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Sequential injection analysis for mercury ion with modified screen – printed carbon electrode

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Abstract

In this study, we developed a simple high-throughput and cost-effective method for monitoring toxic metal ion in an environmental aqueous sample. Mercury ion determination with Sequential Injection Analysis system (SIAs) coupled with the electrochemical detection on the modified screen-printed carbon working electrode (SPCE) is an alternative green analysis of mercury ion. The gold film was used as the modified material for improved mercury ion analysis in the automated system without memory effect on the electrode. Mercury oxidation signal was found at the potential of 0.7 V in 0.1 M HNO_3 and 1.0 M HCl with the concentration low to 0.25 ± 0.18 $mg\cdot L^{-1}$. Online sample preparation and separation will study in the further experiment.

1. Introduction

Mercury pollution has drawn global concern because mercury can damage the environment and human toxic even at low concentration [1,2]. Mercury is commonly found in the earth's crust. It is generally in the form of elemental mercury (Hg^0) or in the form of a solid. The dissolved mercury can exist in both +2, +1 and 0 charges. The positively charged mercury ions can be bind with other negatively charged ions such as hydroxides, chlorides and sulfides to form compounds such as $Hg(OH)_2$, $HgCl_2$, $HgSO_4$ and HgS. Methylated mercury (CH_3Hg^+) and dimethylmercury ($(CH_3)_2Hg$) are the most highly toxic compared to other forms of mercury [3-6]. It has the ability to move in the environment (bioaccumulation) by accumulating more and more in the organisms at the top of the food chain [7,8]. Through the transfer of pollutants in chains (biomagnification), mercury ions are also able to bind to particles and dissolved organic matter, which binds mercury with these substances to form mercury. Mercury speciation is one of the pollutants that affect the mercury cycle in nature causing the distribution of contaminants in soil, water and air from various activities as well for example from a volcanic eruption soil erosion hydrological cycle and from forest fires, which is a natural phenomenon. Moreover, industrial sectors such as gold mining, coal burning, cement production process, steel and metal manufacturing process, chlor-alkali process for the production of chlorine and caustic soda, oil refining process were also distributed cause [9-11]. This includes the use of mercury in products such as fluorescent lamps, batteries, home paints and dental fillings, which is a human activity. Mercury is often contaminated with raw water used to make tap water, food and cosmetics, which is a long-life cycle of marine fish are eaten and accumulated in fish such as tuna, sharks and king mackerel [12-14]. Inorganic mercury has a good ability to dissolve in fat and can bind to proteins in cells well. Therefore, it is difficult to be excreted from the body and can be accumulated in large quantities. It will accumulate in the fat in the human brain and will injure the central nervous system causing spasms personality changes symptoms of mercury poisoning generally range from severe abdominal pain, headache, body aches, numbness, memory, and depression [15]. It was also affected to the development of the brain and nervous system of the fetus and young children. Therefore, the US Food and Drug Administration are advisable for pregnant women, breastfeeding mothers and children to avoid consuming certain types of fish [16,17]. The amount of mercury that the WHO has defined as in water 6 $\mu g\cdot L^{-1}$ for inorganic mercury and in air is 1 $\mu g\cdot m^{-3}$ (annual average). While the EU has defined the urban area for mercury at 0.1 $ng\cdot m^{-3}$ to 5 $ng\cdot m^{-3}$ and the industrial emission area in the range of 0.5 $ng\cdot m^{-3}$ to 20 $ng\cdot m^{-3}$ [18,19].

To date, monitoring trace amount of mercury is very important. Various techniques have been exploited including atomic absorption spectrometry [20-22], atomic fluorescence spectrophotometry [23],

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quartz crystal microbalance [24], inductively coupled plasma mass spectrometry [25-27], surface-enhanced Raman scattering [28] and X-ray spectrometry [29,30]. However, these techniques suffer from time-consuming, well-trained operators, large sample requirement, expensive costs, and not suitable for a sample pre-treatment or for in situ analysis. Also, the colorimetric method and paper-based device are the preferred assignments to mercury (II) determination [31-34]. The advantage reason for colorimetry is used in the resource-limited, cost-effectiveness, disposability, simple fabrication, low sample volume consumed, and rapid obtained results via the eye, scanner or smartphone. The main disadvantage of colorimetry comparison with SIA electrochemistry is the online sample preparation and separation in the automated system which is to reduce the risk of harmful chemicals and substances.

