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# Nano-branched Silver Dendrites Modified Glassy Carbon Electrode for Detection of Heavy Metal Ions

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Silver dendrites were fabricated on platinum mesh by electro-deposition in pure AgNO<sub>3</sub> solution. The dendritic silver powders with nano-branches were obtained after the platinum mesh treated by ultrasonic process. After characterized by Field-emitting Scanning Electron Microscopy (FE-SEM) and X-ray diffraction (XRD), the powders were modified on glassy carbon electrode (GCE) for detection of heavy metal ions. The electrochemical analysis was carried out by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). Heavy metal ions were detected by square wave anodic stripping voltammetry (SWASV) and differential pulse voltammetry (DPV). The sensitivities of cadmium(II), copper(II), lead(II) and mercury(II) were analyzed by DPV as 220.20 $\mu$ A· $\mu$ M<sup>-1</sup>, 58.31 $\mu$ A· $\mu$ M<sup>-1</sup>, 243.31 $\mu$ A· $\mu$ M<sup>-1</sup> and 243.39 $\mu$ A· $\mu$ M<sup>-1</sup>, respectively. The detection limits of Cd (II), Cu (II), Pb (II) and Hg (II) were tested using SWASV method. Corresponding results were shown as 0.0156 $\mu$ M, 0.03518 $\mu$ M, 0.0448 $\mu$ M and 0.0336 $\mu$ M, respectively. Stability experiment further confirmed that silver with nano-branched structure was an alternative material for the detection of heavy metal ions.

**Keywords:** Heavy Metal Ions; Dendritic Silver; Square Wave Anodic Stripping Voltammetry; Differential Pulse Voltammetry

# **1. INTRODUCTION**

Due to the significant impact on people's lives, heavy metal ions have being notorious for environmental problem [1-3]. It has been reported that they not only harm the growth of plants, but also pollute scarce water resources. Moreover, their excessive accumulation in human body results in various diseases and even death[4-6]. In daily life, heavy metal ions mainly come from fossil fuel combustion, metal smelting, and industrial waste water discharge [7-8]. There are several common heavy metals, including Cd (II) Cu (II) Pb (II) and Hg (II). In this context, the dendritic silver

powders with nano-branches have a large specific surface area and strong conductivity, showing excellent electrochemical properties. Based on these characteristics, the sensor can be considered as a potential sensor to detect heavy metal ions [8-9].

Research methods for heavy metal ions detection, such as complexometric titration, X-ray Fluorescence Spectrometry (XRF) and Atomic Absorption Spectrophotometry (AAS), have lots of detection methods because of technology development [10-11]. However, spectroscopic techniques such as AAS require high cost. By contrast, the electrochemical detection method is widely used for the detection and analysis of heavy metal ions, owing to its easy operation, high sensitivity and low cost [12-13]. As a result, electrochemical analyzation is widely applied to the detection of heavy metal ions [14]. The system combined with three electrodes including working electrode(WE), reference electrode(RE) and counter electrode(CE), these is mainly used for fast and simple testing. SWASV and DPV, by causing changes in current through the presence of heavy metal ions, have been established as an important technique for the determination of heavy metal ions [15-20]. Two detection methods are diffusely used due to their quick and sensitive electroanalytical features.

In addition, the nature of the electrode materials has a decisive influence on the display of chemically modified electrodes [21-24]. Electrodes are often coupled with different chemical sensors for improving their sensitivity and LOD by modifying the electrode material [24-27]. As known, nanomaterials have received great attention caused by their structure, surface properties and applied to the surface of GCE for detection of heavy metal ions [27-30]. According to past reports, many materials such as AuNPs [3], SnO<sub>2</sub>/RGO [24] and Fe<sub>3</sub>O<sub>4</sub>[23] were used to prepare sensors for detection of heavy metal ions. It is noteworthy that silver as the active material is found to improve the electrode performance [35-38]. Nano silver particle modified electrode is known for some advantages, such as accelerating electron transport rate, increasing surface area and enhancing electrochemical sensitivity. In this work, the nano-branched silver powders were obtained by selecting suitable electrodeposition conditions in pure AgNO<sub>3</sub> solution. The nano-branched silver dendrites were prepared by high-efficiency, convenient and low-cost method used for highly sensitive detection of Cd(II), Pb(II), Cu(II) and Hg(II).

