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Original scientific paper

Highly sensitive voltammetric nanosensor based on Cu_2O nanocubes modified screen-printed carbon electrode for determination of 4-aminophenol

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Abstract

X-ray diffraction was used in this work to analyse the Cu_2O nanocubes prepared via a one-pot wet-chemical method. Next, 4-aminophenol (4-AP) was determined using a Cu_2O nanocube-modified screen-printed carbon electrode (Cu_2O/SPCE). Chronoamperometry, differential pulse voltammetry, and cyclic voltammetry were used to assess the electrochemical characteristics of 4-AP at the Cu_2O/SPCE sensor. As the redox peak currents increased, the results showed that the Cu_2O/SPCE exhibited strong electro-catalytic activity for 4-AP. With a limit of detection of 0.008 µM, the oxidation peak current under the chosen circumstances was proportional to the 4-AP in the range of 0.02 to 500.0 µM. With positive outcomes, the suggested approach was used to ascertain the 4-AP content of actual samples.

Keywords

Phenol derivative; electrochemical detection; nano copper oxide; real water samples

Introduction

4-aminophenol, often known as p-aminophenol (4-AP), is a non-opioid derivative of phenol. 4-AP is a hazardous substance that possesses both phenol and aniline toxicity. 4-AP serves as an essential fundamental industrial material that petroleum companies use together with rubber, dyestuffs, chemical inhibitors and organic chemical intermediates. A biomarker enables the evaluation of health status for employees who handle aniline because aniline absorption leads to its oxidation to p-aminophenol, which the body excretes as glucuronide and sulphate conjugates in urine [1-3].

The pharmaceutical industry depends on 4-AP for its important role. 4AP is a significant contaminant in medications containing paracetamol (PAR), a commonly used analgesic and antipyretic, as it forms when PAR breaks down. 4-AP has negative health consequences on humans, which include nephrotoxicity, teratogenicity, respiratory issues and irritation of the skin and eyes [4-7]. The development of easy-to-use, reliable 4-AP detection methods is needed.

Multiple methods exist to analyse 4-AP, which include capillary electrophoresis [8], spectrophotometry [9], chromatography [10], chemiluminescence [11] and fluorimetry [12]. Yet the methods require expensive equipment that takes extensive time to operate and demand advanced technical skills to manage.

The detection of different analytes and compounds has become more popular through electroanalytical techniques, which provide simple operation, accurate results, fast detection times, low expenses and high detection capabilities [13-16]. Screen-printed electrodes (SPEs) serve as electrochemical transducers and have gained popularity for their ease of handling, low equipment costs, portability, and high detection accuracy compared to traditional diagnostic instruments. SPEs can be produced from multiple materials, which include carbon ink, platinum and gold. The accessibility, low cost and simple operation of carbon-based SPEs make them an ideal material for developing sensing technologies [17-20]. However, using bare electrodes in electrochemical investigations has drawbacks for researchers, including limited electrochemical response and poor repeatability. Enhancing the analytical performance of electrochemical sensors is largely dependent on the electrode material [21-23]. High electrochemical catalytic activity and strong conductivity are usually requirements for excellent electrode materials [24-26].

The scientific community has devoted extensive research efforts to nanostructured materials because their superior performance stems from their minimal dimensions, extensive surface area, and unique material properties. The unique properties of size effects enable the creation of new technological solutions that scientists can implement across multiple fields. The high surface area of nanostructures enables researchers to create effective platforms for sensing and catalysis [27-31].

Cuprous oxide (Cu_2O) is a p-type semiconductor and a common transition-metal oxide. Cu_2O serves as the primary catalyst in electrochemistry because it offers low production costs and non-toxicity, and it displays decent catalytic performance and higher stability than precious metal catalysts. The material provides exceptional functional capabilities, which make it valuable for applications in sensors, solar cells and catalytic processes. The structural attributes of a material establish a critical foundation that determines its operational capacities [32-35].

