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Short Communication

Intelligent sensor of glucose based on CuO nanomaterials

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As theory and technology are becoming increasingly mature, the concept of intelligent sensor technology is being introduced into various research fields and gaining increasing attention in current research. Owing to errors when calculating the optimum response voltage of electrodes in the analysis of electrochemical sensors, this paper proposes an algorithm to identify and analyze the data collected from an electrochemical workstation. Subsequently, the optimum working voltage or optimum working voltage range can be found by setting constraints. In addition, a simple preparation process of CuO is introduced, and a series of confirmatory tests are conducted using a CuO electrode with good electrochemical properties. The scientificity, accuracy, and practicability of the intelligent selection voltage are further explained. Finally, through the construction and debugging of the intelligent, portable, and disposable glucose detector hardware circuit, real-time monitoring of glucose concentration is achieved. The results of this research also provide data and a theoretical basis for future studies in the fields of big data analysis and deep learning.

Keywords: Intelligent sensor technology; Electrochemical sensor; CuO electrode; Algorithm; Hardware circuit

1. INTRODUCTION

Intelligent sensor technology [1-4] is developing rapidly in recent years. Its most significant characteristic feature is the fusion of organic integration of sensor detection information function and micro-processing information processing function. Therefore, in a manner, it has a role similar to that of artificial intelligence (AI). AI is the study of utilizing a computer to simulate certain mental processes and intelligent behaviors of humans [5-8]. Since its conception, it has been widely used for intelligent control, image processing, information sensing, logical reasoning, games, machine learning, and so on. With scientific and technological developments, intelligent sensor technology is becoming an integral part of various research fields [9-14]. Hence, it is feasible to apply this technology to the study of electrochemical sensors. It is well known that the detection of glucose in food safety, clinical medicine, biotechnology, and other fields is important [15-20]. Researchers have been working on developing glucose sensors that are cheaper, more sensitive, and exhibit better electrochemical properties [21-25]. In recent years, nano-structured electrode materials [26, 27] have been developed and reported. The glucose sensor studies that use CuO as electrode materials [28-30] were quite mature and self-contained and exhibited improved electrochemical performance, such as higher sensitivity to low detection limits and better stability [31-34]. Presently, there are many reports on the use of cyclic voltammetry (C-V) and chronoamperometry to study the performance of electrochemical sensor electrode materials [35]. In these reports, it is not difficult to observe that the optimum response voltage of the electrode material is artificially estimated from the C-V curve. Obviously, this manual estimation method introduces errors into the analysis of the electrochemical properties of the electrode material. Therefore, to solve this problem, a scientific, accurate, and targeted solution should be proposed.

In this work, the main concept is the use of a computer algorithm[36] based on the concept of least squares[37]. This algorithm is used to intelligently determine the optimal response voltage of the sensor. First, in the experimental part, a CuO material was prepared by a simple preparation process to verify the algorithm mentioned in the next step. This simple preparation process is based on the principle of high frequency induction heating. The preparation process can reduce the preparation cost of the CuO material. Second, an algorithm is proposed. Then, an electrochemical experiment of detecting the glucose concentration with a CuO electrode material is taken as an example to validate the algorithm. The algorithm identifies, processes, and analyzes the C-V curve scan data of the CuO electrode materials. The optimum working voltage or optimum working voltage range of the electrode material can be obtained by setting certain constraints. Finally, the voltage selected by the algorithm is applied to the designed hardware circuit. The hardware circuit is debugged based on the circuit principle and program code, and the real-time detection of glucose concentration is achieved. This breakthrough provides a realistic reference for the development and productization of intelligent, portable, and disposable glucose electrochemical detectors.

