Electrochemical Sensing of Carbon Coated Metal Oxide Nanoparticles

Yuxi Yang, Jun Luo

Jingdezhen Vocational University of Art, Jingdezhen, Jiangxi Province, China
q2216152000@126.com

Abstract: *Conventional electrochemical sensor electrodes have poor electrical conductivity due to their large surface impedance. Therefore, electrochemical sensing of carbon-coated metal oxide nanoparticles is proposed. Based on the experimental preparation, the preparation of carbon-coated metal oxide nanoparticles Fe304@C and sensors required for the experiment were completed by using chemicals and reaction instruments. The Fe304@C/SPEs studied were tested in a three-level system. Fe304@C/SPEs can effectively reduce the surface impedance of sensor electrodes, promote the movement of electrons, and improve the conductivity of electrochemical sensors.*

Keywords: *Carbon coated metal oxide nanoparticles; Sensors; Conductivity; Electrochemistry*

1. Introduction

Conventional electrochemical sensors have some shortcomings in conductivity and sensitivity. Therefore, in order to enhance the conductivity of electrochemical sensors, the electrochemical sensing of carbon coated metal oxide nanoparticles was studied in this paper. On the basis of the original materials, metals and metal oxides with special properties are used as the electrode materials of sensors. By using their unique nano size effect, large specific surface area, many active sites, controllable morphology and physical and chemical properties, as raw materials for carbon based electrochemical sensors, sensors with high sensitivity, strong selectivity and high conductivity are constructed. This research can enrich the electrical analysis method and provide a new research way for improving the comprehensive performance of the sensor.

2. Experiment preparation

In this paper, carbon-coated metal oxide nanoparticles Fe304@C were prepared by a hydrothermal reduction method and combined with SPEs to construct an electrochemical sensor, as shown in Figure 1. The medicines and instruments required during the experiment are shown in Table 1.
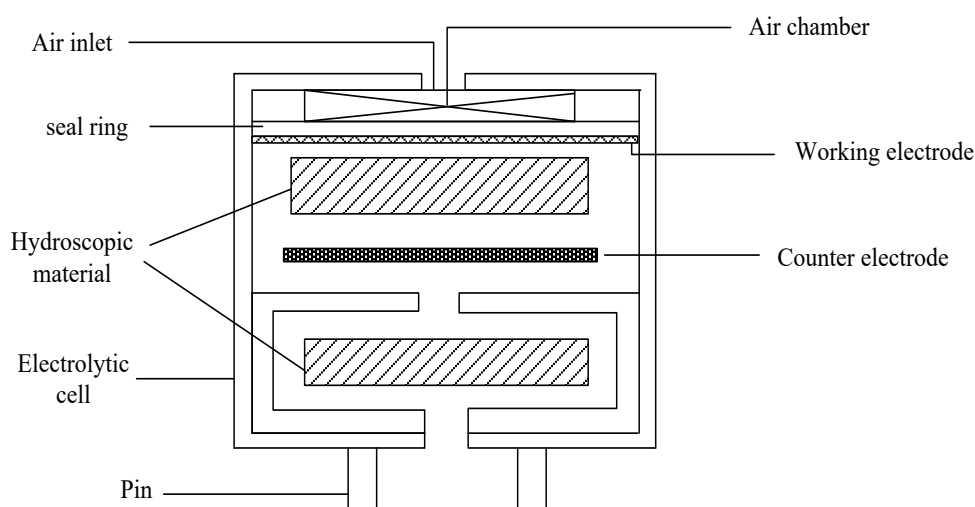