On the other hand, the electrochemical-based automated analytical system is the best alternative and most profitable tool for trace mercury measurement techniques which procures many advantages such as high sensitivity, good selectivity, simple and quick measurement with automated, and low cost [35]. A recently developed online sequential injection analysis system (SIA) is aimed for direct and fast determination of toxic mercury. The electrochemical method integrated with SIA is used to deliver samples directly to the analytical system. Especially the use of a programmable syringe pump and a multiport switching valve as an autosampler leads to improved accuracy and precision of online results. In the part of electrochemical detection, the working electrode is like the heart of the method which is one of the factors that greatly affected the sensitivity. Therefore, the selection of materials used as working electrodes is very important. The most popular electrode materials used in electroanalytical measurements of Hg^{2+} are carbon-based and gold-based materials because it is high conductivity, easy to modify with other material, and inexpensive material [36-38]. Moreover, noble metal thin film modified electrode is the best choice of material used for Hg^{2+} determination [39]. The preparation of noble metal films on thin plastic sheets is attractive because electrodes of any size or shape can easily be cut and used for particular electrochemical purposes. The screen-printing and ink-printing have been proposed for the fabrication of disposable electrodes. The main aims of these electrodes are in the fabrication of inexpensive enzyme sensors or as simple devices for stripping analysis. Gold films have attracted increasing interest over the last ten years due to their unique properties of high selectivity, high surface area, and high electrical conductivity suitable for electrochemical activity and chemical stability [40].

In this article, we report the voltammetric behavior of thin-film gold electrodes for Hg^{2+} sensor prepared by online in situ electrodeposition using a plastic screen-printed carbon electrode as substrate.

2. Experimental

2.1 Reagents

All reagents used in this experiment were analytical reagent (AR) grade and solutions were prepared using high pure water with a resistance of $18\ M\Omega\cdot cm^{-1}$ (Millipore purification 10 system). Gold(III) chloride trihydrate ($HAuCl_4.3H_2O$) was obtained from Sigma-Aldrich (St. Louis, USA). Hydrochloric acid 37% (HCl) was receive from QReC (QRec, Newzealand). Mercury standard solution for AAS grade 1000 $mg{\cdot}L^{-1}$ was purchased from Fulka (Fluka, Buchs, Switzerland). Nitric acid (HNO_3) was suppled form Merck (Darmstadt, Germany).

2.2 Instrumentation

A schematic diagram of the complete SIA manifold is illustrated in Figure 1. A 2.5 mL syringe pump with a 3-way syringe valve, an 8-port selection valve, a 6-port switching valve (not used in this research), a 3.0 mL PTFE holding coil (0.8 mm i.d.) and PTFE tubing (0.8 mm i.d.) with PEEK connectors, were used to aspirate and deliver the various solutions for on-line modification of the electrodes and the determination of Hg^{2+}. The SIA apparatus, the potentiostat and data acquisition were accomplished by a software application program controlled by SIA MPV version 5.0 software (MGC Japan). All electrochemical measurements were carried out in a complete flow cell (cross-flow MF-1093, Bioanalytical