## 2. EXPERIMENT SECTION

#### 2.1 Materials

AgNO<sub>3</sub> were purchased from Sigma Reagent.  $Hg(NO_3)_2$ ,  $3CdSO_4 \cdot 8H_2O$ ,  $Pb(NO_3)_2$  and  $Cu(NO_3)_2$  were purchased from Sinopharm Chemical Reagent Co., Ltd.. Shanghai, China. Other drugs were purchased from BaiWan Reagent. The aqueous solution was obtained through the UHP Milli-Q ultrapure water manufacturing system (URT-11-10T). Acetate buffer solution (HAc-NaAc) was prepared by mixing 0.1M HAc and NaAc.

#### 2.2 Instruments

Field-Emission Scanning Electron Microscopy (FE-SEM, Hitachi S-4700) was used to

characterize the morphology of dendritic silver powders. Maxima 7000S XRD (Shimadzu, Japan) was utilized to acquire the electrochemical atlas of silver. A dual potentiostat (Wuhan Kesite Instrument Co., Ltd.) of model (CS2350H) was used for electrochemical test. The ultrasonic shredder from SFX Mexico was adopted for obtaining dendritic silver powders from substrate.

# 2.3 Preparation of dendritic silver powders

The electrodeposition was implemented using an ordinary three-electrode cell with platinum network ( $1cm\times2cm$ ) as working electrode. The silver dendrites were electrodeposited on platinum network in 0.01M AgNO<sub>3</sub> aqueous solution. Deposition conditions were chosen as below: deposition voltage of -1V and deposition time of 300s. The silver-plated platinum mesh was put in beaker added de-ionized water for ultra-sonication within 30min. The dendritic silver powders were separated from platinum substrate and dispersed into de-ionized water uniformly.

## 2.4 Modified GCE with nano-branched silver powders

The surface of GCE was polished by using 0.3µm alumina slurry and 0.05µm alumina slurry and cleaned sequentially using 33wt. % HNO<sub>3</sub> solution, absolute ethanol and de-ionized water. 10µl of suspended droplets of nano-branched silver particles (1 mg·mL<sup>-1</sup>) were dropped on bare glassy carbon electrode. After the modified electrodes being dried out, that was used for further analyzation.

## 2.5 Detection of heavy metal ions

The four kinds of heavy metal ions including Cd(II), Cu(II), Pb(II) and Hg(II) were detected separately and simultaneously by SWASV and DPV in a NaAc-HAc buffer solution with pH=5.0. The preconcentration step was processed on condition of -1V for 150s. The experimental conditions of SWASV were as follows: the scanning potential of -1V, the enrichment potential of 1V, the enrichment time of 150s, the frequency of 15HZ and the amplitude of 0.025V.

# **3. RESULTS AND DISCUSSION**

## 3.1 The morphologic characterization of silver dendrites

The morphology of dendritic silver powders was characterized by FE-SEM. As shown in Fig. 1(a), deposited silver on platinum network presented silver dendrites with multi-level branch .Branch of the first-order structure was formed on each trunk, and nanometer branch diameter were formed on the first-order branch. Finally, the complex structure with multiple branch paths was formed by electrodeposition. The nano-branched silver particles were shaken down by ultrasound Through FE-SEM observation, the branched structure of well-preserved silver was maintained, as shown in Fig.1 (b-c). The silver by XRD analyzer was shown in Fig.1 (d). The obvious diffraction peaks appeared at 38.1°, 44.2° and 64.4°, respectively, which the corresponding crystalline faces were (111), (200) and

(220). The peak of the result appears in the same position as the previous report, the prepared product can be confirmed as Ag [26]. The diffraction data was consistent with the joint comittee on powder diffraction standards (JCPDS) card.



Figure 1. SEM images of (a) silver deposited on platinum network from AgNO<sub>3</sub> aqueous solution(deposition voltage:-1V,deposition time:300s);(b),(c) nano-branched silver particles and (d) XRD of nano-branched silver particles.