2. EXPERIMENTAL

2.1 Reagents and instruments

$Cu(OH)_2$ was purchased from TianJin Heowns Biochemical Technology Co., Ltd. Glucose was purchased from Sigma-Aldrich and used without further purification. Ethyl alcohol was purchased from TianJin KeWei Chemical Reagent Co., Ltd. The ball mill was purchased from Tianjin Xingke

Instrument Co., Ltd. The electrochemical work instrument was purchased from Shanghai Chenghua Instrument Co., Ltd. The high-frequency induction heater was a CX2000 model. The electric heating drum dryer was a WGLL-230BE model. The glue dispenser was a SM200Sx-3A model. The X-ray diffraction instrument used for material characterization was DMAX2000. The scanning electron microscope was a NOVA NANOSEM 430 model. All chemicals used in the experiment were analytical reagent grade.

2.2 Preparation of CuO electrode

Cu $(OH)_2$ powder of 5 g was heated to generate CuO. The heating device was a high frequency induction heater, which can maintain a temperature of 600 °C. The prepared CuO powder was ground in a ball mill and dissolved in ethyl alcohol. To obtain uniform CuO particles with a larger surface area, the centrifuge was used to disperse the CuO dissolved in ethyl alcohol, and then the superstratum solution was taken. The rotation speed during centrifugation was 500 rad·s^{-1} and the time was 20 min. Then, the superstratum solution is filtered using a filter and nylon filter paper. The prepared CuO powder is collected and subsequently dried it in a drying oven and maintained at a temperature of 100 °C. To further increase the specific surface area of CuO powder, a bowl of marble was used to grind the powder for a second time and dry it, yielding the CuO powder for the experiment. Next, 0.1 g copper oxide powder was taken and mixed with 1 mL ethyl cellulose (CE) and 10 mL terpinol. The prepared mixed solution was applied to the printed graphite electrode by the point glue machine; it was then dried to obtain the CuO electrode used in the final experiment.

2.3 Electrochemical measurement

Based on the three-electrode test system, the stirring speed of the magnetic stirrer is set to 1 rad·s^{-1}, and the solution environment is 250 mL 0.1 M NaOH solution. Fig. 1 shows the C-V scanning curve of the prepared CuO modified electrode at different glucose concentrations.

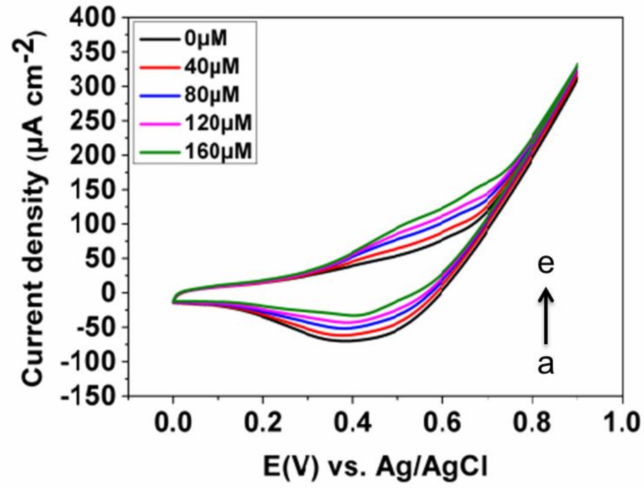

Figure 1. C-V curve scan of the prepared CuO modified electrode at different glucose concentrations.

The curves from a to e represent the C-V scanning curves for different glucose concentrations of 0, 40, 80, 120, and 160 μM [38]. It can be seen from Fig. 1 that the CuO modified electrode prepared by the simple process possesses good sensitivity and selectivity for traces of glucose concentration. To analyze the electrochemical properties of the electrode further, chronoamperometry is often used to test the electrode. Therefore, according to the empirical observation of the C-V curve, we selected 0.39 V as the working potential of the CuO modified electrode to conduct the subsequent test. However, the voltage of 0.39 V cannot be identified as the optimal voltage choice for certain. Therefore, an approach to identify the optimal voltage scientifically and accurately is discussed below.