The researchers used ascorbic acid as a reducing agent to synthesize Cu_2O nanocubes via a one-pot wet-chemical method. A Cu_2O nanocube-modified screen-printed carbon electrode (SPCE) electrochemical sensor was developed for the detection of 4-AP. The voltammetric sensor system built with CuO nanocrystals leveraged the enhanced properties of the nanomaterials, enabling precise detection limits and high performance across its detection spectrum.

Experimental

Chemicals and apparatus

All analytical-grade reagents were used exactly as supplied, without purification. To achieve varying pH values, 0.1 M phosphoric acid and a saturated sodium hydroxide solution were used to create a phosphate buffer solution (PB). The pH of the buffer solution, which served as the supporting electrolyte, was adjusted during the pH measurements using a 710 pH meter (Metrohm, Switzerland).

An Autolab potentiostat/galvanostat (PGSTAT 302N, the Netherlands) was used to conduct the electrochemical experiments, and GPES software (4.9) was used to control the instrument. Commercial carbon-based screen-printed electrodes (SPCEs) were acquired from Dropsens in Spain (model: DRP-110), and consisted of a working electrode (carbon), a pseudo-reference electrode (silver), and a counter electrode (carbon). A Panalytical X'Pert Pro apparatus (Netherlands) was used to analyse X-ray diffraction spectra using Cu–Ka radiation (λ = 0.1540 nm).

Synthesis of Cu_2O nanocubes

A standard process involved adding 5.0 mL of $CuCl_2{\cdot}2H_2O$ (0.1 M) and 15.0 mL of sodium hydroxide (0.2 M) to 200.0 mL of deionized water, followed by magnetic stirring for 5 minutes. The aforesaid solution was then mixed with 10.0 mL of an aqueous ascorbic acid solution (0.1 M). At room temperature, this combination was magnetically agitated for 60 minutes. After repeatedly washing the resulting precipitates with distilled water and ethanol, the Cu_2O nanocubes were dried for 12 hours at 60 °C.

Preparation of the Cu_2O nanocubes modified screen-printed carbon electrode

The initial Cu_2O nanocubes (1 mg) were dissolved in 1 mL of deionized water and sonicated for 45 minutes to form a homogeneous solution, thereby preparing the Cu_2O nanocube-modified SPCE. The SPCE surface was covered with two microliters of this suspension, and the solvent was allowed to evaporate at room temperature.

Electrochemical measurements

After transferring the modified SPCE into a PB, the oxidation signal of 4-AP was obtained using DPV or CV. Five modified SPGEs were prepared identically, and the average signal of 4-AP was obtained prior to each experiment for 4-AP determination.

Result and discussion

Characterization of Cu_2O nanocubes

Figure 1 shows the XRD pattern of the Cu_2O nanocubes. According to JCPDs No. 01-078-2076, the diffraction peaks in the Cu_2O XRD pattern at 2θ = 29.6, 36.5, 42.3, 61.4, 73.5 and 77.4.3° may be indexed as (110), (111), (200), (220), (311) and (222) to a cubic structure of Cu_2O.

