indices (0 0 2) and (1 0 2), respectively. One can know that both Cu/SiMO and Cu/SiMO/MWCNT show no SiMO patterns might be due to the core (SiMO)-shell (Cu) formation in the Cu and SiMO electrodeposition on bare and MWCNT-modified electrodes. It also can be used to explain why the the redox peaks of Cu/SiMO are different from those of Cu and SiMO. The Cu/SiMO hybrid composites can be easily electro synthesized by this method.

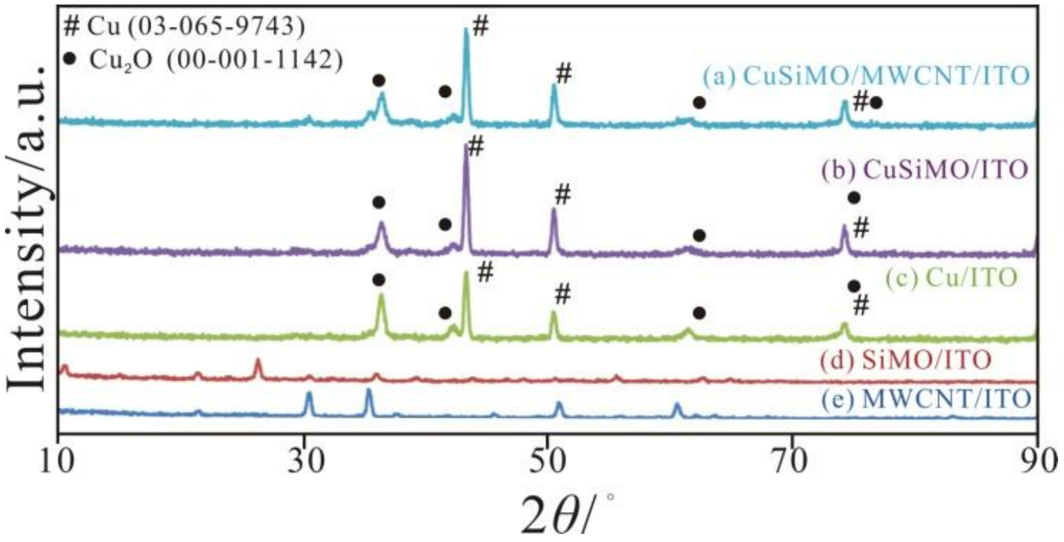

Figure 3. XRD patterns of (a) Cu/SiMO/MWCNT, (b) Cu/SiMO, (c) Cu, (d) SiMO, and (e) MWCNT modified ITO electrodes.

3.2. Electrocatalytic reduction of H_2O_2 at the Cu/SiMO/MWCNT electrode

Fig. 4 shows the voltammograms of Cu/SiMO/MWCNT/GCE examined in the absence/presence of H_2O_2. It shows high current response to H_2O_2 concentration. The cathodic peak current increases in the increase of H_2O_2 concentration. Inset of Fig. 4 shows the linear relationship between peak current and H_2O_2 concentration. It is noticed that the cathodic peak potential is shifted from -0.15 V to -0.25 V. This phenomenon indicates the high H_2O_2 concentration might cause obviously diffusion transfer effect results in peak shift in this concentration range.

The Cu/SiMO/MWCNT/GCE shows the lower overpotential (at E_{pc} = -0.15 V) for the electrocatalytic reduction of H_2O_2 in the presence of 1×10^{-4} M H_2O_2. The reduction potential is corresponding to a redox process for Cu(II)/Cu(I). This result is in good agreement with the previous work.21 As indicated in the literature, the reduction of H_2O_2 to O_2 was catalysed by the Cu(II)/Cu(I) redox process according to the following reactions:

$$\text{Cu(II)-composite} + e^- \rightarrow \text{Cu(I)-composite} \qquad (1)$$

$$\text{2Cu(I)-composite} + H_2O_2 + 2H^+ \rightarrow \text{2Cu(II)-composite} + 2H_2O \qquad (2)$$