## 3.2 Electrochemical characterization of silver powders

The performances of the modified glassy carbon electrodes were characterized on cyclic voltammetry(CV). In Fig.2(a), compared with the bare GCE, there was an distinct increase in cathodic and anodic cathodic peaks for GCE modified with nano-branched silver particles in 5mM K<sub>3</sub>[Fe(CN)<sub>6</sub>] containing 0.1M KCl solution [5]. The peaks of the oxidation current of the bare GCE and the GCE modified with nano-branched silver particles were 113.4 $\mu$ A and 207.3 $\mu$ A, respectively. The experiment result indicated that the attachment of nano-branched silver particles to the GCE surface could accelerate electron transfer. Metal accelerates electron transfer rate on electrode surface that similar result was demonstrated in relative report [7]. In Fig.2(b) demonstrated the linear correlation of anodic

and cathodic peak current with square root of scan rate for GCE modified with nano-branched silver particles. The R<sup>2</sup> of the peaks of oxidation and reduction currents were observed by linear fitting to be 0.991 and 0.995, respectively, indicating the linear increase in square root of scan rate from 20 mV·s<sup>-1</sup> to 100 mV·s<sup>-1</sup>. According to the Randle-Sevick Eq [6], the result shown diffusion control current on GCE modified with nano-branched silver particles surface and the value of diffusion coefficient of reactants was calculated as  $2.09 * 10^{-7}$  cm<sup>2</sup>·s<sup>-1</sup>. The electrode modified by nano-branched silver particles was further characterized with electrochemical impedance spectroscopy (EIS). In Fig.2(c), the Nyquist diagram was divided into two parts. The semicircle portion reflected high-frequency range, which corresponded to electron-transfer resistance (Ret). The linear part was equivalent the lowfrequency range, where there was an electron transfer-limited process [20, 21]. The addition of dendritic silver particles caused insignificant change in the Ret value. According to the literature reported in the past, the result consistent with expectations [22].



**Figure 2.** Cyclic voltammogram response of (a) and Nyquist plots (c) bare GCE(black) and modified GCE(red) in the solution of 0.1M KCl containing 5mM [Fe(CN)<sub>6</sub>]<sup>3-/4-</sup> with scan rate 100mV·s<sup>-1</sup>.(B)Cyclic voltammogram response of nano-branched silver particles modified GCE with scan rate 20mV·s<sup>-1</sup>, 40mV·s<sup>-1</sup>, 60mV·s<sup>-1</sup>, 80mV·s<sup>-1</sup>, 100mV·s<sup>-1</sup> and the variation of anodic and cathodic peak current as a function of scan rate (inset).

# 3.3 Individual detection of heavy metal ions

The nano-branched silver particles-modified GCE was used for the individual detection of

heavy metal ions of Cd(II), Pb(II), Cu(II) and Hg(II). The results are shown in Fig. 3(a), the current peak of Cd(II) ranging 0.05µM-0.45µM in 0.1 M HAc-NaAc (pH 5.0) under the deposition potential of -1.0 V for 150 s, proportional to the concentration of Cd(II), and the corresponding calibration were clearly observed. A strong peak reached about -0.78V. According to previous literature reports, the peak position of Cd(II) appears at about -0.8V [23-24]. The linearization equation was i/µA=-0.28+27.55 c /µM and R<sup>2</sup>=0.998 (inset of Figure 3a). The limit of detection (LOD) was calculated to be 0.01 uM. The LOD was calculated from  $3\sigma/s$ , where  $\sigma$  was the standard deviation of the intercept of the concentration-current curve and s was the slope of the curve [44]. Similar, in Fig. 3(b), the current peak of Pb(II) appeared at -0.56V. The satisfied linear equation and correlation coefficient were i/µA=-3.41+75.44c/µM, R<sup>2</sup>=0.975, respectively. LOD was obtained as 0.04µM. In Fig.3(c) and Fig.3(d), the current peaks of Cu(II) and Hg(II) were around 0V and 0.3V, respectively [25]. The satisfied linear equation and correlation coefficient were  $i/\mu A=6.43+33.91c/\mu M, R^2=0.990$  for Cu(II). The calibration curves and correlation coefficients were found to be  $i/\mu A=12.36+42.25c/\mu M$ , R<sup>2</sup>=0.984 for Hg(II). The LODs were 0.082µM for Cu(II) and 0.039µM for Hg(II). The detection limit, sensitivity and correlation coefficient of Cd(II) Cu(II) Pb(II) and Hg(II) in this study were summarized and compared in Table 1. It was reported that compared to past reports, the nano-branched silver particles modified GCE sensor showed higher sensitivity and lower LOD in the process of separate testing [29-30]. Besides, it was found that the LOD of individual detection was lower than simultaneous detection. Furthermore, the sensitivity of Hg(II) and Cd(II) is obviously enhanced due to the presence of a Hg film a during the detection process [26].