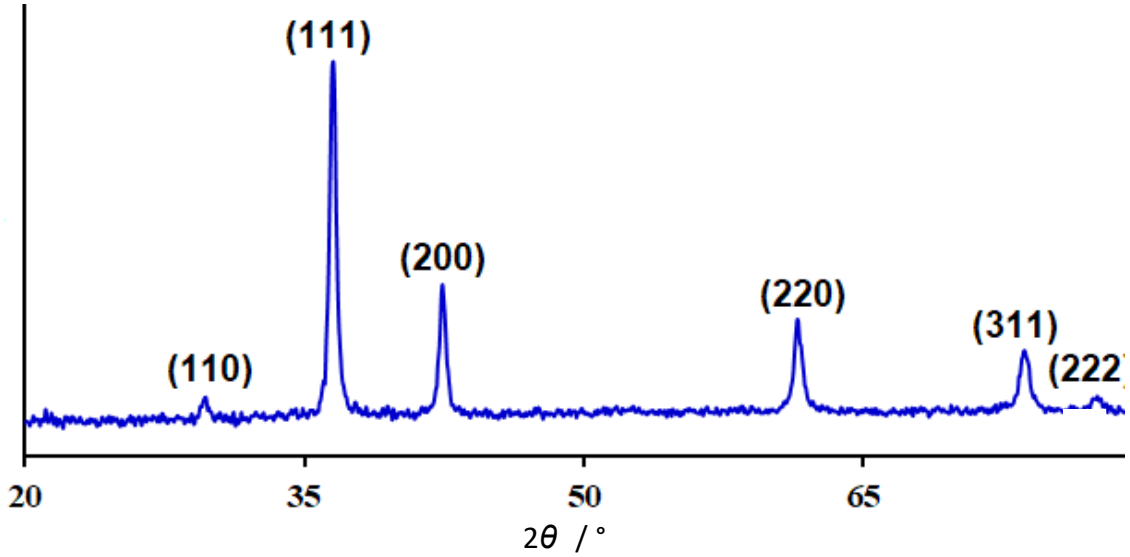

Figure 1. *XRD pattern of Cu_2O nanocubes*

Electrochemical profile of 4-AP at the Cu_2O/SPCE

The pH of the solution significantly affects the performance of electrochemical sensors. Thus, differential pulse voltammetry (DPV) was used to examine the effect of pH on the electrochemical behaviour of 50.0 µM 4-AP in 0.1 M PB over the pH range 2.0-9.0. The findings show that as pH rises from 2.0 to 7.0, the oxidation peak current (I_{pa}) of 4-AP increases, but above pH 7.0, I_{pa} decreases. Thus, pH 7.0 yields the highest peak current and is therefore selected for the upcoming studies to increase the sensitivity of the Cu_2O/SPCE sensor.

The CVs of 100.0 µM 4-AP on the surfaces of bare SPCE and Cu_2O/SPCE in 0.1 M PB at pH 7.0 are shown in Figure 2. The findings show that the redox reaction of 4-AP is reversible, as anodic and cathodic redox peaks of 4-AP with acceptable symmetry are clearly observed at both the bare SPCE and the Cu_2O/SPCE. Furthermore, compared to the bare SPCE (4.54 µA), the oxidation peak current of 4-AP at Cu_2O/SPCE (8.94 µA) is noticeably higher. These findings show that Cu_2O nanocubes are a superior electrocatalyst for the oxidation of 4-AP.