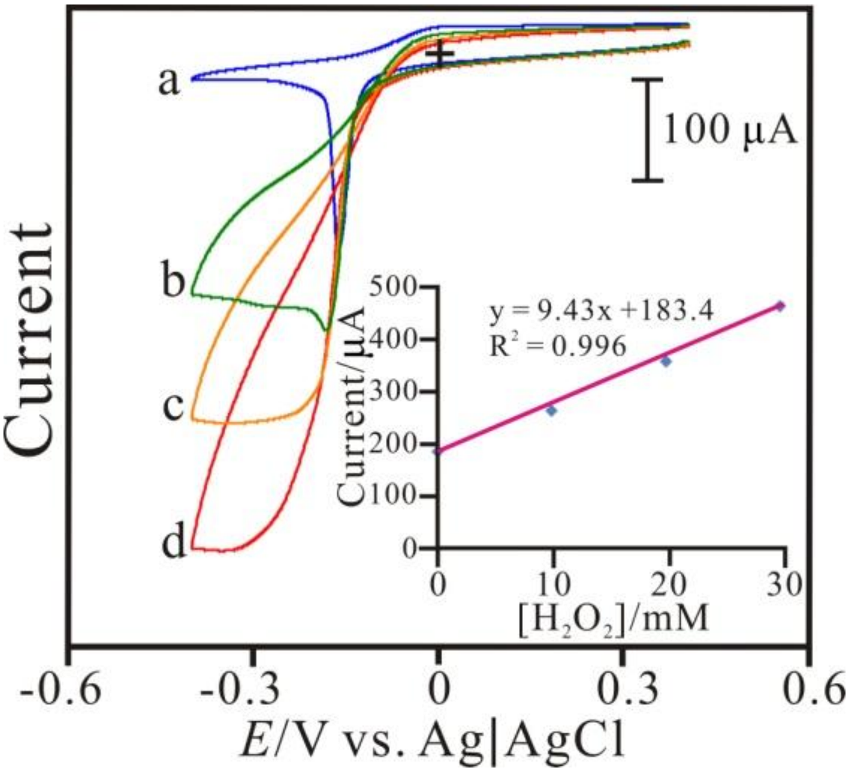

Figure 4. Cyclic voltammograms of Cu/SiMO/MWCNT/GCE examined in pH 7 PBS solution containing $[H_2O_2]$ = (a) 0, (b) 9.8×10^{-3} M, (c) 1.96×10^{-2} M, and (d) 2.94×10^{-2} M, respectively. Scan rate = 0.1 Vs^{-1}.

Table 1 shows the activity comparison of Cu/SiMO/MWCNT compared with related modifiers for electrocatalytic reduction of H_2O_2 in pH 7 PBS. It is also noticed that the Cu/SiMO/MWCNT shows much higher net cathodic current response (ΔI_{pc} = 213.4 μA mM^{-1}) which is 2.7–62.8 times greater than that obtained using Cu (ΔI_{pc} = 3.4 μA mM^{-1}), MWCNT (ΔI_{pc} = 15 μA mM^{-1}), Cu/MWCNT (ΔI_{pc} = 78.4 μA mM^{-1}), and Cu/SiMO (ΔI_{pc} = 44.6 μA mM^{-1}). The high electrocatalytic current response can contributed to the specific Cu/SiMO composite and further enhanced by MWCNT. As the results, the Cu/SiMO/MWCNT has fairly low overpotential and high current response, indicating that it is so active and can be the candidate for the electrocatalytic reduction of H_2O_2.