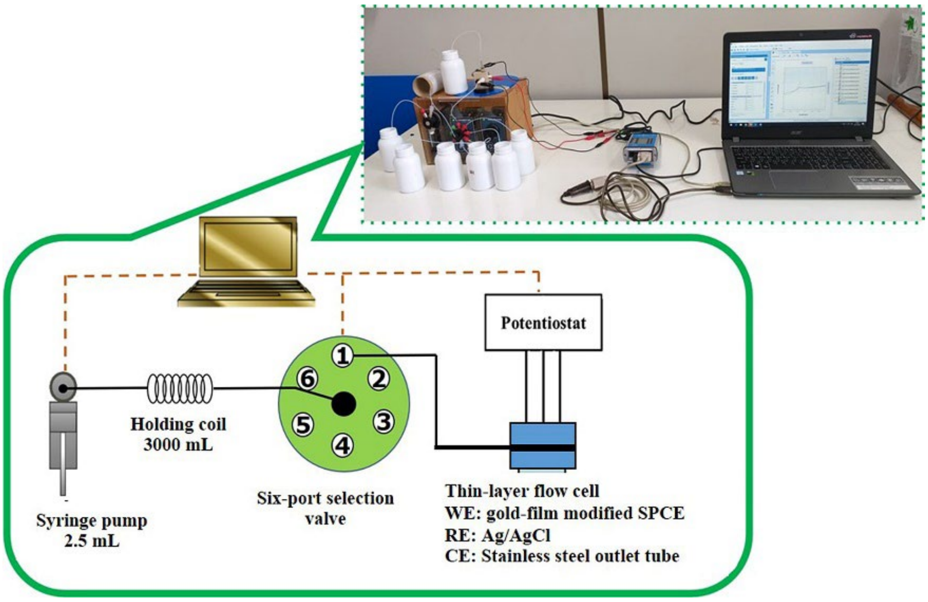

Figure 1. Schematic diagram of the sequential injection.

System Inc., USA) using a portable 910 PSTAT Mini potentiostat (Metrohm Autolab, Switzerland). The complete flow cell consisted of a 0.5 mm thick silicone gasket as a spacer, and a three-electrode configuration of a gold-modified SPCE, an Ag/AgCl electrode (3 M NaCl) and stainless steel outlet tube as a working electrode, a reference electrode and a counter electrode, respectively.

The screen- printed carbon working electrode (SPCE) was manually fabricated on the polyvinyl chloride (PVC) substrate using a screen-printing method. Initially, the carbon ink was printed through the 150 mesh polyester screens onto the PVC substrate to be used as a conducting pad and working electrode area. Next, the electrode pattern was dried in an oven at 55°C for 1 h. Finally, the SPCE were cut into the dimensions of 2.5 cm × 1.0 cm for each electrode.

2.3 Experimental procedure

In the experiment, the effect of supporting electrolyte, amount of sample, and gold plating solution were studied because it is related to the sensitivity of the proposed method. by using the electrochemical method for mercury ion determination, two supporting electrolytes were widely used, namely HNO_3 and HCl [41]. Then the oxidation current of mercury ion in SIA system is analyzed with the various volume of sample solution, gold plating solution, and two kinds of supporting electrolyte. The step of experimental sequence is summarized in Figure 2 and in Table 1 with the total solution in the syringe pump at 2,400 mL and the ratio of sample solution to plating solution at 1:1 and 2: 1, respectively. The concentration and volume of Au solution are affected to electrode surface roughness (surface area) and related to the mercury (II) ion concentration range. Then, the solution ratio of sample solution and plating solution was investigated.

A zone of cleaning solution, 1M HNO_3, was firstly aspirated into the holding coil followed by sample solution, gold plating solution (50 $mg \cdot L^{-1}$ Au^{3+}) and 1 M HCl as the supporting electrolyte, respectively (step 1). The dispensing of the solution was delivered to the cell while the potential of the working electrode was maintained for cleaning and conditioning the electrode at +1.2 V for 30 s, following by the delivery of gold plating solution and sample solution to the cell with the potential at -0.5 V for 180 s for gold and Hg^{2+} simultaneously deposited on the electrode surface (step 2). Next, the flow was stopped, and after equilibration time for 5 s (step 3), an anodic potential scan in the voltammetry mode was applied and the voltammogram was recorded from -0.2 V to +0.4 V in this quiescent solution (step 4). Finally, the electrode was cleaned with 1 M HNO_3 from trace residues metal. The electrode was then ready for a new voltammetric cycle.