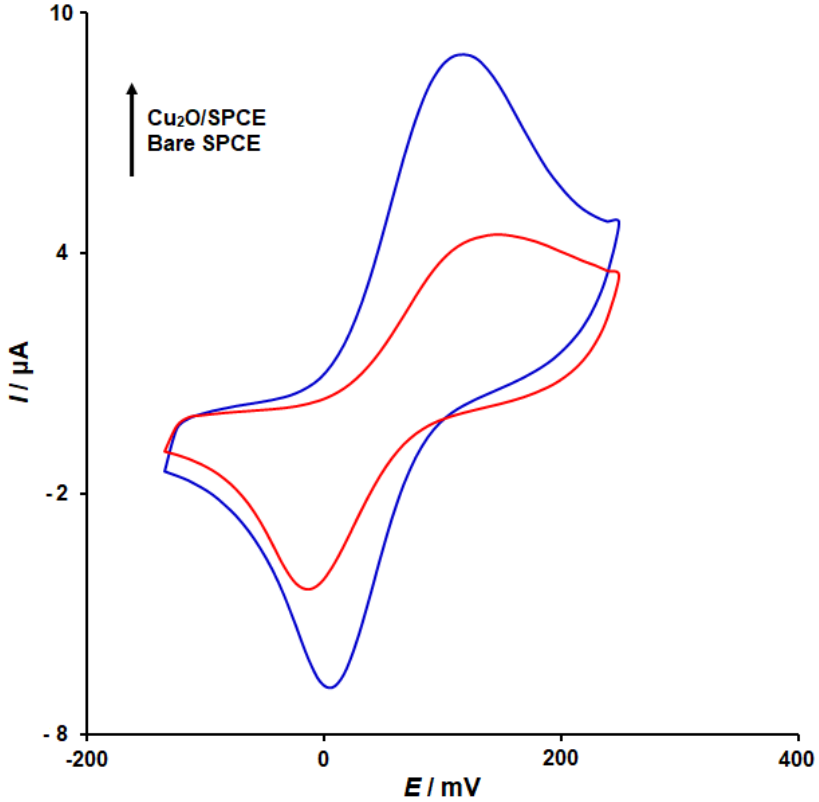

Figure 2. *CV curves of 100.0 µM 4-AP in 0.1 M PB, pH 7.0, at the bare SPCE (red curve) and Cu_2O/SPCE (blue curve) with a scan rate of 50 mV s^{-1}*

Effect of scan rate on the results

Using cyclic voltammetry (CV), the impact of scan rate (v / mV s^{-1}) on the redox reaction of 4-AP at the Cu_2O/SPCE was also examined. The CV curves shown in Figure 3 were obtained for 100.0 µM 4-AP on a Cu_2O/SPCE at scan rates of 10-500 mV s^{-1}. As the scan rate increases, the anodic and cathodic peak currents (I_{pa} and I_{pc}) of 4-AP increase. The linear regression equations are: $I_{pa} = 1.679v^{½} + +2.6319$ ($R^2 = 0.9997$) and $I_{pc} = -1.5616v^{1/2} + 3.7268$ ($R^2 = 0.9978$), respectively. The peak currents show strong linear connections with the square root of the scan rate (insets of Figure 3). These results demonstrate diffusion-controlled electrode reaction.

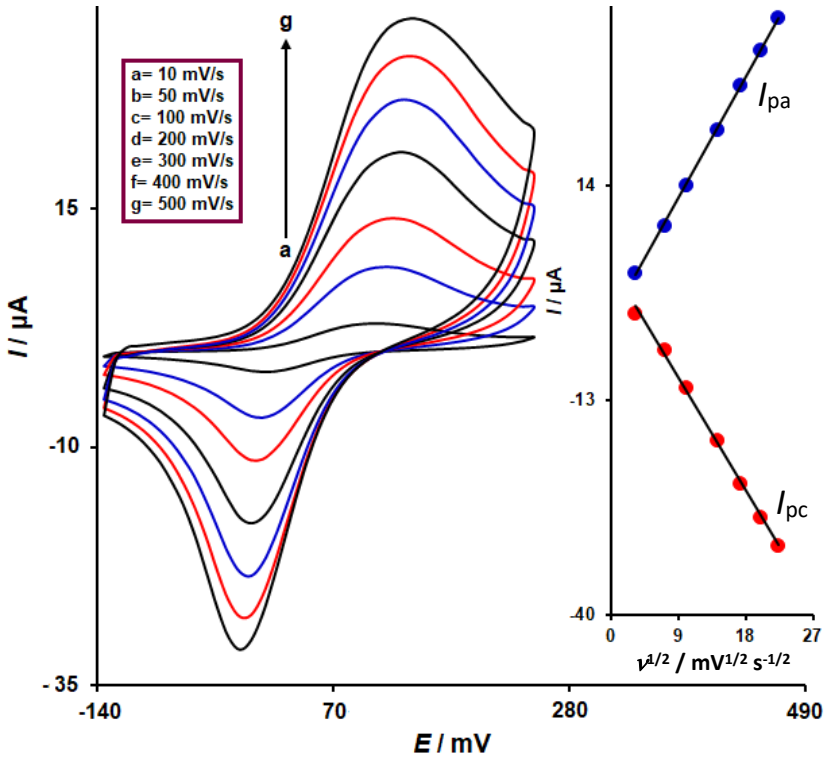

Figure 3. *CVs of Cu_2O/SPCE for 100.0 µM 4-AP in 0.1 M PB, pH 7.0, at different scan rates. Inset: the correlation between I_{pa} and I_{pc} and $v^{1/2}$*

Chronoamperometric analysis

In order to conduct the chronoamperometry investigation of 4-AP at Cu_2O/SPCE, the working electrode potential was set to 0.16 V. As seen in Figure 4, the Cottrell Equation (1) may be used to get the diffusion coefficients (*D*) of 4-AP.