The electrocatlytic property of Cu/SiMO/MWCNT/GCE was studied with various pH conditions. The cathodic peak potential is significantly shifted to negative potential with the increase of pH conditions. The maximal net current response is found in pH 1 (ΔI_{pc} = 1463.6 μA mM^{-1}). This result indicates that the Cu/SiMO/MWCNT has better electrocatalytic activity for H_2O_2 reduction in strong acidic solution. Particularly, the second high net current response is found in the solution at pH 7 (ΔI_{pc} = 319.8 μA mM^{-1}). This phenomenon might be caused by the stability of the specific Cu/SiMO/MWCNT hybrid composite in neutral condition. This result also provides a chance for practical application to determine H_2O_2 in neutral condition without horseradish peroxidase.

Table 1. Activity comparison of Cu/SiMO/MWCNT compared with related modifiers for electrocatalytic reduction of H_2O_2 in pH 7 PBS.

Modifiers	$E_{pc}{}^{a}$/V (vs. Ag/AgCl)	$\Delta I_{pc}{}^{b}$/μA
Cu	-0.27	3.4
MWCNT	-0.30	15
Cu/MWCNT	-0.20	78.4
Cu/SiMO	-0.26	44.6
Cu/SiMO/MWCNT	-0.16	213.4

[a] The electrocatalytic reduction peak potential (E_{pc}) of different modifiers for H_2O_2 reduction.
[b] The net current (ΔI_{pc}) estimated at the electrocatalytic reduction peak in the absence/presence of 5×10^{-4} M H_2O_2.

3.3. Amperometric response of the Cu/SiMO/MWCNT electrode to H_2O_2

Fig. 5 shows the amperometric response of the Cu/SiMO/MWCNT electrode examined with several additions of 6.53 μM H_2O_2 into pH 7 PBS. The response is increasing with increasing the concentration of b H_2O_2, is due to the increasing amount of intermediate species is adsorbed onto the electrode surface, prolonging the reaction time. The calibration curve for amperometric current response vs. concentration of H_2O_2 is shown in the inset (b) of Fig. 5, which provides the regression equation, I_{pc}(μA) = 0.046C_{H2O2}(μM) + 1.7, with correlation coefficient of R^2 = 0.997. The fabricated sensor has a linear response range from 6.53×10^{-6} to 5.2×10^{-3} M with a sensitivity of 650 μAmM^{-1} cm^{-2} and a detection limit of 6.53 μM (6.53×10^{-6} M) (signal/noise = 3). A response time was found as 5 s.

Various nonenzymatic H_2O_2 sensors were compared with present sensor and comparative results are summarized in Table 2. The Cu/SiMO/MWCNT modified electrode shows superior sensitivity to those using Prussian blue, MWCNT, and other materials, such as noble metal-MWCNT hybrid composite. When compared to some noble metal-based hybrid composites such as Pt, Au, and Ag metals, the Cu/SiMO/MWCNT electrode also shows benefit in low cost. Good performance is clearly disclosed due to the specific Cu/SiMO decorating MWCNT on the electrode surface. The hybrid composite significantly increases the electrocatalytic active areas and promotes electron transfer in the reduction of H_2O_2. The fabricated sensor showed a high sensitivity, lower overpotential, low detection limit, wide linear concentration range than that of previously reported similar sensors, as shown in Table 2.