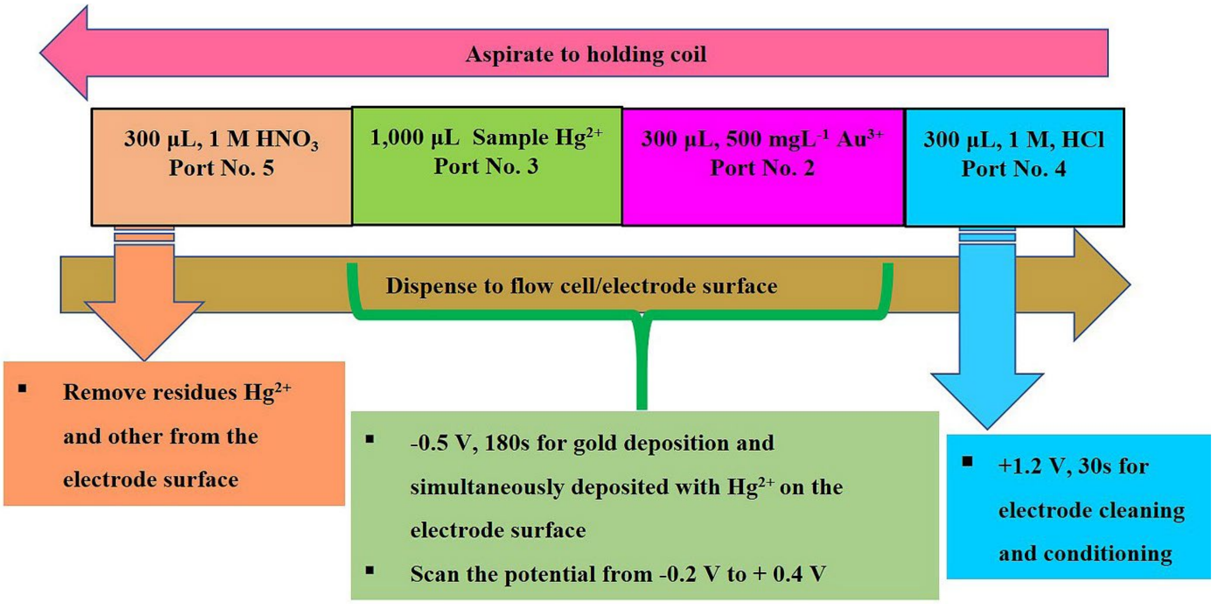

Figure 2. The sequence of solutions in the aspiration and dispensing direction.

Table 1. Step sequence for the determination of Hg^{2+} with in situ gold modification on screen-printed carbon electrode by SIA system.

Step	Description	Valve position	Pump status	Volume (µL)	Flow rate ($\mu L \cdot s^{-1}$)	WE (V)	Duration (s)
1	Aspirate solution into holding coil	5 (HNO_3) 3 (Hg^{2+}) 2 (Au) 4 (HCl)	Aspirate	300 1,500, 900 300, 900 300	100	-	21
2	Condition and preconcentration	1	Dispense	1,800	10	-0.5	180
3	Equilibration	-	Delay	-	-	+0.0	5
4	Stripping, recording	-	Delay	-	-	-0.2 to +0.4	60
5	Electrode cleaning	1	Dispense	300	100	+1.2	3
Total analysis time (s)							269