$$I = nFAD^{1/2}C_b\pi^{-1/2}t^{-1/2} \quad (1)$$

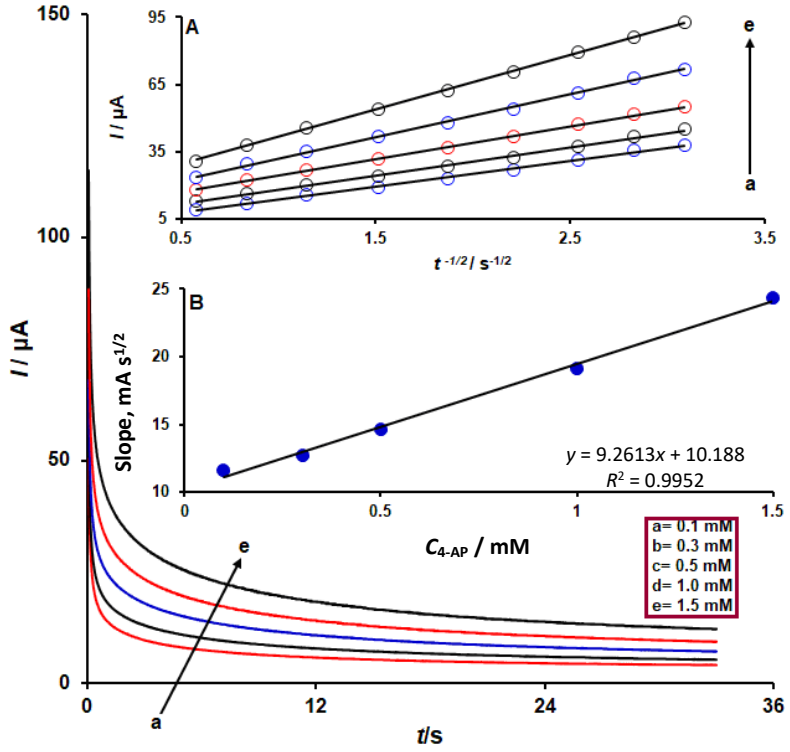

Figure 4. *Chronoamperograms of Cu_2O/SPCE at 0.1 - 1.5 mM 4-AP in 0.1 M PB, pH 7.0. Insets: (A) I vs. $t^{-1/2}$ graphs derived from 4-AP chronoamperograms. (B) Plot of straight-line slopes from (A) against different $C_{4\text{-AP}}$*

As shown in Equation (1), the parameter D / cm² s^{-1} represents the diffusion coefficient of the target analyte, while C_b / mol cm^{-3} denotes its bulk concentration.

Figure 4 displays the chronoamperometric responses at the Cu_2O/SPCE for various 4-AP concentrations (0.1-1.5 mM). Figure 4A and B, respectively, show the experimental plots of I *vs.* $t^{-1/2}$ with the best fits for various 4-AP concentrations and the slopes of the resulting lines versus the 4-AP concentration. The D value of 4-AP was calculated to be 730 nm^2 s^{-1} using the Cottrell equation and the slope *vs*. 4-AP concentration plot.

Calibration curve

Compared with other techniques, the DPV is more sensitive for the electroanalytical determination of electroactive substances. Herein, DPV was used to assess the linear detection range, current sensitivity, and LOD of 4-AP. The DPVs obtained for a range of 4-AP concentrations in 0.1 M PB (pH 7.0) for the oxidation of 4-AP at the Cu_2O/SPCE are displayed in Figure 5. The increase in 4-AP concentration led to an increase in I_{pa}. The plot of I_{pa} against 4-AP concentration shows linearity with a slope of 0.08020 µA $µM^{-1}$ over the concentration range of 0.02 to 500.0 µM (Inset of Figure 5). The LOD was calculated as 0.008 µM using the formula $3s_b/m$, where m is the slope of the calibration curve, and s_b is the standard deviation of the calibration curve.