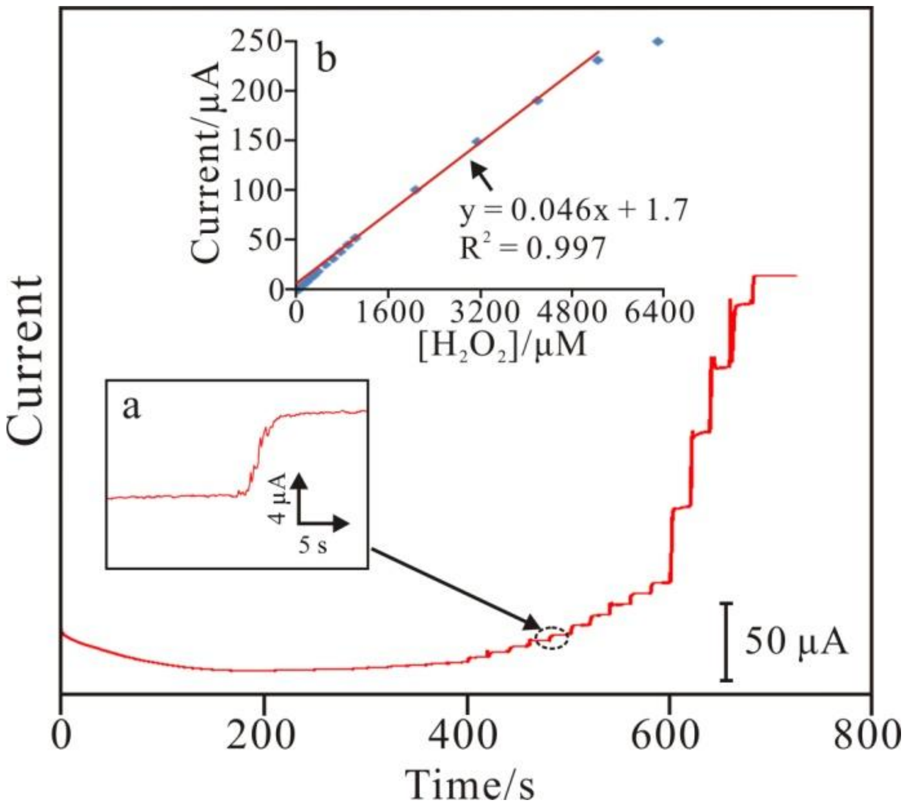

Figure 5. Amperograms of Cu/SiMO/MWCNT/GCE examined in pH 7 PBS containing [H_2O_2] = 6.53×10^{-6} ─ 6.26×10^{-3} M. $E_{app.}$ = -0.16 V. Electrode rotation speed = 2000 rpm. Insets are the scale-up amperogram and the calibration curve.

Table 2. Performance of the Cu/SiMO/MWCNT electrode compared with various nonenzymatic H_2O_2 sensors.

Modifiers	$E_{app.}$^{a} (V vs. Ag/AgCl)	Sensitivity (μA mM^{-1} cm^{-2})	LOD^b (μM)	Linear range (μM)	Ref.
PB/RTIL/CNTs	0.21	185.9	0.49	0.49–700	[23]
Ag-MnO_2-MWCNT	-0.3	82.5	1.7	5–10400	[24]
GO/PB	0.1	408.7	0.22	5–1200	[25]
PB@MWCNTs/Au nanochains/CS/Au	0	300	0.5	1.75–1140	[26]
PB/[Bmin][Cl]	0	225.2	─	5000–30000	[27]
PB/CA	-0.2	190	2.2	10–250	[28]
PtAu/G-CNTs	-0.47	313.4	0.6	2–8561	[29]
LDH-PB/PSS/PANI	-0.1	190	0.38	6–186	[30]
Nafion-PB-MWCNTs/ SPCE-IL	0	436	0.35	5–1645	[31]
Cu/SiMO/MWCNT	-0.16	650	6.53	6.53–5200	This work

^{a}$E_{app.}$ = Applied potential.
^{b}LOD = Limit of detection.

3.4. Reproducibility, stability, and anti-interference of the Cu/SiMO/MWCNT electrode

The reproducibility and stability of the sensor were evaluated. Five Cu/SiMO/MWCNT electrodes were investigated by amperometry ($E_{app.}$ = -0.16 V). The amperometric responses of the Cu/SiMO/MWCNT/GCE were obtained in pH 7 PBS with sequential additions of 5×10^{-4} M H_2O_2. The relative standard deviation (R.S.D.) was 3.2%, confirming the high reproducibility of the preparation method. Ten successive measurements of H_2O_2 on one Cu/SiMO/MWCNT electrode yielded an R.S.D. of 4.1%, indicating that the sensor was stable. The long-term stability of the sensor was also evaluated by measuring its current response to H_2O_2 within a 7-day period. The sensor was exposed to air, and its sensitivity was tested every day. The current response of the Cu/SiMO/MWCNT electrode was approximately 92% of its original counterpart, which can be mainly attributed to the chemical stability of Cu/SiMO in neutral solution. Based on these results, the Cu/SiMO/MWCNT electrode shows a nearly constant peak current towards H_2O_2 reduction, indicating that the electrode is very stable in the determination of H_2O_2.