Figure 1: Electrochemical sensor

Table 1: Experimental drugs and instruments

Drug Name	Specification	Drug Name	Specification	Equipment name	Quantity
Ferric chloride(FeCl3)	AR	Ethanol(C2H5OH)	AR	Electric heating constant temperature blast drying oven	1 set
Glucose(C6H12O6)	AR	Acetone(CH3COCH3)	AR	Thermostatic Magnetic Stirrer	1 set
Deionized water(H2O)	AR	Bisphenol A （C15H16O2)	AR	Reactor	1 set
Sodium hydroxide(NaOH)	AR	Polyvinyl alkanone(PVP)	AR	CHI660E Electrochemical Workstation	1 set
Toner(VulcanXC-72)	AR	Hydrazine hydrate(N2Ha.H2O)	AR	Vacuum drying oven	1 set
Formamide solvent(DMF)	AR	Chitosan solution	AR	Ultrasonic cleaner	1 set

Based on the above experimental preparations, carbon coating was carried out Fe304@C Electrochemical sensing experiments. The experimental system is a three electrode experiment, including working electrode, reference electrode and counter electrode. Next, the experimental materials were prepared.
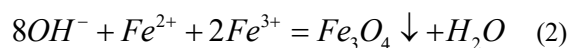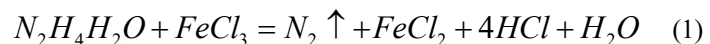
3. Preparation of experimental materials

3.1. Preparation of carbon-coated metal oxide nanoparticles Fe304@C

The metal oxide nanoparticles used in this study are: Fe304@C , prepared by hydrothermal reduction method, the preparation process is as follows:

1). Accurately weigh 0.32g of ferric chloride (FeCl3) with a tray balance, add it into 30mL of water prepared in advance, stir clockwise with a stirring bar to make it mix evenly, then add 0.3g of surfactant while stirring, and stir for another 30 minutes;

2). Use a measuring cylinder to measure 10ml of 70% water and hydrazine solution, add 0.7mg of graphene oxide and 0.3mL of sodium hydroxide (NaOH) to it, fully mix, add 12mL of deionized water, and add the obtained mixed liquid to the above solution; The reaction principle is:

$$N_2H_4H_2O + FeCl_3 = N_2 \uparrow + FeCl_2 + 4HCl + H_2O \quad (1)$$

$$8OH^- + Fe^{2+} + 2Fe^{3+} = Fe_3O_4 \downarrow + H_2O \quad (2)$$

3). Keep stirring the above solution for 5 minutes to obtain a uniform orange-yellow solution, put it into a beaker and place it in a reaction kettle, confirm that the sealing is good, and use a vacuum drying oven to carry out high temperature water at 200 ° C for 12 hours. Thermal reduction reaction [1,2], take out the container after the end, after the liquid is cooled to 20~25℃, use a centrifuge to collect at a suitable speed, and wash with deionized water and ethanol 5~8 times until a clear solution is obtained;

4). Dissolve 2 mL of glucose in dimethyl formamide solvent, mix the aqueous solution of formamide solvent with the above mixture, place it in a 200 mL round-bottomed flask, and drop 10 mL of polyvinyl alkanone into the mixture several times. , forming a homogeneous solution. At a high temperature of 80 °C, magnetic stirring for 3 hours, and finally washing with ethanol for several times to obtain a transparent solution;

5). Dissolve the above transparent solution together with an equal amount of bisphenol a solution in the aqueous nanosheet solution, and fully stir at 90 ° C for 20 minutes under high magnetic stirring. Add sodium hydroxide solution to adjust its pH to 8.0 ~ 9.0 [3,4], and finally wash, centrifuge, collect and dry to obtain dry Fe3O4@C Powdery objects, reserved for use. So far, metal oxide nanoparticles have been completed Fe3O4@C Preparation of[5].