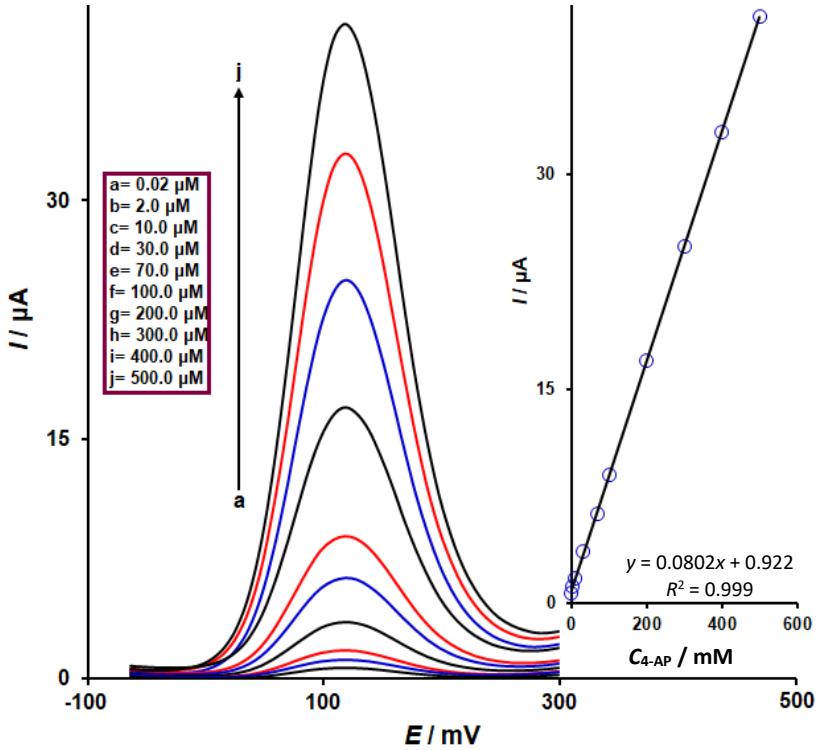

Figure 5. *DPVs for Cu_2O/SPCE at different doses ranging from 0.02 µM to 500.0 µM for 4-AP in 0.1 M PB, pH 7.0. Inset: plot of I_{pa} against 4-AP concentration*

Analysis of 4-AP in real samples using Cu_2O/SPCE

The DPV method was successfully applied to investigate 4-AP in actual water samples (tap water and river water) to demonstrate the practical applicability of the proposed approach. To determine whether the excipient in tap and river water affects the analyte's current response, recovery experiments were conducted using calibration curves obtained after a known amount of pure sample was added to pre-analysed tap and river water samples. This was done to ensure that the developed method was accurate and feasible under ideal experimental conditions. Table 1, which summarizes the recovery and relative standard deviation (RSD), demonstrates that recoveries for

genuine samples are adequate, with RSD values below 3.7 % and recovery near 100 %. This implies that Cu_2/SPCE has potential applications and could be a practical voltammetric sensor for quantitatively measuring 4-AP in real samples.

Table 1. *4-AP determination in real water samples by Cu_2O/SPCE sensing platform (n = 5)*

Sample	4-AP concentration, µM		Recovery, %	RSD, %
	Added	Found		
Tap water	4.0	3.9	97.5	3.3
	6.0	6.2	103.3	2.2
	8.0	7.9	98.7	1.8
	10.0	10.4	104.0	2.6
River water	5.0	5.1	102.0	2.4
	7.0	6.8	97.1	3.7
	9.0	9.4	104.4	1.9
	11.0	10.9	99.1	2.7

Conclusion

Cu_2O nanocubes were prepared in this work using a simple, affordable chemical process. To detect 4-AP, an electrochemical sensor based on SPCE modified with Cu_2O nanocubes was developed. For 4-AP detection, the produced Cu_2O nanocubes exhibit significant electrocatalytic activity. For the 4-AP determination, the Cu_2O/SPCE voltammetric sensor performs well analytically, owing to its low LOD, broad linear range, and high sensitivity. Additionally, the preparation process is easy and inexpensive. Lastly, 4-AP was effectively determined in actual water samples using Cu_2O/SPCE.

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