Fig. 6 shows the amperometric response of Cu/SiMO/MWCNT/GCE in pH 7 PBS with sequential additions of H_2O_2 and potential interfering species such as glucose, galactose, fructose, sucrose, ascorbic acid, dopamine, and uric acid (1×10^{-4} M for each addition). A well-defined amperometric response was observed for the addition of H_2O_2, while the subspecies species does not show any apparent response at the same working potential. The result indicates that the Cu/SiMO/MWCNT composite electrode has high selectivity toward H_2O_2.

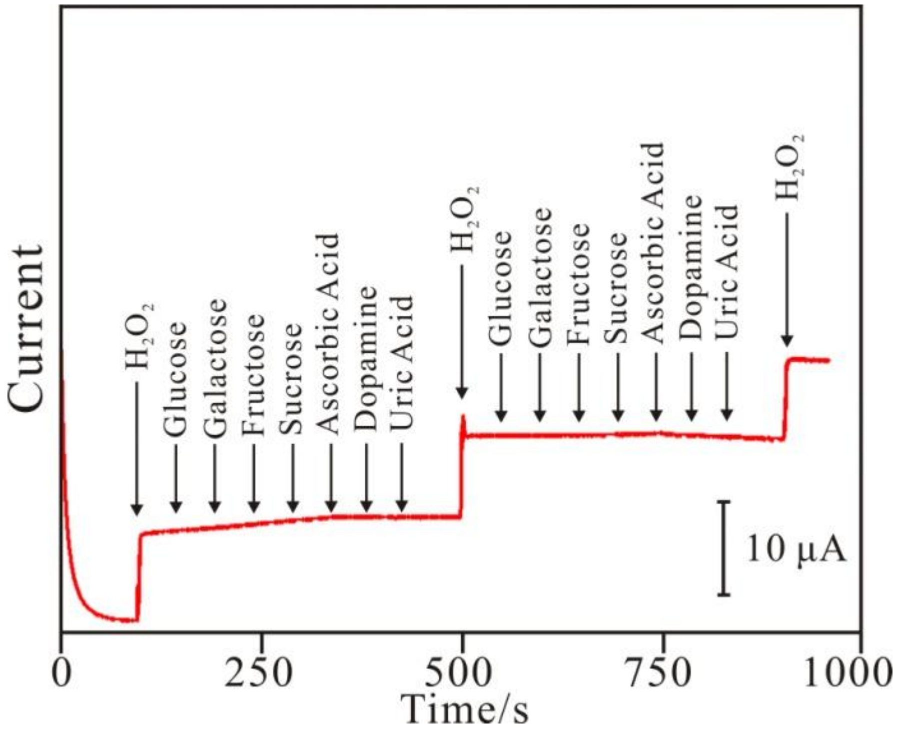

Figure 6. Amperograms of Cu/SiMO/MWCNT/GCE examined in pH 7 PBS with H_2O_2 and potential interferents: glucose, galactose, fructose, sucrose, ascorbic acid, dopamine, and uric acid (10^{-4} M for each addition). $E_{app.}$ = -0.16 V. Electrode rotation speed = 2000 rpm.

4. CONCLUSIONS

The Cu/SiMO composite has been successfully prepared on bare and MWCNT modified electrodes. The Cu/SiMO/MWCNT electrode showed attractive features towards the detection of H_2O_2, such as low overpotential, high sensitivity, low detection limit, fast response, good stability and good selectivity. The fabricated sensor can be used for selective sensing of H_2O_2 in real samples with high sensitivity.

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