3. Results and discussion

From the studies of mercury determination, we were started with SIA condition - linear sweep voltammetry for determination signal of gold ion and mercury (II) ion. Linear sweep voltammetry in the solution containing 50 mg·L^{-1}, Au^{3+} and 1 mg·L^{-1} Hg^{2+} in 1 M HCl were conducted in the range -0.5 V to +1.2 V. The Au^{3+} and Hg^{2+} oxidation step was not clearly defining in the anodic scan (Figure 3). The oxidation peak of Au^{3+} and Hg^{2+} was observed at the potential of +0.7 V and + 0.05 V, respectively. It demonstrated that anodic stripping voltammetric detection is possible with the thin-film gold electrodes and can be utilized for the detection of mercury (II) ion in an automated system. These results may occur from Nano gold (AuNPs) form to the thin film on the carbon surface. The phenomena of mercury (II) on the gold electrode surface was described by W. Charles Martin [41] The gold film modified screen-printed carbon electrode can pass good electrons in the electrochemical system because the rough surface gives a high surface area for transferring electrons. Therefore, gold film-modified electrodes are more capable of transferring electrons from Hg^{2+} measurements more than unmodified electrodes [41]. The surface morphologies of in situ gold film modified screen-printed carbon electrode was showed in Figure 4. The surface roughness of the screen-printed carbon electrode was increased, and it was affected to the surface area for Hg^{2+}determination. In the further step, the variables affecting the stripping response of Hg^{2+} were investigated by differential pulse voltammetry with -0.5 V deposition potential for 180 s.

Initially, different supporting electrolyte (1 M HCl and 1 M HNO3) were assessed as a carrier and supporting electrolyte solution for the determination of Hg^{2+} by anodic stripping voltammetry. For the subsequent experiments, a 1 M HCl was selected because it produced the highest and sharpest stripping peak current (Figure 5(a). The effect of the sample volume at 900 µL and 1,500 µL on the stripping of Hg^{2+} was examined. The current response increased with increasing sample volume. A sample volume at 900 µL was selected as a compromise sensitivity and analysis time. The reproducibility of the in-situ gold modification on screen-printed carbon electrode by SIA system studied with 900 µL of 1 mg·L^{-1} Hg^{2+} showed an acceptable %RSD value of 4.08 (Figure 5(b). From the study, a sequential injection analysis has been applied for determination of Hg^{2+} with automatic analysis under the selection parameters. The system was performed with many advantages such as rapid analysis time, reduce reagent and hazard reagent consumption, and less waste generation which leads to environmental contamination and negative human health effects. Nonetheless, a green analysis for Hg^{2+} has been presented. The calibration of Hg^{2+} determination with in situ gold modification on screen-printed carbon electrode by SIA system showed a linear relationship at the R2 = 0.9468 (Figure 6). The acceptability criterion of the correlation coefficient value in chemical analysis is higher than 0.95 [42]. Then, the result of this study demonstrates the need for continued development and evaluate alternative methods for use in mercury determination.

The detection limit was evaluated by the peak responses of a solution containing Hg^{2+} at a low concentration of 1 ppm (N=10); this is called the method of detection limit (MDL). The MDL can be calculated from the 3S/sensitivity ratio, where S is the standard deviation of the peak responses, and the sensitivity ratio is the average of the peak responses over the selected concentration. It was found that the MDL of the proposed method is 0.25 ± 0.18 mg·L^{-1}. Therefore, the MDL has been confirmed by analyzing a standard sample containing 0.25 ± 0.18 mg·L^{-1}, which is resulting in Figure 7 with the oxidation peak of Hg^{2+}.

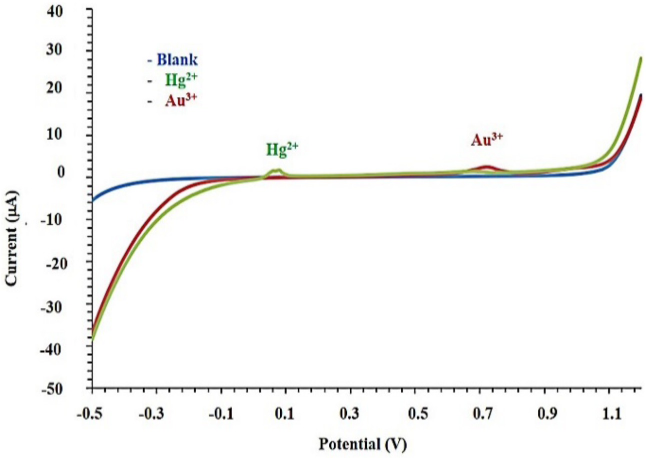

Figure 3. Voltammogram of 1mg·L^{-1} Hg^{2+}, 50 mg·L^{-1} Au^{3+} in 1 M HCl and blank solution (1 M HCl).