2.4 Intelligence optimization

Typically, when examining the performance of an electrochemical sensor, its working voltage is estimated by testing its C-V curve. Then, a timing current test is conducted for the estimated voltage to illustrate the electrochemical performance of the electrode through the obtained data. Thus, the following questions arise: Is the voltage value estimated through the C-V curve accurate, and is it the optimal response voltage for this electrode? Can some methods be used to accurately and scientifically select the best response voltage for the electrode? In this paper, we propose an intelligent method to identify the working voltage of electrochemical sensor. A novel algorithm is implemented in MATLAB to intelligently screen the best working voltage of the measured electrode.

2.5 Hardware design

To make the research more practical, this study designs the back-end hardware circuit for the electrochemical sensor. It mainly comprises signal acquisition, AD conversion, an OLED display, and other modules. Real-time monitoring of the glucose concentration is realized through joint debugging between the software and hardware circuits. This provides the basis for the development of disposable, portable, and intelligent electrochemical sensors.

3. RESULTS AND DISCUSSION

3.1 Material characterization

Fig. 2(a) shows the XRD patterns for CuO. Diffraction peaks existing at 2θ values of 32.371°, 35.412°, 38.666°, 48.858°, 53.212°, 58.132°, 61.514°, 66.429°, 67.940° and 74.814° could be assigned to the (-110), (002), (111), (-202), (020), (202), (-113), (-311), (113), and (004) planes of the crystalline CuO, respectively[35, 39]. Fig. 2(b) presents the morphology of the CuO.

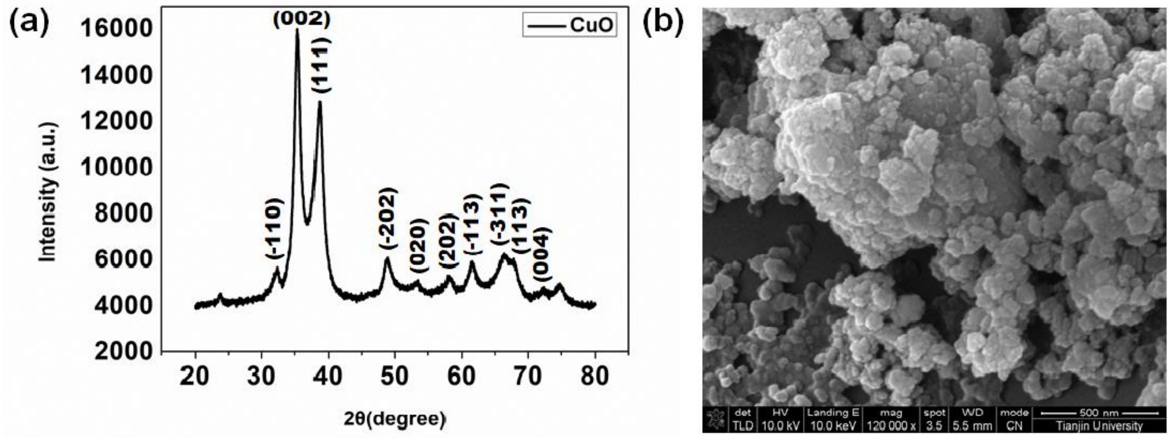

Figure 2. Diagram of material characterization. (a) XRD patterns for CuO. (b) Morphology of CuO.

3.2 Algorithms

Considering the problems mentioned in 2.3, this paper proposes a solution, which is to design an algorithm and implement it in MATLAB [40]. The algorithm is used to identify and process the data obtained from the C-V scan curve test. The program flow diagram is shown in Fig. 3(a). Using the least squares method [41], this paper linearized the value of current, which agreed with the electrodes of different glucose concentrations under the same voltage on the C-V scan curve. Assisted by the computer, the correlation coefficient R^2 and the slope K of the corresponding fitting curve for each voltage are calculated. According to the requirements, the values of R^2 and K are constrained, and the voltage or voltage range that meets the requirements is selected. In theory, the higher the correlation coefficient and the slope, the better the response of the electrode at the corresponding voltage. The operation result graph with constraints as R^2 greater than or equal to 0.998 and K greater than or equal to 0.095 ($R^2 \geqslant 0.998$, $K \geqslant 0.095$) is shown in Fig. 3(b). It can be seen from Fig. 3(b) that the optimal voltage value of the electrode response is 0.49 V. Thus, the electrochemical glucose sensor response voltage has been intelligently determined. To verify that the voltage value of 0.49 V is more accurate and scientific than the artificially estimated voltage value of 0.39 V, a comparative verification test is conducted. The specific analysis is shown for different voltage analyses and comparisons in section 3.3.