Figure 3. (a-d) Electrochemical responses by SWASV and the corresponding calibration plots(inset of Figure 3(a-d) of nano-branched silver particles modified GCE for the individual determination of Cd(II),Pb(II),Cu(II) and Hg(II) Condition: deposition potential:-1.0V ,deposition time 150s, amplitude: 25mV; increment potential: 4mV; frequency: 15Hz. potential scan range from - 1.0V to 1.0V.

DPV (Differential Pulse Voltammetry) was used to detect four kinds of heavy metal ions separately. The current peaks of Cd(II) Cu(II) Pb(II) and Hg(II) appeared near -0.75V, 0V, -0.56V and 0.3V, respectively, which is almost identical with previous reports [31-32]. With the rise in the ion concentration, the current peak experienced proportional to the concentration of Cd(II) Cu(II) Pb(II) and Hg(II). The linear relationship between current and concentration of four metal ions Cd(II) Cu(II) Pb(II) and Hg(II) was obtained as  $i/\mu A = -11.72 + 220.20 c/\mu M$ ,  $i/\mu A = -1.68 + 58.31 c/\mu M$ , i/µA=5.49+243.31c/µM and i/µA=-13.25+243.39c/µM, respectively, with the correlation coefficient of 0.989, 0.994, 0.998 and 0.990 (inset of Figure 4(a-d)). DPV provided a way for the detection of heavy metal ions. The experimental results showed that the separate detection of heavy metal ions by DPV was extremely sensitive.



**Figure 4.** (a-d) Electrochemical responses by DPV and the corresponding calibration plots(inset of Figure 4(a-d) of nano-branched silver particles modified GCE for the individual determination of Cd(II),Pb(II), Cu(II) and Hg(II) from 0.05μM to 0.45μM. Condition: deposition potential :- 1.0V ,deposition time 150s, amplitude: 25mV; increment potential: 4mV; frequency: 15Hz. potential scan range from - 1.0V to 1.0V.



**Figure 5.**(a)SWASV responses the sensitivity versus Cd(II), Pb(II), Cu(II) and Hg(II) of nanobranched silver particles modified GCE.(b) DPV responses the sensitivity versus Cd(II), Pb(II), Cu(II) and Hg(II) of nano-branched silver particles modified GCE.

The four kinds of heavy metal ions were detected by square wave stripping voltammetry (SWASV) and Differential Pulse Voltammetry (DPV). The sensitivity of Cd(II) Cu(II) Pb(II) and Hg(II) can be seen in Fig. 5(a). The sensitivity of Pb(II) was found to up to 75.44 $\mu$ A· $\mu$ M<sup>-1</sup>, followed by Cd(II) of 57.55 $\mu$ A· $\mu$ M<sup>-1</sup> and Hg(II) of 57.32 $\mu$ A· $\mu$ M<sup>-1</sup>. The sensitivity to Cu(II) was the least, 33.91 $\mu$ A· $\mu$ M<sup>-1</sup>. In Fig. 5(b), the sensitivity of Cd(II) Cu(II) Pb(II) and Hg(II) were 220.20 $\mu$ A· $\mu$ M<sup>-1</sup>, 58.31 $\mu$ A· $\mu$ M<sup>-1</sup>, 243.31 $\mu$ A· $\mu$ M<sup>-1</sup>, and 243.39  $\mu$ A· $\mu$ M<sup>-1</sup> by DPV.