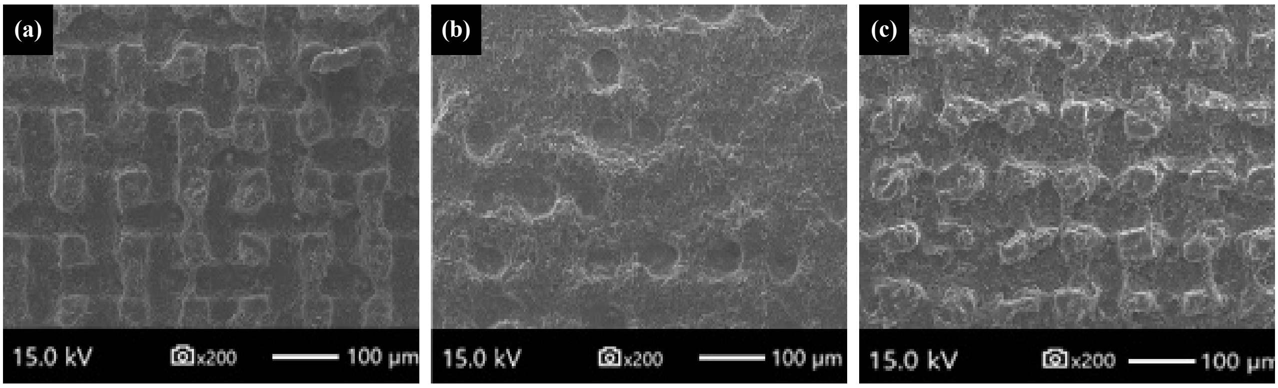

Figure 4. Surface morphologies of screen-printed carbon electrode (a) bare screen-printed carbon electrode, (b) gold film modification on the screen-printed carbon electrode, and (c) mercury deposition on the modified screen-printed electrode.