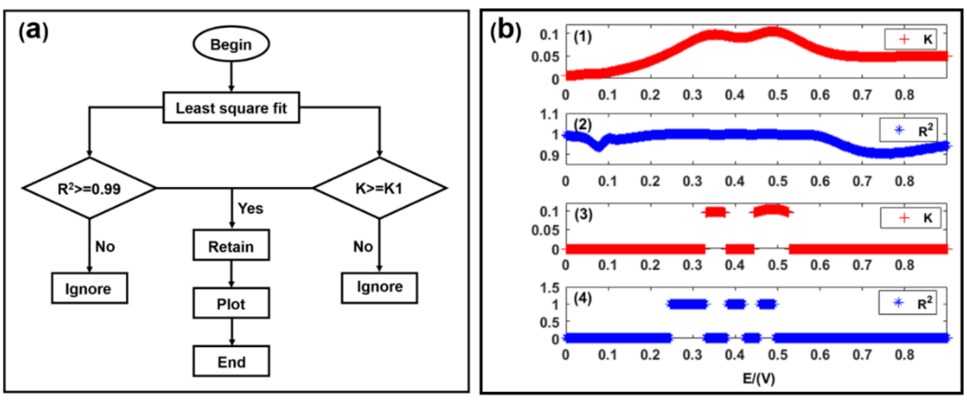

Figure 3. (a) Program flow chart. (b) Operation result diagram of intelligent selection voltage.

3.3 Intelligence evaluation of electrochemical glucose sensor

Fig. 4(a) shows the timing current [42, 43] curves of the electrode under voltages 0.49 V and 0.39 V. The figure clearly indicates that the current response of the electrode at 0.49 V is better than that at 0.39 V. To verify that the intelligent selection of voltage is more scientific and accurate than artificial estimation, the average value of the measured data (Fig. 4(a)) was calculated via multiple measurements. Subsequently, the relationship between the glucose concentration and the response current was analyzed. Fig. 4(b) shows that the relationship was linear. The figure clearly indicates that the CuO electrode has high sensitivity to glucose and exhibits a linear response. Furthermore, the figure indicates that the correlation coefficient of the fitting line of the electrode at 0.49 V is 0.998, and that at 0.39 V is 0.997. Moreover, the linear slope for a voltage of 0.49 V is larger than that for a voltage of 0.39 V. Therefore, from this rigorous data analysis, it can be concluded that the voltage value obtained via intelligent selection is more accurate and more scientific than the artificial value. It also validates the scientificity and desirability of this algorithm.