### 3.5 Simultaneous detection of heavy metal ions

In the process of simultaneous detection of multiple metal ions, selectivity of DPV showed obvious instability. Multiple metal ions were frequently detected during practical sensor applications. As known, sensitivity and selectivity were of equal importance for detection of the heavy metal sensors. Hence, multiple co-existed metal ions were detected by square wave stripping voltammetry (SWASV), the experimental conditions were the same as those of separate detection [33-35]. Simultaneous detection of two, three, four kinds of heavy metal ions were shown in Fig. 6(a), (c) and (e). The single current stripping peak of the four kinds of metal ions of Cd(II) Cu(II) Pb(II) and Hg(II) was clearly observed in the simultaneous detection of heavy metal ions, respectively observed at - 0.75V, 0V, -0.56V and 0.3V. To compare the sensitivity of separate detection and simultaneous detection of four kinds of metal ions, the sensitivity, correlation coefficient and detection limit were summarized in Table 1.



Figure 6. (a) SWASV responses of nano-branched silver particles modified GCE for the simultaneous detection of Pb(II) and Hg(II) (b) Calibration plots toward Pb(II) and Hg(II). (c) SWASV response of nano-branched silver particles modified GCE for the simultaneous detection of Cd(II), Pb(II) and Cu(II). (d) Calibration plots toward Cd(II), Pb(II) and Cu(II). (e) SWASV response of nano-branched silver particles modified GCE for the simultaneous detection of Cd(II), Pb(II) and Cu(II). (f) The respective Calibration plots toward Cd(II), Pb(II), Pb(II), Hg(II) and Cu(II). (f) The respective Calibration plots toward Cd(II), Pb(II), Cu(II) and Hg(II) corresponding to Figure 6(e).

Table 1. Comparison between individual and simultaneous four metal ions detection

Analysis	metal ion	Detection limit(µmol/ L)	Correlatio n Coefficient	Sensitivity (mA mmol1)
Individual	Cd(II)	0.016	0.998	57.55
	Pb(II)	0.032	0.979	75.44

	Cu(II)	0.045	0.990	33.91
	Hg(II)	0.034	0.997	57.32
Simultaneous	Cd(II)	0.145	0.989	30.07
	Pb(II)	0.104	0.994	86.62
	Cu(II)	0.115	0.986	63.22
	Hg(II)	0.087	0.982	42.25

 Table 2. Comparison of our proposed electrode material with other electrode material for the determination

Modified electrodes	metal ion	LOD	Method	Reference
				S
Silver of twig	Cd(II)	0.145µM	SWASV	Present
structure	Pb(II)	0.104µM		
	Cu(II)	0.115µM		
	Hg(II)	0.087µM		
MgSiO <sub>3</sub> /Nafion/GC	Cd(II)	$2.01  imes 10^{-10} \mathrm{M}$	SWASV	[20]
E	Pb(II)	$2.80 imes10^{-10}~{ m M}$		
	Cu(II)	$1.01 imes 10^{-10}~\mathrm{M}$		
	Hg(II)	$1.49  imes 10^{-10} \mathrm{M}$		
rGO-Fe <sub>3</sub> O <sub>4</sub> /GCE	Cd(II)	0.008µM	SWASV	[22]
	Pb(II)	0.006µM		
	Hg(II)	0.004µM		
Fe <sub>3</sub> O <sub>4</sub> /GCE	Cd(II)	0.213µM	SWASV	[23]
	Pb(II)	0.06μΜ		
	Cu(II)	0.22µM		
	Hg(II)	0.059µM		
NiO/GCE	Cd(II)	0.07µM	SWASV	[25]
	Pb(II)	0.08µM		
AgNPs/RGO/GCE	Cd(II)	0.287µM	SWASV	[26]
	Pb(II)	0.245µM		
	Cu(II)	0.171µM		
	Hg(II)	0.180µM		
Fe <sub>3</sub> O <sub>4</sub> /GCE	Pb(II)	0.119 µM	SWASV	[29]
Bi/HP-β-CD- RGO/Nafion/GCE	Pb(II) Cd(II)	0.09μM 0.07μM	SWASV	[31]