3.2. Preparation of electrochemical sensors

Electrochemical sensors can be obtained by metal carbon-coated metal oxide Fe3O4@C modified SPEs electrodes. Before modifying the electrode, the surface of the SPEs was polished with alumina Al2O3 for no less than 100 cycles, and the cells were carefully washed with sterile phosphate buffer for 3 to 6 times. After removing the medium, add 8 mL of deoxygenated Phosphate buffer solution, placed in an electrolytic cell, followed by direct irradiation of sodium hydroxide solution several times in a row, the grade was activated in a sodium hydroxide base solution by scanning cyclic voltammetry [6,7], and then the solution was allowed to stand for 30 minutes to allow it to The solid precipitated slowly, and the solid product was extracted, and ultrasonic cleaning was carried out in an ultrasonic cleaning apparatus with highly difficult ethanol of deionized water, until the surface was clean and translucent, and no obvious impurities were attached to the surface. Use a tray balance to accurately weigh 3 mg of Fe3O4@C and 2 mg of carbon powder prepared, drop 0.5 mL of acetone into each, stir with a large magnetic force, dissolve them in 2 mL of bisphenol A after thorough mixing, and ultrasonically disperse for 20 minutes. A thin film is formed that can immobilize the previously prepared nanoparticles on the electrode [8,9]. It was then sonicated with 10 mL of a mixture of ethanol and distilled water for 30 minutes under ultrasound and left in air until completely dry. A small amount of the above-mentioned mixed solution of Fe3O4@C and carbon powder was added dropwise on the three electrodes of SPEs respectively, and it was naturally volatilized and dried to obtain two modified electrodes, namely Fe3O4@C/SPEs and C/SPEs. So far, the preparation of the sensor is completed[10].

4. Electrochemical performance test

In this experiment, electrochemical methods were used to test the effect of carbon coated metal oxide nanoparticles on the electrochemical performance of the sensor[11]. The above marked Fe3O4@C /SPES, bare electrode SPEs and C / SPEs were used to conduct the conductivity experiment of the sensor at the same time, and three kinds of electrodes were extracted by AC impedance method for characterization. The test results and comparison are shown in Figure 2.
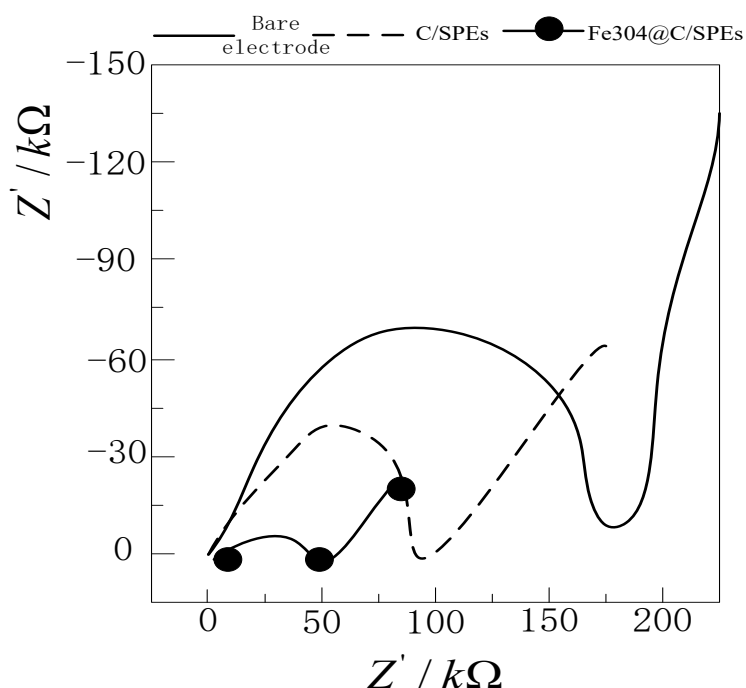

Figure 2: Comparison results

As shown in Figure 1, the bare electrode SPEs, C/SPEs and Fe3O4@C/SPEs electrodes all show semicircular shapes with different radii in specific regions, and then show an approximate straight line. Among them, the semicircle radius formed by the bare electrode SPEs is the largest, indicating that there is a large impedance on the surface of the bare electrode, which makes it difficult for electrons to reach the electrode surface and hinders the transfer of electrons, so its conductivity is poor; C/SPEs electrodes decorated with carbon powders The radius of the circle that appears is significantly reduced,

resulting in the improvement of C, its surface impedance is suppressed, and the electrical conductivity is slightly improved; while the carbon-coated metal oxide nanoparticles Fe3O4@C/SPEs studied in this paper have the smallest semicircle radius, which is Due to the synergistic effect of C and Fe3O4, it can act as a conductor of electrons to facilitate electron movement [12], which greatly improves the conductivity of Fe3O4@C/SPEs.