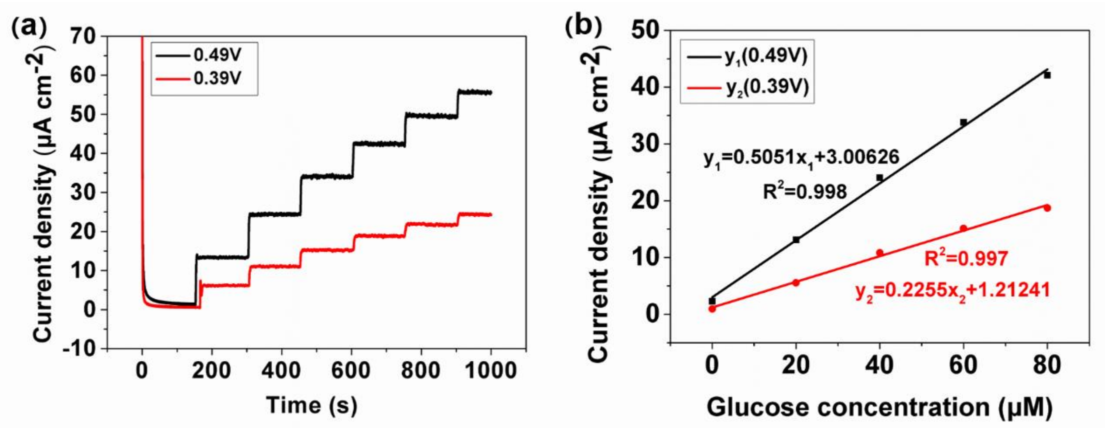

Figure 4. (a) Timing current curve of the electrode under voltages of 0.49 V and 0.39 V. (b) Linear relationship between glucose concentration and electrode response current.

3.4 Comparison of parameters

Table 1 compares the parameters of this work with those of some previous studies. It can be seen that, for similar sensitivity, the detection limit of the glucose sensor electrode prepared herein is relatively low. This is mainly because the prepared CuO nanoelectrode has many nanopores, which increases its specific surface area. Furthermore, in this study, we compared the glucose concentration detection limits of CuO electrodes at different voltages. From the calculation, the detection limit for an artificial estimation voltage of 0.39 V was 1.24 μM. However, the detection limit for an intelligent selection voltage of 0.49 V was 0.557 μM, which is half of the previous value. Therefore, this result also demonstrates the superiority of the intelligent selection voltage and proves the scientificity and practicability of this study.

Table 1. Comparison of parameters based on nonenzymatic glucose sensor based on CuO materials.

	Electrodes materials	Applied potential (V)	Sensitivity ($\mu A\ mM^{-1}\ cm^{-2}$)	Linear range (mM)	Detection limit (μM)	Reference
1	CuO nanowires/GCE	0.55	650	--	2	[44]
2	NPG/CuO	0.4	370	Up to 12	2.8	[45]
3	CuO nanorods	0.6	371.4	Up to 8	4.0	[46]
4	CuO	0.39 (artificial estimation)	225.5	Up to 0.25	1.24	This work
5	CuO	0.49 (intelligent selection)	505.1	Up to 0.25	0.557	This work

3.5 Hardware circuit applications

The research can be made more complete, and the new type of intelligent, portable, and disposable glucose detector can be examined further. Thus, the back-end hardware circuit of the sensor was designed and constructed. Fig. 5(a) and (b) show the module diagram and the welding circuit board of the hardware circuit, respectively [47, 48]. The hardware circuit design mainly comprises the mode selection, signal adjust circuit, AD conversion, data processing, Bluetooth, and display modules, as shown in Fig. 5(a). By debugging the hardware circuit, different glucose concentrations could be monitored and the measured glucose concentration value was displayed in real time through the OLED display, as shown in Fig. 5(b). Fig. 5(c) shows an example of the first generation of intelligent and portable glucose detectors, which is a product of our own research and development. The product's shell is mainly constructed via 3D printing. Next, we will expand the first generation of products to make them multi-functional. In fact, at the time of writing this paper, we have begun the development of the second generation of this product. This research has a definite effect on the development of the medical field, but more importantly, it has guiding significance for the study of intelligent glucose detectors. The real-time monitoring data of glucose concentration can be transmitted to an intelligent terminal, such as a mobile phone, via Bluetooth. People can check their blood glucose concentration at any time or place by using a mobile phone app. In addition, the data can be transmitted to a computer terminal in real time to provide data and theoretical basis for research in the fields of big data analysis and deep learning, assuming that the user has provided consent.