In the simultaneous detection of two and three kinds of metal ions, the detection sensitivity got reduced, which may be attributed to the competitive adsorption of metal ions. In the process of simultaneously detecting four kinds of heavy metal ions, the corresponding linear equations were  $i/\mu A=-1.18+30.07c/\mu M$ ,  $i/\mu A=2.01+86.62c/\mu M$ ,  $i/\mu A=-2.13+63.22c/\mu M$  and  $i/\mu A=-0.59+42.25c/\mu M$  for Cd(II), Pb(II), Cu(II) and Hg(II) respectively, with the correlation coefficients of 0.989, 0.994, 0.986 and 0.982, respectively. The result indicated that the detection sensitivity of Cd(II) and Pb(II) got

increased(inset of Figure 6(e-f)).The comparison of heavy metal ions detection was summarized in Table 2. It can be found that the LODs for Cd(II) Cu(II) Pb(II) and Hg(II) were found 0.145 $\mu$ M, 0.104 $\mu$ M, 0.115 $\mu$ M, 0.087 $\mu$ M in this research. The detection limits and sensitivities of this experiment were compared with other papers. The four kinds of metal ions were detected simultaneously by SWASV, Fe<sub>3</sub>O<sub>4</sub> [23] and AgNPs/RGO [26] had reported that compared with this sensor, the nanobranched silver particles modified GCE sensor showed similar detection limit and a lower detection limit on the detection of certain ions. Compared to other materials, such as rGO-Fe<sub>3</sub>O<sub>4</sub> [22], NiO [25] and Bi/HP- $\beta$ -CD-RGO/Nafion [31], this sensor had detected four kinds of metal ions simultaneously, simple materials preparation process and less synthetic steps. In the results of simultaneous detection of four kinds of metal ions, the reason can be considered as the combination of inter-metallic assemblies of the two metals during the enrichment process when both Cd<sup>2+</sup> and Pb<sup>2+</sup> were present. Also, the decrease in the detection sensitivity of Cu(II) and Hg(II) can be explained by the adsorption and interaction of metal ions in the solution[37-38]. Obviously, In comparison with Fig. 3, one single stripping peak for Pb (II) was observed at around -0.55 V. This stripping peak should be due to the formation of Hg film, which is fully compatible with previous reports [45].

#### 3.6 Stability measurement

To determine the reliability and stability of the nano-branched silver particles modified GCE was measured in parallel experiment for seven times. The parallel experiment is that the modified electrode, after 3 days of storage in air, used to detect Hg(II) at 2.0mM, as shown in Fig. 7. The test results indicated that the current peak was practically constant during the process. The results of this experiment demonstrate that the nano-branched silver particles-modified GCE showed good cycling stability and long-term durability as a sensor for detecting heavy metal ions. This confirmed that the glassy carbon electrode modified with nano-branched silver particles could be used as an electrochemical sensor.



**Figure 7.** SWASV responses towards Hg(II) of nano-branched silver particles modified GCE in the 1<sup>st</sup> cycle and after the 7th cycle.

# 4. CONCLUSIONS

In this work, an electro-deposition method was applied to dendritic silver powders with unique morphology. The modified material prominently enhanced the electrochemical response for detection of Cd(II), Pb(II), Cu(II) and Hg(II). Meanwhile the current peak of each heavy metal ions were obviously defined and separated during the simultaneous detection of four kinds of heavy metal ions. More significantly, the modified electrode exhibited high sensitiveness in the simultaneous detection of heavy metal ions, which was attributed to the nano-branched silver particles quantum characteristics and unique structures. The present study offered such a simple and efficient sensor applicable to simultaneous detection of heavy metal ions with high sensitiveness.

### AUTHOR CONTRIBUTIONS

Ling Liu, Kai Zhuo and Shengbo Sang designed the experiments; Liu Ling and Kai Zhuo performed the experiments; Qiang Zhang analyzed the data; Ling liu ,Kai Zhuo and Shengbo Sang wrote the paper; all authors discussed the results and commented on the manuscript.

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# CONFLICT OF INTEREST STATEMENT

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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