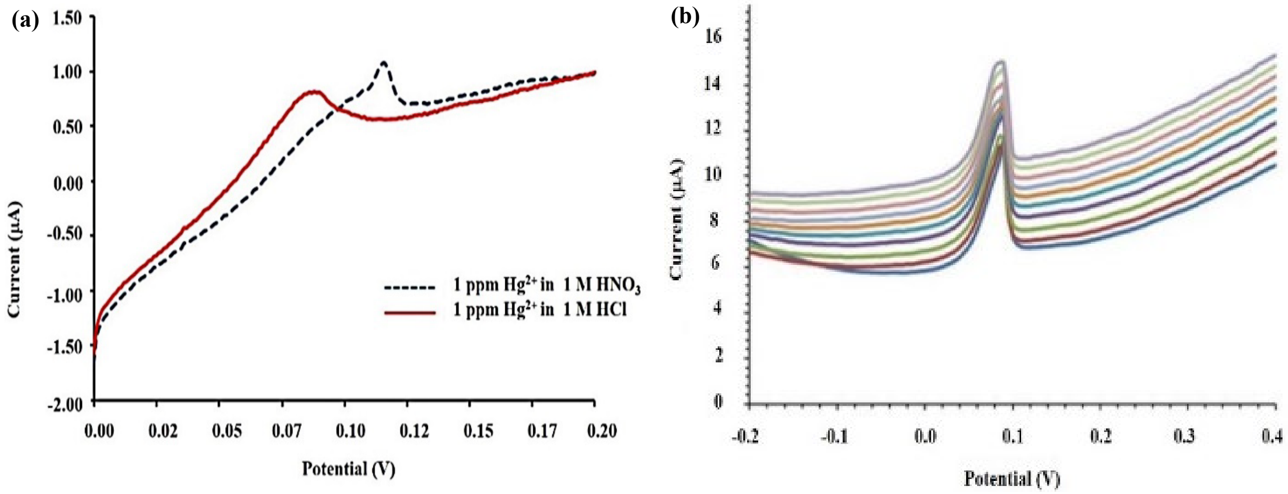

Figure 5. Voltammogram of 1 mg.L^{-1} Hg^{2+} in 1 M HCl and 1 M HNO$_3$ as the supporting electrolyte (A) and the voltammogram of 1 mg·L^{-1} Hg^{2+} in 1 M HCl, volume ratio 1: 1 sample solution (900 μL) and gold paltering solution at the initial potential scan from -0.2 V to +0.4V, step potential 0.005V and deposition time for 180s at the potential of -0.5V.

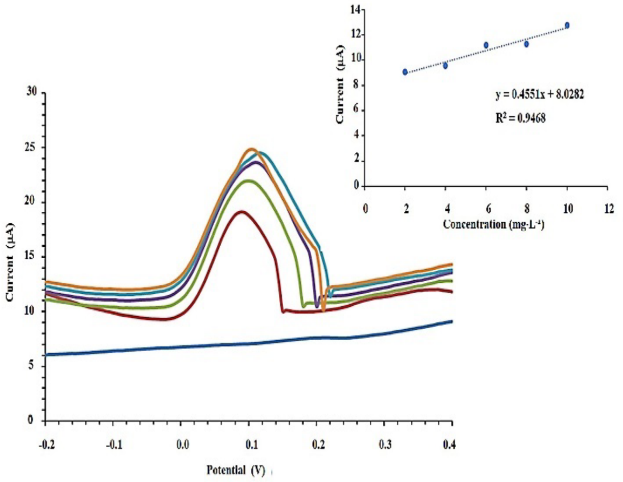

Figure 6. The calibration voltammogram and the linear concentration port of 2, 4, 6, 8 and 10 mg·L^{-1} Hg^{2+} in 1 M HCl with SIA on the situ gold film modification on screen-printed carbon electrode.

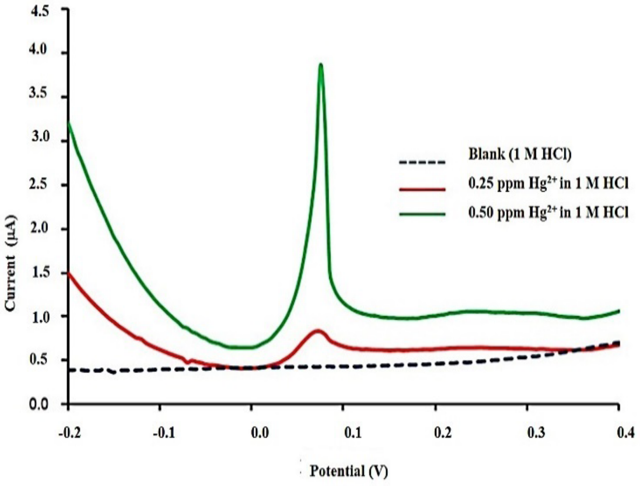

Figure 7. Voltammogram of 0.25 and 0.50 mg·L^{-1} in 1.0 M HCl as the supporting electrolyte (electrochemical condition: scan potential from -0.2 to +0.4V; step potential, 0.005V; scan rate, 0.01 V·s^{-1}).

4. Conclusions

The study found that in situ gold film modification on screen-printed carbon electrode were successfully fabricated. The gold film modification was used in the sequence injection system for the determination of Hg^{2+} by stripping analysis as low as 0.25 ± 0.18 mg·L^{-1}. The preconcentration potential was used at -0.5 V, the deposition flow rate was 10 μL·s and appropriately selected sample was 1,000 μL. The precision data of the method with this gold film modified screen-printed carbon electrode were satisfactory methods. In addition, the combination of SIA enables fast, automated determination of Hg^{2+} at a low cost.

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