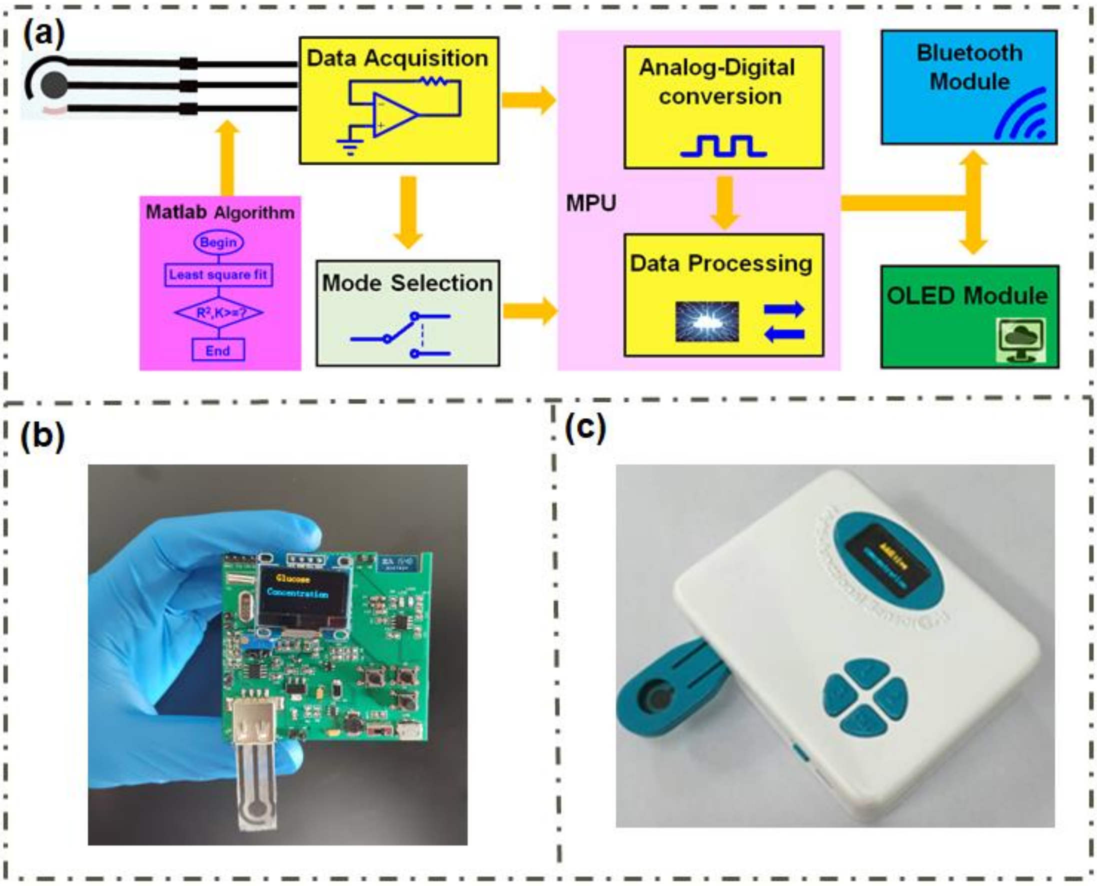

Figure 5. (a) and (b) respectively show the module diagram and the welding circuit board of the hardware circuit . Fig. 5(c) shows the first generation of intelligent and portable glucose detectors.

4. CONCLUSIONS

In conclusion, we have demonstrated the good electrochemical performance of the prepared CuO electrode material. More importantly, its simple preparation process can greatly reduce production costs. In addition, the optimal response voltage of the electrochemical sensor may be erroneous when artificially estimated. Therefore, an intelligent voltage selection method is proposed, and it is verified via a comparison test. The results show that the current response of the electrode is better when the voltage is selected via intelligent optimization. The detection limit of the glucose concentration under the voltage of intelligent selection is lower, and the corresponding correlation coefficient and slope are greater. Therefore, it can be concluded that the intelligence detection method for electrochemical glucose sensor is scientific, and highly accurate, and valuable. Finally, the successful design of the back-end hardware circuit and the implementation of the circuit functions can contribute to the development of intelligent, portable, and disposable glucose detectors in future. Through intelligent sensor technology and big data analysis, people can check and analyze their health status at any time or place through mobile apps and other equipment software, thereby improving their quality of life.

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PATENTS
The optimal response voltage selection method introduced in this paper has been applied for a patent, and the patent application number is: 201811175318.8

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