5. Conclusion

In this paper, iron nanoparticles were successfully prepared by hydrothermal reduction method Fe3O4@C , using the drop coating method Fe3O4@C An electrochemical sensor based on carbon coated metal oxide nanoparticles was obtained by modifying the two electrodes of SPEs. Its conductivity was tested by the three electrode system method. The results showed that compared with the bare electrode and the carbon modified electrode, Fe3O4@C /SPES modified electrode can effectively suppress the impedance of the electrode surface and improve the conductivity of the sensor.

Acknowledgements

References

[1] Wei Liang, Huang Xinlong, Wang Yanli, et al. Graded Porous Carbon Prepared with Different Activators Was Used for Electrochemical sensing analysis of dihydroxybenzene isomers[J]. Analytical Chemistry,2022,50(06):899-911.
[2] Zhang Lida, Wang Yaowu, Liu Jiazhi, et al. Aluminothermic reduction reaction in dry impervious material during aluminum electrolysis [J]. Light Metals, 2021 (5): 30-35.
[3] Cui Jinwen, Feng Taotao, Gao Nan et al. Preparation of Conductive Poly (3,4-ethylenedioxythiophene): Poly(styrene sulfonate) Hydrogel by One-step Method for Electrochemical Sensor[J]. Analytical Chemistry,2022,50(06):859-868.
[4] Geng Ping, Xu Zhiai. Electrochemical sensors based on carbon spheres/gold nanocomposites for the detection of lead and cadmium [J]. Chemical Research and Application, 2020, 32 (8): 30-35.
[5] Yu Hongmei, Hua Ping, Li Jianhua, et al. Magnetic solid acid catalyst Fe_ 3O_ 4/C-SO_ Preparation and Characterization of 3H [J]. Daily Chemical Industry, 2022,52 (04): 396-403.
[6] Sun Yuzhu, Zhang Libing, Zhang Ruizhong. Progress in Preparation of Molybdenum Disulfide and its Application in Electrochemical Sensors[J]. Chemical Reagents,2022,44(07):1063-1070.
[7] Caii Xuefan, Sun Sheng. Cyclic Voltammetry Simulation of Porous Electrode Cells [J]. Electrochemistry, 2021, 27 (6): 646-657.
[8] Liu Jianhua; Xia Zhigang, Zhou Xiangang et al. Preparation of ZIF-8/Carbon Nanotubes Composite and Its Application in Electrochemical Sensing of 4-Chlorophenol[J]. Journal of Analytical Science,2021,37(04):541-546.
[9] Ren Linjiao, Li Chenlong, Qin Zirui, et al Analysis of influencing factors on preparation of gold nanoparticles by sodium citrate reduction [J]. Functional materials, 2021, 052 (002): 2211-2215.
[10] Liu Qiang, Qian Jun, Cheng Zhipeng, et al. Core-shell structure Fe3O4@C Dielectric properties of modified PVDF flexible films [J]. Journal of East China University of Science and Technology: Natural Science Edition, 2020, 46 (2): 179-188.
[11] Ye Faping, Xie Yulong, Guo Qianni, etc. Preparation and electrochemical properties of multi walled carbon nanotubes/cobalt nickel layered bimetallic hydroxide nanocomposites [J]. Modern Chemical Industry, 2022,42 (4): 187-191.
[12] Wang Linyu, Hong Shasha, Li Yanyan, et al. Dopamine Electrochemical Sensor Based on Three-Dimensional Macroporous Carbon/Covalent Organic Framework Integrated Electrode[J]. Analytical Chemistry,2021,49(06):1053-1060.